

Development of Microwave-Microfluidic Sensors for Microplastic Detection in Environmental Samples

by

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Author's Declaration

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

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Abstract

Microplastics (MPs), plastic particles smaller than 5 mm, are emerging as a significant environmental threat due to their widespread presence in ecosystems and potential health impacts. They originate from both primary sources, such as microbeads in personal care products, and secondary sources, like the degradation of larger plastics. MPs can accumulate in aquatic life, pose risks to food chains, and carry toxic pollutants. Despite their environmental significance, detecting MPs in natural settings is challenging due to complex particle characteristics and the limitations of current detection methods.

Several well-established methods have been developed for detecting and monitoring MPs in aqueous samples. Fourier-transform infrared and Raman spectroscopy are among the most widely used techniques due to their unique ability to identify chemical compositions at the molecular level. However, these methods generally require bulky, expensive equipment and skilled personnel. Additionally, they are offline techniques that involve time-consuming and labor-intensive sampling processes. As a result, there is growing demand for affordable and user-friendly MP sensing techniques suitable for on-site applications.

Electrical sensing methods—including resonance microwave spectroscopy, dielectric spectroscopy, high-frequency impedance spectroscopy, and electrical impedance spectroscopy—offer unique advantages for on-site detection of MPs due to their compact detection systems and scalability for multi-location testing. Each method interacts differently with the electrical properties of the material, offering diverse capabilities for MP detection.

Although most electrical sensing methods share similar working principles, resonance microwave spectroscopy stands out as a promising solution due to its broader frequency range (typically 0.1–100 GHz), enabling more versatile and precise detection of various particle types. Microwave sensing differentiates materials based on their permittivity, making it highly sensitive for detecting MPs, which typically exhibit permittivity values (~ 2 – 3.5) distinct from their natural surrounding materials, such as water (~ 80), wet sediments (~ 10 – 30), and blood (~ 50 – 60). Furthermore, microwave sensors can be integrated with planar technologies, such as printed circuit boards (PCBs) and microstrip antennas, to create compact, lightweight, durable, and cost-effective systems, offering a practical solution for continuous measurements in real-world applications.

This thesis presents the development of microwave-microfluidic sensors for detecting and characterizing MPs in aqueous environmental samples, offering a scalable and cost-effective solution for real-time monitoring. It starts with an exploratory study capable of only concentration monitoring and progresses to an enhanced sensing platform capable of monitoring both size and concentration. Then, continuous flow is added to the sensing

platform to enable single-particle monitoring, which leads to MP size, type, and concentration characterization. In the final stage, the application of the microwave-microfluidic sensor extends from environmental to biomedical contexts.

The thesis begins by exploring the integration of microwave sensing with microfluidic platforms for detecting MPs in water. Experimental investigations were conducted using polyethylene microspheres of two different sizes (20 μm and 70 μm). The results indicate that the resonance frequency shift depends on particle size, concentration, and temperature. While experimental trends largely align with numerical simulations, the observed shifts were less pronounced than predicted, and the detection limits were higher than MP concentrations typically encountered in freshwater environments. These findings highlight the need for improved sensitivity and expanded applicability.

Building on this, a sensitivity-enhanced microwave sensing platform was introduced using coupled planar microwave resonators to characterize both the size and concentration of MPs in real time. The design incorporates an interdigital capacitor (IDC) structure with a traditional split-ring resonator (SRR) to enhance sensitivity. A disposable sample holder enables multiplex testing without cross-contamination, making the system field-deployable. The sensor was optimized through simulation and validated experimentally with MPs of three sizes (20 μm , 70 μm , and 275 μm) at various concentrations (100k, 1000k, and 10,000k particles/L). The results confirmed the sensor’s ability to monitor particle size and concentration accurately. However, since experiments were conducted with fixed sample volumes in the microliter scale, continuous flow integration was needed to improve statistical robustness.

The next project introduces an innovative AI-powered microwave-microfluidic platform that enables comprehensive analysis of MPs. The system analyzes particle size, concentration, and type using a K-nearest neighbors (KNN) algorithm trained on raw sensor data. Environmental samples are prefiltered into specific size ranges, and single-particle detection enables precise quantification of MP concentration. This approach offers a scalable solution for real-time monitoring of MP contamination across a wide size range (20–300 μm).

The final project demonstrates the potential of the microwave-microfluidic sensor in biomedical diagnostics. The platform was adapted to detect biomarkers in biological samples, focusing on monitoring amylase levels in postoperative peritoneal drainage fluid as an indicator of anastomotic leakage. This work highlights the broader utility of the sensor system for cost-effective, non-invasive real-time monitoring in both environmental and clinical settings.

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Dedication

To **Kian Pirfalak**, a nine-year-old boy whose boundless curiosity for engineering and vibrant joy for life will always be remembered, taken from this world far too soon.

Table of Contents

Examining Committee	ii
Author’s Declaration	iii
Abstract	iv
Acknowledgements	vi
Dedication	vii
List of Figures	xiii
List of Tables	xx
1 Introduction	1
1.1 Introduction	1
1.2 Thesis Outline	5
2 Literature Review	7
2.1 Environmental Issues and Concerns	7
2.1.1 Water Pollution	7
2.2 Microplastics: A Growing Issue	8
2.3 The State-of-the-Art Sensing Methods for Microplastic Detection	9

2.3.1	Chemical Methods	11
2.3.2	Mechanical Methods	14
2.3.3	Electrical Methods	16
2.4	Microwave Sensing	22
2.4.1	History of Microwave Sensing	22
2.4.2	Microwave Applications	23
2.4.3	Microwave Basic Principles	23
2.4.4	Microwave Structures	24
2.4.5	Microwave Sensing Parameters	26
2.4.6	Microplastic Detection Using Microwave Sensing	28
2.4.7	Frequency Dispersion and Temperature Dependence of Polymer Dielectric Properties	28
2.5	Microfluidic Systems for Environmental Monitoring and Microplastic Detection	29
2.5.1	Introduction to Microfluidic Systems	29
2.5.2	Applications of Microfluidic Systems in Environmental Monitoring	30
2.5.3	Integration of Microfluidic Devices with Microwave Sensors	31
3	Methodology: Design, Fabrication, and Experimental Setup for Microwave-Microfluidic Sensors	33
3.1	Numerical Simulation in Ansys HFSS	33
3.1.1	Problem Definition	34
3.1.2	Solution Process	34
3.1.3	Boundary Conditions	35
3.1.4	Excitation	36
3.1.5	Mesh Generation and Operations	36
3.2	Microwave Sensor Fabrication	37
3.3	Microfluidic Chip Fabrication	39
3.3.1	Fabrication with 3D-Printed Molds	39
3.3.2	Fabrication with Soft Lithography	40
3.4	Experimental for Characterization	41

4	Detection of Microplastics by Microfluidic Microwave Sensing: An Exploratory Study	42
4.1	Overview	42
4.2	Introduction	43
4.3	Materials and Methods	46
4.4	Numerical Simulations	48
4.5	Experimental Results and Discussion	54
4.6	Where Do We Go from Here?	58
4.7	Uncertainty Analysis	60
4.8	Summary	61
4.9	Supplementary Information	62
5	Size and Concentration Characterization of Microplastic Particles in Aqueous Samples Using Sensitivity-enhanced Coupled Planar Microwave Resonators	67
5.1	Overview	67
5.2	Introduction	68
5.2.1	Measuring principle	71
5.3	Sensor Design and Theory of Operation	71
5.3.1	Sensor Design and Simulation	71
5.3.2	Correlation between Microsphere Size and Deposition Speed in Aqueous Samples	77
5.3.3	Behavior of Electromagnetic Waves in Multi-Layer Medium with Varying Permittivity	78
5.4	Materials and Methods	80
5.5	Experimental Results and Discussions	81
5.6	Limitations and Future Outlook	89
5.7	Uncertainty Analysis	91
5.8	Summary	92

5.9	Supplementary Information	93
5.9.1	Forces Acting on a Sphere Moving Through a Viscous Fluid	93
5.9.2	Exponential Fitting Analysis of Figure 6 in the Manuscript	96
5.9.3	Linear Fitting Analysis of Figure 7 in the Manuscript	97
5.9.4	Sensor Dimensions	97
6	An AI-Powered Microwave-Microfluidics Platform for Comprehensive Microplastic Detection: Multi-variable Analysis of Size, Concentration, and Type	99
6.1	Introduction	100
6.2	Materials and Methods	103
6.2.1	Materials.	103
6.2.2	Fabrication of Microwave Microfluidic Sensing Platform.	104
6.2.3	Sample Preparation.	105
6.2.4	Experimental Procedure.	105
6.2.5	Particle Classification Using k-Nearest Neighbors.	105
6.2.6	Particle Sizing and Pre-Filtration Method.	106
6.3	Results and Discussions	106
6.3.1	Sensor Design and Simulations.	106
6.3.2	Analysis of Resonance Profiles of Selected Materials.	107
6.3.3	Accuracy of k-NN Model in Predicting Particle Type.	109
6.3.4	Performance of the Microwave Microfluidic Sensing Platform Across Different Particle Size Ranges	109
6.3.5	Efficiency of Sieve Shaker for Particle Size Sorting Across Different Materials	112
6.3.6	Evaluation of Microwave Microfluidic Sensing Platform Performance Across Different Liquid Carriers	113
6.4	Environmental Implications	113
6.5	Uncertainty Analysis	115
6.6	Summary	116
6.7	Supplementary Information	117

7	Expanding Applications of Microwave-Microfluidic Sensors: From Environmental to Biological Samples	121
7.1	Overview	121
7.2	Introduction	122
7.3	Experimental Section	125
7.3.1	Sensing Principle	125
7.3.2	Sensor Fabrication	127
7.3.3	Sensor Coating Procedure	127
7.3.4	Measurement Setup	128
7.3.5	Sample Preparation and Testing Protocol	128
7.3.6	Comparison with Standard Amylase Assay Kit	129
7.4	Results and Discussion	129
7.4.1	Sensor Design	130
7.4.2	Quasi-Continuous Monitoring of Amylase	132
7.4.3	Specificity and Environmental Effects	133
7.4.4	Comparison with Optical Methods and Clinical Relevance	138
7.5	Uncertainty Analysis	140
7.6	Summary	141
8	Conclusion and Recommendations	143
8.1	Contributions of this Thesis	143
8.2	Recommendations for Extension of Work	144
	References	147

List of Figures

2.1	Overview of scientific studies focused on the effects of micro and nanoplastics on human health. Colored squares represent pollutants (organic and inorganic) that could be present in environmental matrices (free or associated with micro and nanoplastics) and that could enter into the human body through different entry routes[1].	10
3.1	Different types of tetrahedral meshing elements.	37
3.2	Mesh refinement across adaptive passes: (a) Coarse initial mesh without seeding; (b) Finer initial mesh with seeding; (c) Enhanced mesh refinement in high-field regions for improved convergence.	38
3.3	Fabrication process of microfluidic networks in PDMS elastomer using the soft-lithography technique. (a) Steps for fabricating SU-8 molds, including exposure, development, and bonding processes to create sealed microfluidic devices. (b) Replication of the microfluidic structures in PDMS from the SU-8 master mold, followed by elastomer curing, hole punching, and bonding to complete the device assembly [2].	40
4.2	Numerically simulated S11-frequency plots for the samples with particle size of $70\text{ }\mu\text{m}$ at $10\text{ }^{\circ}\text{C}$, and for concentrations of 0, 100k, 1000k, and 10000k particles L^{-1}	50

4.3	Model-predicted frequency shifts as a function of particle concentration for suspensions with particle size of 20-300 μm and concentration of 0-10000k particles L^{-1} at a temperature of 10°C (a), 20°C (b), and 30°C (c). Panel (d) compares the results for the 200 μm particles at the temperatures of 10, 20 and 30°C. (Note: The frequency shifts for particles larger than 200 μm at a concentration of 10,000k particles L^{-1} are significantly larger compared to other data points and they have been excluded from the plots due to their exceptionally large magnitudes, which would make it challenging to effectively visualize them alongside other data points.)	52
4.4	(a) The model-generated S11curves for the 70 μm particles at a concentration of 1000k particles L^{-1} over three different temperatures of 10, 20, and 30°C, and (b) the effect of temperature on the resonance frequency.	53
4.5	Experimental frequency spectra of the Tween 20 0.1% solution and the 70 μm microsphere suspensions of 1000k particles L^{-1} at 10, 20, and 30°C.	55
4.6	Experimentally measured frequency shifts over the concentration range 0-10000k particles L^{-1} for suspensions with particle sizes of 20 and 70 μm , at (a) 10 °C, (b) 20°C and (c) 30°C, and (d) for the 70 μm microsphere suspensions at 10, 20, and 30°C.	56
4.7	(a) Schematic of the AI-powered QWave system (QuantWave Technology) with detailed microwave sensor design, and (b) cross-sectional view of the microwave sensor highlighting its components.	62
4.8	(a) Schematic of the fabricated sensor featuring two planar concentric ring-shaped electrodes with outer and inner ring radius of 9.75 mm and 7.25 mm, respectively. (b) The PDMS-made micro-reservoir, with a volume of 100 μL , positioned atop the sensor.	63
5.1	Microwave sensing mechanism for monitoring the concentration and size of microplastics: (a) Schematic of the system consisting of a microwave sensor with a reservoir hosting the sample under test which is a mixture of Tween 20 0.1% and microplastics and a vector network analyzer (VNA) used to monitor the transmission coefficient; (b) Numerically simulated transmission coefficient (S_{21}) as a function of the deposition rate of microplastics; and (c) Demonstration of the dependence of the experimentally measured resonance frequency shift as a function of the size of microplastics.	69

5.2	(a) The sensing platform schematic: the planar microwave reader and the chipless tag resonator. The planar microwave reader is responsible for transmitting and receiving microwave signals, while the chipless tag resonator interacts with the transmitted signals. The schematic diagram provides an overview of the arrangement and connection of these components. In addition, (b) presents a lumped-element equivalent circuit, which represents the electrical behavior and characteristics of the sensing platform. This circuit model helps in understanding the interactions and dynamics of the components within the platform.	73
5.3	(a) An analysis of the S_{21} response of the proposed sensor, a combination of the Interdigital Capacitor (IDC) and Split Ring Resonator (SRR) structure and that of the conventional SRR, (b) the electric field intensity surrounding the reservoir, which holds the sample under test and is positioned on top of the resonator, observed from the lateral perspective.	75
5.4	The absolute value of resonance frequency shifts across a permittivity range of 1 to 5, with an inset highlighting shifts at specific permittivity values (1, 1.0025, and 1.005). Since the resonance frequency at a permittivity of 1 is used as the reference, the resonance frequency shift at this permittivity equals zero and cannot be represented on the logarithmic scale of the main plot. However, it is included in the inset for clarity.	76
5.5	(a) Experimental setup for microplastic deposition monitoring. The characterization process of the sensor involves using a VNA, a device that measures and analyzes the electrical properties of the sensor, (b) zoomed view of the sensor with a Canadian dollar coin included for scale, (c) detailed view of the fabricated microwave reader, tag resonator, and PDMS container. . . .	82
5.6	Deposition monitoring of microplastic samples with varying sizes and constant concentration of 1000 k Particles/L, over 5 hours with 17 time intervals, demonstrating size-dependent slope variation. The findings reveal that the samples consisting of larger microplastics exhibited steeper slopes in comparison to those containing smaller microplastics.	83

5.7	Resonance Frequency Shift of Microplastic Samples with Varied Concentrations and Sizes: The resonance frequency shift of the samples was measured for three different concentrations: 100k Particles/L, 1,000k Particles/L, and 10,000k Particles/L. The samples contained microplastics of three different sizes: 20 μm , 70 μm , and 275 μm . Specifically: The resonance frequency shift of the sample containing microplastics with a size of (a) 20 μm , (b) 70 μm , and (c) 275 μm	86
5.8	Resonance frequency shift as a function of time for 275 μm polyethylene (PE) microplastic particles at a concentration of 100k particles/L. (a) Effect of temperature (10°C, 20°C, and 30°C) on resonance frequency shift, showing higher shifts at elevated temperatures. (b) Influence of different host mediums (DI water, tap water, 0.1 M NaCl solution, and 0.05 M urea solution) on resonance frequency shift, demonstrating minimal variations across different mediums.	87
5.9	Resonance frequency shift as a function of time for 275 μm polyethylene (PE) and soda lime glass (SLG) particles at a concentration of 1000k particles/L. The PE particles exhibit a higher final resonance frequency shift compared to SLG particles, indicating a material-dependent response.	89
5.10	Forces on a sphere in a viscous fluid: Gravitational (F_w), buoyant (F_b), and viscous drag (F_d) forces acting during motion.	94
5.11	Displacement of a microsphere (Δy) in a viscous fluid over time (t_1 to t_2).	95
5.12	The fabricated sensor including the microwave reader, resonator, and reservoir with labeled dimensions.	98
6.1	Overview of the Microwave-Microfluidic Platform for Microplastic Detection: (a) Microplastics (ranging in size from 1 μm to 5 mm) are a growing environmental issue that poses risks to both human health and aquatic ecosystems. This underscores the importance of developing a platform for their characterization, alongside an overview of the developed microwave microfluidic sensing platform for microplastic detection in this work. (b) Sensor breakdown illustrating the components of the microwave microfluidic sensor, including the planar microwave reader, microfluidic channel, and tag resonators. The figure also presents the raw data transmission curve and an AI-based prediction model for determining the size and type of detected microplastic particles.	102

6.2	Simulation Results: (a) Transmission coefficient (S_{21}) for three cases: Case 1: No particle, Case 2: Particle in the small resonator sensing region, and Case 3: Particle in the large resonator sensing region. (b) Electric field intensity on the two resonators at their respective resonance frequencies. .	108
6.3	Changes in resonance frequency and amplitude of S_{21} for four different materials. (a) Frequency shifts (Δf_r) at 1.2 GHz and 2 GHz for Polyethylene, Polystyrene, Soda Lime Glass, and Brine Shrimp Egg, with 50 data points for each material. (b) Corresponding changes in the amplitude of S_{21} at the two resonance frequencies for the same materials.	110
6.4	Performance evaluation of the microwave microfluidic sensing platform. (a) Number of correct predictions (out of 10) for each particle type predicted by the 4D k-NN model. The model achieved perfect classification for Polyethylene and Brine Shrimp Egg (10 out of 10 correct predictions), 9 out of 10 correct predictions for Soda Lime Glass, and 8 out of 10 correct predictions for Polystyrene. (b) Percentage of missed particles for each size range, based on a total of 20 reads (two sets of 10 particles per size range). The plot shows that smaller particles exhibit a higher percentage of missed detections. (c) Success rate (%) for each particle type, demonstrating the efficiency of the sieve shaker machine in sorting different materials. (d) Frequency shifts (Δf) at 1.2 GHz and 2 GHz for different liquid carriers. The plot shows the average resonance frequency shifts for each liquid carrier, illustrating the impact of the liquid carrier's dielectric properties on the sensor's performance.	111
6.5	Experimental setup for particle size analysis: Environmental sample with particles of varying sizes is prefiltered using a five-stage sieve system, followed by microfluidic chip-based particle detection. The planar microwave reader is compatible with all five chips for particle size classification.	117
6.6	Schematic representation of the microwave microfluidic sensin platform, with detailed dimensions of the planar microwave reader, tag resonators, glass slide, and microfluidic channels used for particle characterization.	118
6.7	Measured relative permittivity (real part, ϵ_r) of selected liquid carriers using the SPEAG DAK-TL2 probe, across a frequency range of 800 MHz to 2000 MHz.	119
6.8	Measured conductivity of selected liquid carriers using the SPEAG DAK-TL2 probe, across a frequency range of 800 MHz to 2000 MHz.	120

7.1	Overview of Anastomotic Leakage Monitoring. The top section illustrates the general diagnostic process for detecting anastomotic leakage (AL), showing the typical delay in diagnosis between postoperative day (POD) 6 and POD 9. The bottom section depicts our microwave biosensor, placed in-line with the drainage system from POD 1. The biosensor operates via an amylopectin-amylase enzymatic reaction, enabling continuous monitoring of amylase concentration in the drainage fluid. The readout offers sensitive detection once per hour, facilitating early and ongoing detection of AL throughout the entire postoperative period.	124
7.2	Principle of Working. Illustration of the experimental setup, with a focus on the reflection coefficient (S11) as the primary measured parameter. An inset displays the resonance profile changes resulting from the amylopectin-amylase interaction.	126
7.3	Full-wave simulation results: (A) Electric field distribution on the surface of the microwave-based biosensor, with an inset showing the electric field intensity in the X-Z plane at the sensor's midsection. (B) Numerical simulation results showing changes in the reflection coefficient before and after the Amylase-Amylopectin reaction. (C) Numerical simulation depicting the relationship between sensor surface amylopectin coverage and frequency shift, with 100% coverage defined as the initial state (sensor before reaction), for two different coating thicknesses of 5 μm and 20 μm	131
7.4	Quasi-Continuous Monitoring of Amylase. (A) Resonance profile of the microwave biosensor at an amylase concentration of 5000 U/L, measured at multiple time intervals (0, 15, 30, 45, and 60 minutes). The inset shows a zoomed-in view, highlighting the frequency shift more clearly. (B) Frequency shift over time for five different amylase concentrations (1000 to 5000 U/L), measured at regular intervals from 0 to 60 minutes.	133
7.6	Coating Device, and Characterization. (A) Fabrication Jig for Sensor Coating: Overview of the components used for assembling the coating jig, including holders, sensors, PDMS layers, anti-scratch protectors, and slides. The setup enables precise addition of amylopectin solution for uniform coating. (B) Coating Characterization: Visual and microscopic analysis of the sensor before and after reaction, highlighting the amylopectin layer's formation and uniformity. (C) Coverage vs. Reaction: Quantitative analysis of amylopectin coverage percentage across varying amylase concentrations (0–5000 U/L), demonstrating coating efficiency under different conditions.	136

7.7	Characterization of sensor performance with varying conditions: (A) Effect of amylopectin coating amount on amylase sensing results. (B) Comparison of tin and gold sensor surfaces with a 100 μ L amylopectin coating at 37°C.	138
7.8	Comparison of the proposed sensor with the optical amylase kit test. The left side of the figure shows the fabricated microwave biosensor placed in the testing jig, with a Canadian dollar coin for scale. The right side displays a comparison between the resonance frequency shift of the microwave biosensor (red line) and the flux intensity of the optical amylase kit test (blue line), both for different concentrations of amylase. The microwave biosensor operates within the clinically relevant concentration range of 1000 to 5000 U/L in deionized (DI) water and simulated peritoneal fluid (SPF) with 100 μ L of amylopectin coating at room temperature. In contrast, the optical amylase kit test is shown for amylase concentrations ranging from 0 to 2000 U/L in both DI water and SPF at room temperature.	139

List of Tables

2.1	Overview of Chemical, Mechanical, and Electrical Methods for Microplastic Detection	19
2.2	Overview of Chemical, Mechanical, and Electrical Methods for Microplastic Detection	20
4.1	Dielectric constants of the microsphere suspensions with varying particle size and concentration calculated with Equation (1). The table provides the values at a temperature of 10°C. The values at 20 and 30°C can be found in Tables 4.6 and 4.9.	50
4.2	Magnitudes of resonance frequencies simulated with HFSS for microsphere suspensions with varying sizes and concentrations at 10°C. Corresponding values at 20 and 30°C can be found in Tables 4.7 and 4.10.	51
4.3	Frequency shifts of the microsphere suspensions with varying sizes and concentrations at 10°C simulated with HFSS. Corresponding values at 20 and 30°C can be found in Tables 4.8 and 4.11.	51
4.4	Simulated and experimental frequency shifts of microsphere suspensions of 20 and 70 μm and concentrations ranging from 0 to 10000k particles L^{-1} at 30°C.	58
4.5	Comparative analysis of sensing technologies based on size range, size and concentration estimation capabilities, and environmental factor consideration.	59
4.6	Dielectric constants of the microsphere suspensions with varying particle size and concentration calculated with Equation (1). The table provides the values at a temperature of 20°C.	63
4.8	Frequency shifts of the microsphere suspensions with varying sizes and concentrations at 20°C simulated with HFSS.	64

4.7	Magnitudes of resonance frequencies simulated with HFSS for microsphere suspensions with varying sizes and concentrations at 20°C.	64
4.9	Dielectric constants of the microsphere suspensions with varying particle size and concentration calculated with Equation (1). The table provides the values at a temperature of 30°C.	65
4.10	Magnitudes of resonance frequencies simulated with HFSS for microsphere suspensions with varying sizes and concentrations at 30°C.	65
4.11	Frequency shifts of the microsphere suspensions with varying sizes and concentrations at 30°C simulated with HFSS.	66
5.1	Performance Comparison with State-of-the-Art	90

Chapter 1

Introduction

1.1 Introduction

Microplastics (MPs) are small plastic particles, typically defined as being less than 5 mm in size, that have emerged as a significant environmental concern due to their widespread distribution and persistence in ecosystems [3, 4, 5, 6]. These particles originate from two main sources: primary MPs, which are intentionally manufactured for use in products such as cosmetics, cleaning agents, and industrial abrasives, and secondary MPs, which result from the fragmentation of larger plastic items due to physical, chemical, and biological processes [7], [8], [9], [10]. Their pervasiveness in aquatic environments, soil, air, and even human tissues has raised serious concerns about their ecological and health impacts [11, 12, 13, 14, 12]. Microplastics have been detected in marine organisms, drinking water, and even atmospheric fallout, highlighting their ability to infiltrate food chains and biological systems [15], [16], [17], [18]. This infiltration poses potential threats to aquatic life through ingestion and bioaccumulation, and there is growing evidence that MPs can act as carriers for toxic pollutants, including heavy metals, persistent organic pollutants (POPs), and pathogens [19, 20, 21]. These particles may also cause physical damage and oxidative stress when ingested, further compounding their harmful effects [20, 21]. Given the rapid increase in global plastic production, estimated at approximately 320 million tons annually [22], and low recycling rates of only 9%-14% [23], microplastic pollution is expected to intensify unless effective monitoring and mitigation strategies are developed. Studying MPs is therefore crucial for assessing their prevalence, understanding their transport mechanisms, and evaluating their biological and ecological impacts [24], [25], [26], [27]. Developing accurate and scalable methods for their detection is essential not only for

environmental monitoring but also for assessing their risks to human health and designing mitigation strategies to minimize their spread [28, 29, 30, 31].

So far, there are several methods available for MP characterization in environmental samples [32]. Most of these methods rely on laboratory-based techniques, which are often time-consuming, expensive, and unsuitable for on-site applications [33]. Among the most widely used techniques are Fourier-transform infrared (FTIR) spectroscopy and Raman spectroscopy. These methods are highly accurate in identifying the chemical composition of MPs based on their unique vibrational fingerprints, enabling differentiation between polymer types [34], [35], [36] [37], [38], [39], [40], [41], [42], [43], [44], [45], [46], [47], [35], [39], [48], [40]. FTIR techniques, such as attenuated total reflectance (ATR-FTIR), can provide detailed spectral data for MP identification by scanning their surfaces. However, they require dry samples, precise alignment, and are generally not feasible for field applications [49]. Similarly, Raman spectroscopy is effective for analyzing MPs in both solid and aqueous forms, but it often struggles with interference from biological materials and involves lengthy sample preparation steps [50, 51, 52]. Both methods are highly sensitive and capable of detecting MPs down to micron sizes, but their reliance on sophisticated equipment and skilled operators makes them impractical for real-time and on-site monitoring [53, 54, 33].

In addition to spectroscopy techniques, gas chromatography-mass spectrometry (GC-MS) has been employed for analyzing MPs through thermal degradation, enabling identification of polymeric composition. Pyrolysis-GC/MS, for example, can effectively detect polymer types and relative masses but requires dry samples and extensive pre-treatment, posing challenges for in situ monitoring [55]. Similarly, thermal analysis methods, including thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), have been explored for MP quantification based on thermal degradation patterns. Despite their utility, these approaches are destructive and unsuitable for preserving particle morphology [56]. Hyperspectral imaging techniques, such as short-wave infrared (SWIR) spectroscopy, have also shown potential for rapidly classifying MPs based on spectral absorption features. However, their reliance on bulky and expensive equipment limits their application outside laboratory settings [57].

More recent approaches aim to reduce sample preparation requirements and enable real-time analysis. Techniques such as fluorescent dye staining, like Nile Red, have emerged as cost-effective methods for tagging MPs in environmental samples [58], [59]. However, these methods suffer from high false-positive rates due to interference from organic materials, making them unreliable without extensive pre-treatment [60], [61]. While these methods represent advancements in MP detection, they still face significant limitations in terms of scalability, field deployment, and cost-effectiveness.

The limitations of existing methods highlight the urgent need for a detection technique that is accurate, fast, cost-effective, and capable of real-time on-site monitoring. Most current approaches require centralized laboratories, involve destructive processes, or cannot effectively handle the complex and heterogeneous nature of MP samples in environmental settings. These drawbacks underscore the necessity of developing new platforms that can overcome these challenges while maintaining high sensitivity and specificity.

Electrical sensing methods offer unique advantages for on-site detection of MPs due to their compact detection systems and scalability for multi-location testing. Several approaches have been explored, including resonance microwave spectroscopy, dielectric spectroscopy, high-frequency impedance spectroscopy, and electrical impedance spectroscopy [62], [63], [64], [65]. Each method has a unique interaction with the electrical properties of the material, providing diverse capabilities for MP detection. Although most electrical sensing methods share similar working principles, RMS stands out as a promising solution for on-site MP detection due to its broader frequency range (typically 0.1 to 100 GHz), enabling more versatile and precise detection of various particle types. This wider frequency range enables versatile and precise detection of MPs by exploiting their dielectric and conductive properties, which differ significantly from those of natural surrounding materials, such as water and sediments [66], [67].

Microfluidic platforms complement electrical sensing methods by enabling precise manipulation of small sample volumes and integrating multiple analytical steps into a single device [68, 69, 70, 71, 72, 73, 74, 75]. These systems are well-suited for high-throughput and automated measurements, significantly reducing analysis time. Techniques such as acoustic and dielectric approaches, including acoustophoresis, have also been investigated for separating and concentrating MPs within microfluidic channels based on size and density [76, 77, 78, 79]. Despite their promise, existing microfluidic systems still face challenges related to scaling up for larger sample volumes and improving sensitivity to detect MPs at trace levels [80].

The integration of microwave sensors with microfluidic systems has emerged as a powerful strategy to address the limitations of traditional MP detection methods. Such hybrid platforms combine the advantages of high sensitivity and selectivity from microwave sensing with the versatility and precision of microfluidics, enabling scalable and real-time analysis. These systems generally fall into two categories: bulk measurements and single-particle detection. Bulk measurement techniques monitor the overall response of a sample containing multiple MPs, offering faster processing times and the ability to handle larger sample volumes. While most bulk methods focus primarily on determining MP concentration, some approaches, such as spiral resonators and multi-complimentary split ring resonators (CSRR), can also estimate particle size within a certain range. However, these meth-

ods require physical filters to sort particles, which complicates the system and reduces its simplicity and scalability [81], [63].

In contrast, single-particle detection methods, including high-frequency impedance spectroscopy and electrical impedance spectroscopy, can determine particle size, concentration, and type by analyzing each particle individually as it flows through the detection system [62], [64]. These techniques offer detailed and highly accurate measurements but necessitate complex setups capable of capturing both low- and high-frequency responses, increasing complexity and cost.

Both bulk and single-particle detection methods have their own advantages and limitations, making them suitable for different applications depending on the requirements for detail, speed, and scalability. Bulk measurement techniques are generally faster and capable of handling larger sample volumes, making them ideal for applications where overall concentration and size distributions are sufficient. However, most bulk methods rely on physical filters to separate particles based on size, which adds complexity to the system and may limit scalability. In contrast, single-particle detection methods provide detailed information about individual MPs, including size, concentration, and type, offering higher accuracy and specificity. Despite their advantages, these methods require precise flow control and more complex setups, which increase cost and limit throughput.

To address these challenges, this thesis presents two microwave microfluidic sensing platforms designed to overcome the limitations of traditional methods. The first approach, introduced in the Chapter 5, employs a microwave microfluidic sensor for bulk measurements, capable of characterizing MPs in terms of size and concentration without using physical filters. Instead, it introduces a deposition rate of particles, enabling simpler and scalable detection. The second approach, discussed in the Chapter 6, uses a microwave microfluidic sensor for single-particle sensing, providing detailed characterization of MPs based on size, concentration, and type. While the bulk method offers a rapid overview and averages measurements across particles in the sample, the single-particle approach delivers richer information about each particle, making it ideal for detailed analysis in more complex samples. Both approaches demonstrate the versatility of microwave microfluidic platforms, offering scalable, cost-effective, and field-deployable solutions for MP detection and monitoring.

Overall, the integration of microwave sensing and microfluidics, as proposed in this thesis, offers a powerful and versatile approach to overcome the limitations of existing MP detection methods. By leveraging differences in the electrical properties of MPs and combining them with the precision of microfluidic handling, this hybrid platform provides a scalable, cost-effective, and high-throughput solution for detecting and monitoring MPs in

both laboratory and field settings. In addition to its primary application for environmental monitoring, the platform’s adaptability extends its potential use to biological and clinical applications. This is demonstrated in Chapter 7, where the sensor is applied for detecting biomarkers, such as amylase, in postoperative peritoneal drainage fluid. This extension highlights the broader applicability of microwave microfluidic systems, showcasing their potential not only for MP detection but also for biomedical diagnostics and monitoring.

1.2 Thesis Outline

This thesis is organized into eight chapters, each addressing different aspects of the development, design, and application of microwave-microfluidic sensors for MP detection and extending their use to biomedical diagnostics.

Chapter 1 introduces the problem of microplastic pollution, emphasizing its environmental and health impacts, and highlights the need for scalable, accurate, and cost-effective detection methods. It outlines the research objectives and contributions, setting the foundation for the proposed microwave-microfluidic sensing platforms.

Chapter 2 provides a comprehensive literature review, covering existing MP detection techniques such as visual analysis, spectroscopy, thermal analysis, and electrical sensing methods. The chapter highlights the advantages and limitations of each approach, emphasizing the need for new technologies that overcome current constraints related to sensitivity, sample preparation, scalability, and field deployment. It concludes by introducing microwave sensing integrated with microfluidics as a promising solution.

Chapter 3 describes the design, fabrication, and experimental setup of the proposed sensors, starting with numerical simulations to optimize electromagnetic performance. It outlines the fabrication of microwave sensors and microfluidic chips using 3D printing and soft lithography techniques, followed by the characterization of sensor performance through experimental setup.

Chapter 4 presents the first experimental study, serving as a proof of concept, demonstrating the feasibility of using microwave-microfluidic sensors to detect MP concentrations based on permittivity changes. It validates the approach through simulations and experiments, identifying key design limitations and areas for improvement.

Chapter 5 expands on this by introducing a sensitivity-enhanced sensor capable of characterizing both MP size and concentration without physical filters. It explains the theoretical principles behind particle deposition rates and their effect on resonance frequency

shifts, supported by experimental validation that highlights the improved performance of the platform.

Chapter 6 describes the most advanced version of the sensor, integrating AI algorithms for multi-variable analysis of size, concentration, and type. It details the design and optimization process for single-particle sensing, demonstrating high accuracy and precision in characterizing individual MPs, which is particularly useful for toxicological and environmental studies.

Chapter 7 explores the broader applicability of the microwave-microfluidic platform by extending its use to biomedical diagnostics. It presents a side project focused on detecting biomarkers, such as amylase, in postoperative peritoneal drainage fluid for monitoring anastomotic leakage, showcasing the versatility of the technology in clinical applications.

Finally, Chapter 8 summarizes the key findings and contributions of the thesis, emphasizing the advantages of microwave-microfluidic sensors in overcoming the limitations of traditional methods. It provides recommendations for future research, including enhancing sensor sensitivity and selectivity, as well as expanding applications to other fields, such as biomedical applications, agro-environmental monitoring, and water quality assessment.

Chapter 2

Literature Review

2.1 Environmental Issues and Concerns

The health and well-being of people across the globe are intricately linked to the state of the natural environment. A balanced and thriving ecosystem not only provides essential resources like clean water, air, and food but also supports economic growth and shields communities from natural disasters [82]. However, environmental challenges have grown increasingly complex and widespread, posing significant risks to both economic stability and ecological sustainability.

In the past, environmental impacts were often perceived as isolated, confined to specific regions or sectors. Today, it is evident that these boundaries have dissolved, revealing the interconnected nature of environmental problems. This growing awareness highlights the urgent need for global cooperation to address critical issues such as water pollution, deforestation, air pollution, land degradation, waste accumulation, and climate change [83]. Among these challenges, water pollution holds particular relevance to this study and is examined in greater depth below.

2.1.1 Water Pollution

Water, often regarded as the planet's most vital resource, is fundamental to sustaining life and ecosystems [84]. However, the contamination of water bodies, especially through plastic waste, has become one of the most pressing environmental concerns of our time. The production of plastic has surged globally, with an estimated 360 million metric tons

produced in 2018 alone. This figure grows even larger when accounting for plastics used in the manufacture of synthetic textiles, rubber, and various additives [85].

Despite its utility, the recycling rate for plastic remains disproportionately low. Most plastic waste is disposed of in landfills, open dumps, or natural environments, where it frequently finds its way into rivers, lakes, and oceans. Current consumption trends and ineffective waste management practices exacerbate the issue, with projections suggesting that by 2050, approximately 12 billion metric tons of plastic debris will accumulate in landfills, dumps, and natural habitats [85].

The growing scale of plastic pollution calls for creative solutions, especially to tackle the problem in aquatic environments. This research takes on this challenge by developing advanced sensors to detect plastic contamination in water, playing a small but vital role in efforts to protect and preserve this essential resource.

2.2 Microplastics: A Growing Issue

In today’s world, it is nearly impossible to imagine life without plastics. Their ability to withstand extreme weather conditions, resist water, remain durable, and be cost-effective has made plastics a preferred material for manufacturing a wide range of everyday items, including packaging, electronics, and clothing. However, these same properties have contributed to plastics becoming a significant component of global waste streams. Despite efforts to reduce plastic consumption, improve waste management, and promote recycling, the volume of discarded plastic waste continues to grow annually. Much of this waste is often mismanaged, ultimately making its way into the environment [86], [87].

Although plastics are known for their durability, they inevitably degrade over time. Larger plastic items fragment into smaller pieces, which are classified as microplastics (MPs) when their size is less than 5 millimeters [88], [89]. These tiny fragments are estimated to persist in the environment for hundreds, if not thousands, of years [90]. MPs are particularly concerning as environmental contaminants due to their potential to be ingested by organisms, with evidence indicating they can harm both ecosystems and individual species. Beyond their role as particulate pollutants, MPs can also act as sources of organic chemicals, as plasticizers and other additives leach from degrading plastics into the surrounding environment [91].

The recognition of MPs as environmental contaminants dates back to the early 1970s when researchers first identified small plastic particles in marine samples [92], [93]. This discovery revealed that plastic pollution extended beyond the visible litter scattered through-

out the environment to include microscopic fragments. MPs are highly mobile, capable of spreading far from their original sources through wind, water currents, or human activity [94], [95], [96]. Once introduced into ecosystems, MPs can accumulate in sediments or move through the water column, making them bioavailable to organisms across various trophic levels [97], [98]. The environmental and ecological effects of MPs are influenced by several factors, including their size, shape, polymer type, and associated chemicals [98], [99].

MPs are not only an environmental concern but also a potential human health risk. They can enter the human body through ingestion of contaminated food, trophic transfer, inhalation, or even skin contact. However, their fate and effects inside the body remain a topic of ongoing research and debate. For MPs to penetrate human organs, they must be smaller than 20 micrometers in diameter. Particles around 10 micrometers in size have the potential to cross biological barriers such as cell membranes, the blood-brain barrier, and the placenta. Once inside the body, these particles may be distributed to secondary tissues, including the liver, muscles, and brain [1], [100], [101]. An overview of scientific studies focused on the effects of micro- and nanoplastics on human health is illustrated in Figure 2.1. The figure also depicts the potential pollutants (organic and inorganic) associated with micro- and nanoplastics in environmental matrices and their possible entry routes into the human body. These pollutants, represented by colored squares, highlight the complex interplay between environmental contaminants and human exposure pathways.

2.3 The State-of-the-Art Sensing Methods for Microplastic Detection

Detecting MPs is essential for monitoring their presence and evaluating the effectiveness of mitigation measures aimed at reducing their environmental impact [102]. This foundational step is critical for addressing MP pollution and informing the development of effective treatment strategies. Over the years, numerous techniques have been developed for MP detection; however, the results obtained from these methods often lack consistency and reproducibility. This section provides a comprehensive overview of MP detection techniques and their applications.

Due to their high surface area-to-volume ratio and hydrophobic nature, MPs have a unique ability to adsorb persistent organic pollutants (POPs), such as polychlorinated biphenyls and polycyclic aromatic hydrocarbons. These pollutants, along with plasticizers, can leach into the gastrointestinal fluids of organisms and accumulate in edible tissues,

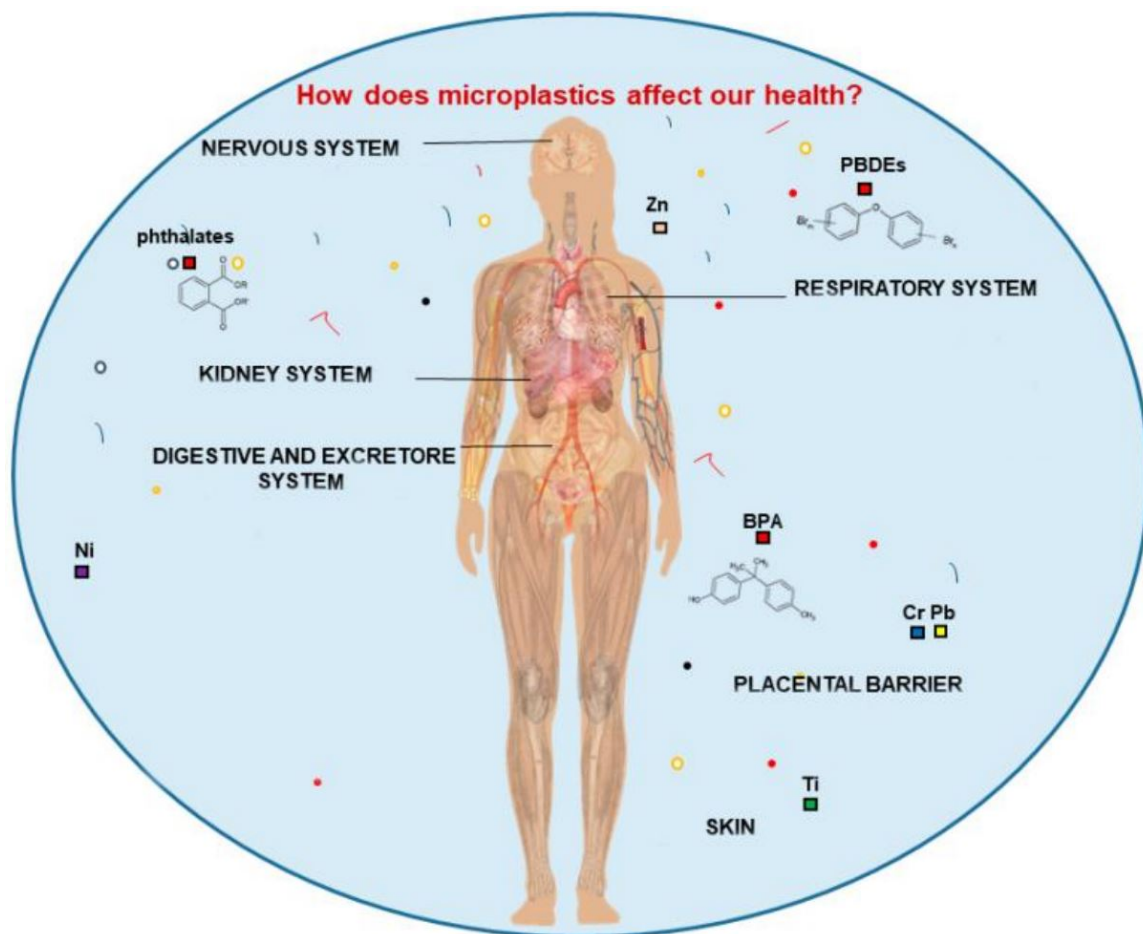


Figure 2.1: Overview of scientific studies focused on the effects of micro and nanoplastics on human health. Colored squares represent pollutants (organic and inorganic) that could be present in environmental matrices (free or associated with micro and nanoplastics) and that could enter into the human body through different entry routes[1].

posing significant risks to the food chain. In aquatic environments, the distribution of MPs varies widely depending on their shape and density, influencing trophic interactions across multiple levels. As the size of MP particles decreases, their bioavailability increases dramatically, which amplifies their potential to cause ecological and biological harm [103]. Consequently, it is crucial to accurately determine the concentration, size, shape, and type of MP particles in environmental samples.

MP quantification generally follows two approaches: quantitative concentration and mass concentration. Quantitative concentration involves counting individual MP particles and converting these counts into mass-based metrics for environmental samples. Common methods used for this purpose include visual analysis and spectroscopy. However, the brittle nature of MPs often leads to fragmentation during analysis, resulting in inconsistencies in particle counts and reduced accuracy in quantitative concentration measurements [104].

Conversely, mass concentration measurements are less affected by the physical and chemical properties of the environment, offering a more reliable representation of the overall environmental load of MPs. This approach also provides valuable insights into the sources of MP pollution [105]. Among the various techniques available, thermal analysis stands out as a widely used and effective method for assessing the mass concentration of MPs in environmental samples. Its reliability and broader applicability make it a critical tool for MP pollution studies [106].

To effectively quantify and analyze MPs, various methods have been developed, categorized based on the physical, chemical, and mechanical properties of MPs. Each method offers unique advantages and limitations, depending on the target application and environmental context. Chemical methods focus on the molecular and compositional analysis of MPs, providing detailed insights into their polymeric structure. Conversely, mechanical methods leverage the physical characteristics of MPs, such as size, density, and elasticity, to facilitate their separation and identification. The following sections provide an in-depth review of these techniques, highlighting their principles, applications, and challenges. [107]

2.3.1 Chemical Methods

Chemical analysis methods target the molecular composition and chemical properties of MPs, enabling detailed insights into their polymer types, size, and distribution. These techniques are indispensable for accurately characterizing MPs in various environmental samples.

Pyrolysis-Gas Chromatography/Mass Spectrometry

Pyrolysis-Gas Chromatography/Mass Spectrometry (Py-GC/MS) is a widely used technique for analyzing MPs by thermally decomposing plastic materials into smaller molecular fragments. These fragments are separated by gas chromatography and identified using mass spectrometry, providing a precise chemical signature of each polymer type. Py-GC/MS is particularly effective for mixed and complex polymer samples, offering reliable polymer identification and relative mass quantification [108].

Py-GC/Differential Mobility Spectrometry (Py-GC/DMS) builds upon Py-GC/MS by incorporating a differential mobility spectrometry system, which enhances portability and reduces equipment size and costs. Py-GC/DMS has demonstrated robust performance in field applications, enabling rapid polymer identification with lower operational complexity. Both methods, however, require dry samples and involve time-intensive data collection, limiting their scalability for high-throughput analysis [109].

Spectroscopy Techniques (FTIR and Raman Spectroscopy)

Spectroscopy techniques, particularly Fourier Transform Infrared (FTIR) and Raman spectroscopy, are extensively used for the classification and quantification of MPs based on their unique vibrational spectra. These methods provide valuable information about the polymer composition, size, and morphology of MPs while preserving the integrity of the samples due to their non-destructive nature.

FTIR spectroscopy is widely employed to identify MPs larger than 20 μm using techniques such as attenuated total reflectance (ATR). By analyzing the absorption of infrared light, FTIR provides detailed polymer-specific spectral fingerprints, enabling accurate identification of plastic types in environmental samples [110, 111]. However, this method requires chemical pretreatment, the use of dried samples, and precise scanning, making it more suitable for laboratory applications due to its time-intensive nature and reliance on costly equipment.

Raman spectroscopy, on the other hand, is more effective for detecting and quantifying MPs smaller than 20 μm . This technique involves measuring inelastic scattering of light, providing detailed molecular information. The integration of Raman microscopy has significantly enhanced resolution and sensitivity, enabling the detection of MPs as small as 1 μm [112, 113]. Despite these advantages, Raman spectroscopy is hindered by high equipment costs, complex sample preparation requirements, and susceptibility to interference from biofilm coatings.

To improve the accuracy and efficiency of MP detection, FTIR and Raman spectroscopy are often used in tandem, complementing each other’s strengths. Advanced approaches such as μ -FTIR, which integrates FTIR with microscopy, have been developed to enhance detection capabilities. For example, Zhang et al. demonstrated that μ -FTIR enables the detection of MPs as small as 10 μm , improving quantification in environmental samples [114]. Similarly, Loder et al. introduced focal plane array detector-based micro-FTIR imaging, which is capable of detecting MPs approximately 20 μm in size within marine plankton and sediment samples [115, 116]. In parallel, micro-Raman spectroscopy has emerged as a promising non-contact and non-destructive technique that combines Raman spectroscopy with microscopy to analyze MPs in various samples. However, micro-Raman is limited by its capacity to process large numbers of particles, often underestimating MP abundance in complex samples due to the technique’s constraints on particle size detection [117, 118].

While spectroscopic techniques are indispensable for MP detection, their reliance on expensive and bulky optomechanical equipment, as well as their requirement for dried and pretreated samples, restricts their utility in field settings. Additionally, interference from biofilm coatings and other environmental contaminants can reduce accuracy. Nonetheless, ongoing advancements in miniaturization, automation, and integration of spectroscopic methods hold significant promise for improving their accessibility and applicability in real-world MP pollution monitoring.

Hyperspectral and Multispectral Imaging

Hyperspectral and multispectral imaging techniques leverage the optical properties of MPs across different wavelengths to provide data on polymer type, size, and count. These methods use short-wave infrared (SWIR) regimes to identify MPs based on their spectral signatures.

Hyperspectral imaging offers high-resolution data but requires precise optical alignment and dried samples, making it time-intensive and unsuitable for rapid field analysis [119].

Multispectral imaging provides faster and more portable imaging capabilities compared to hyperspectral techniques. It is particularly advantageous for field applications, but like other imaging methods, it is less effective when biofilm contaminants are present [120].

Fluorescent Dye-Based Imaging

Fluorescent dye-based imaging is a low-cost and accessible method for detecting MPs. Dyes such as Nile Red selectively bind to the surfaces of MPs, enabling visualization under fluorescence microscopy. This technique is portable and straightforward, making it suitable for field studies. However, it suffers from limitations, including false positives due to biofilm contamination and interference from organic matter. Developing environmentally friendly, low-toxicity dyes with higher specificity remains an active area of research [61, 121].

2.3.2 Mechanical Methods

Mechanical methods leverage the physical properties of MPs, such as size, density, and elasticity, to facilitate their separation and identification. These methods are particularly valuable as pre-analysis techniques, enabling the isolation and purification of MPs from complex environmental matrices. While they are not always sufficient for comprehensive chemical characterization, they often complement chemical methods to improve overall detection accuracy.

Laser Optical Trapping

Laser optical trapping utilizes highly focused laser beams to manipulate and isolate individual MPs based on their optical and physical properties. This method has shown potential for separating MPs from non-MPs when coupled with Raman spectroscopy, enabling simultaneous size-based sorting and chemical identification. However, the technique requires a controlled laboratory environment and precise calibration, limiting its field applicability. Early demonstrations in microfluidic setups have shown promise for reducing sample preparation time and enhancing sorting efficiency [122, 123, 124, 125].

Photonic Optical Trapping

Photonic optical trapping, a related technique, employs light waves to sort and manipulate MPs. This method has been integrated into microfluidic systems for particle sorting and imaging. While it offers high precision, its reliance on advanced optical equipment and controlled conditions restricts its use outside of laboratory settings. Current research focuses on optimizing its applications for bio-sensing and imaging, potentially expanding its utility in environmental monitoring [126, 127, 128].

Field Flow Fractionation (FFF)

Field flow fractionation (FFF) is a powerful separation technique that utilizes a combination of centrifugal, thermal, or asymmetric flow fields to fractionate MPs based on their size and density. This method is often integrated with chemical analysis techniques, such as Raman spectroscopy, to achieve both separation and polymer identification. FFF has been demonstrated in microfluidic systems, reducing sample preparation time and enabling the separation of MPs from organic matter in environmental samples. However, its dependence on specialized equipment and operational complexity limits its scalability for field applications [129].

Acoustophoresis

Acoustophoresis employs sound waves to manipulate and sort MPs based on their acoustic properties. This method is particularly effective for separating MPs from biological and organic particles in water. It is highly portable and can be integrated into microfluidic devices, making it suitable for field studies. Recent advancements in microfluidic acoustic devices have demonstrated their capability to achieve high sorting efficiencies with minimal sample preparation [130, 131].

Ultrasound

Ultrasound techniques use high-frequency sound waves to isolate MPs by exploiting their unique acoustic signatures. This method has been integrated into microfluidic systems to minimize sample preparation times and enable precise particle manipulation. Ultrasound-based sorting is relatively low-cost and portable, making it a promising candidate for field-deployable MP detection systems. However, its effectiveness is influenced by particle size and the acoustic properties of the surrounding matrix, which may limit its accuracy in complex samples [132].

In conclusion, while significant advancements have been made in the development of analytical methods for detecting MPs, many existing techniques still face notable limitations. Chemical and mechanical methods, though effective in providing detailed compositional, morphological, or size-based data, often struggle to balance precision, efficiency, and field applicability. Most methods require extensive sample preparation, costly equipment, or laboratory settings, which limit their scalability for real-world applications. Furthermore, reliance on a single technique often results in incomplete data, with risks of false-positive or false-negative signals, highlighting the need for complementary or hybrid approaches.

At present, no single method can comprehensively address the challenges of MP detection across all environmental matrices, particularly in complex scenarios involving mixed contaminants. To advance the field, future efforts must focus on developing integrated approaches that combine the strengths of multiple techniques, prioritizing rapid, cost-effective, and practical solutions. In this context, electrical sensing methods have emerged as a promising alternative, offering innovative opportunities for non-invasive, scalable, and efficient MP detection. By leveraging advanced electrical sensing technologies, researchers aim to address existing limitations and establish more reliable platforms for environmental monitoring. The next section explores the principles, applications, and recent progress in electrical sensing as a cutting-edge approach to MP detection.

2.3.3 Electrical Methods

Electrical methods have gained significant attention as effective approaches for MP detection, offering the advantages of rapid, non-invasive analysis and compatibility with various environmental matrices. These methods are particularly suitable for field applications due to their ability to provide real-time data with minimal sample preparation. By exploiting the distinct electrical properties of MPs, such as permittivity and impedance, these techniques enable reliable differentiation of MPs from biological and other natural particles. This section reviews the key electrical techniques, including impedance spectroscopy, capacitance sensing, dielectrophoresis, dielectric spectroscopy, and microwave sensing, highlighting their principles, applications, and advancements in MP detection.

Impedance Spectroscopy

Impedance spectroscopy is a versatile technique that evaluates the electrical properties of particles as they interact with an electric field. This method is categorized into two key approaches: Electrical Impedance Spectroscopy (EIS) and High-Frequency Impedance Spectroscopy (HFIS).

EIS measures impedance over a wide range of frequencies, offering insights into the electrical characteristics of MPs and their surrounding media. This method is particularly effective in distinguishing MPs from biological materials due to differences in frequency-dependent dispersion. EIS has been successfully integrated into microfluidic systems for real-time MP detection, enabling high-throughput analysis with minimal sample preparation. However, EIS is more effective at lower frequencies, where MPs exhibit relatively stable impedance characteristics, making it ideal for water-based samples [133], [134], [62].

HFIS extends the frequency range to higher values, allowing for the detection of more subtle dielectric contrasts between MPs and their environment. This approach is highly sensitive to changes in the dielectric properties of MPs, enabling the characterization of smaller particles and the detection of trace contaminants. HFIS is particularly valuable for applications where precision and sensitivity are critical, such as detecting MPs in complex environmental matrices or mixed contaminant scenarios. Despite its higher sensitivity, HFIS often requires advanced equipment and computational processing, which may limit its field deployability [135], [64].

Dielectrophoresis (DEP)

DEP is a promising method for manipulating MPs by applying a non-uniform electric field, which generates a force that moves particles based on the contrast between their permittivity and that of the surrounding medium. This approach is advantageous because it is non-invasive and does not require chemical labeling, making it a cost-effective and selective technique for separating MPs from non-MPs and even differentiating between MPs of different polymer types. DEP is particularly well-suited for integration into microfluidic systems, enabling miniaturized and portable sensing devices that can concentrate, sort, and potentially classify MPs based on their physical and dielectric properties. However, DEP does face certain challenges, including reduced effectiveness in highly conductive environments such as seawater, where electrical losses can diminish its ability to manipulate particles. Additionally, biofilm-coated MPs or particles with surface contaminants can alter dielectric properties, reducing the accuracy and selectivity of the method. The technique's performance also decreases for very small particles, such as nanoplastics, due to weaker forces acting on these particles. Furthermore, DEP systems require precise control of electric fields and complex electrode designs, increasing the operational complexity and cost, while potential localized heating effects from high-intensity fields may interfere with measurements. Despite these limitations, the ability of the DEP to perform selective separation and its scalability for high-throughput analysis make it a valuable tool for MP sensing, particularly as advancements are made to overcome its current drawbacks [136, 137] .

Dielectric Spectroscopy

Dielectric spectroscopy is a powerful technique that evaluates the frequency-dependent dielectric properties of MPs, such as permittivity and loss factor, to provide detailed insights into their composition and morphology. This method is highly effective in distinguishing

MPs based on their unique dielectric signatures, enabling the classification of polymer types and contributing to enhanced detection accuracy. One of the key advantages of dielectric spectroscopy is its ability to analyze a wide range of frequencies, offering a comprehensive profile of the material’s electrical properties. This makes it particularly useful for identifying MPs in complex environmental matrices where chemical methods may fall short. However, dielectric spectroscopy has certain limitations, including its sensitivity to sample preparation and environmental conditions. The presence of water or other highly conductive media can interfere with measurements, potentially masking the dielectric properties of MPs. Additionally, the technique often requires specialized equipment and controlled laboratory conditions, which can limit its applicability for field-based studies. Despite these challenges, dielectric spectroscopy remains a valuable tool for complementing other electrical sensing methods, as it provides unique material-specific information that can enhance the reliability and comprehensiveness of MP detection systems [65].

Microwave Sensing

Microwave sensing leverages the interaction between electromagnetic waves and MPs to analyze their electrical properties, such as permittivity and loss tangent. This technique provides several advantages, including non-invasive analysis, high sensitivity, and the ability to detect MPs even when coated with biofilms, making it particularly suitable for complex environmental samples. Recent advancements, such as the integration of microwave sensors with microfluidic systems, have further enhanced their potential for high-throughput and precise MP detection across diverse environmental matrices [138], [139], [81], [140], [63], [66]. As the primary focus of this thesis is the development of a microwave sensor specifically tailored for MP detection, a more detailed discussion of this method will be presented in the subsequent section.

The chemical, mechanical, and electrical methods discussed in this section, and summarized in Table 2.1, provide a concise overview of techniques available for MP detection, with their respective advantages and disadvantages detailed further in Table 2.2. Chemical methods excel in polymer characterization and compositional analysis, offering high precision but often requiring extensive sample preparation and laboratory settings. Mechanical methods effectively leverage physical properties for separation and identification, but scalability and field applicability remain challenges. Electrical methods, such as impedance spectroscopy and microwave sensing, provide innovative, non-invasive solutions with high sensitivity and field potential, though environmental noise and cost can limit their utility. Together, these methods underscore the need for integrated approaches to overcome individual limitations and ensure scalable and accurate solutions for environmental monitoring.

Table 2.1: Overview of Chemical, Mechanical, and Electrical Methods for Microplastic Detection

Category	Method	Outputs
Chemical	Py-GC/MS	Polymer Type, Relative Mass
	FTIR Spectroscopy	Polymer Type, Count, Size
	Raman Spectroscopy	Polymer Type, Size, Count
	Hyperspectral Imaging	Polymer Type, Count, Size
	Py-GC/DMS	Polymer Type, Relative Mass
	Multispectral Imaging	Polymer Type, Count, Size
	Fluorescent Dye	Count, Size
Mechanical	Laser Optical Trapping	Separation from Non-MPs
	Photonic Optical Trapping	Size, Separation from Non-MPs
	Field Flow Fractionation	Size, Separation from Non-MPs
	Acoustophoresis	Polymer Type, Size, Separation from Non-MPs
	Ultrasound	Polymer Type, Size
Electrical	Impedance Spectroscopy	Polymer Type, Count, Size
	Dielectrophoresis	Polymer Type, Count, Size
	Dielectric Spectroscopy	Relative Mass
	Microwave Sensing	Polymer Type, Count, Size

Table 2.2: Overview of Chemical, Mechanical, and Electrical Methods for Microplastic Detection

Category	Method	Advantages	Limitations
Chemical	Py-GC/MS	Highly precise chemical identification and quantification of polymers in mixed samples. Useful for relative mass determination.	Requires dry samples, time-intensive, costly, and primarily suitable for laboratory use.
	FTIR Spectroscopy	Non-destructive identification of MPs larger than 20 μm . Provides detailed polymer-specific spectral fingerprints.	Time-intensive, requires chemical pretreatment and dried samples. Expensive equipment limits field use.
	Raman Spectroscopy	Effective for MPs smaller than 20 μm . Non-contact and non-destructive. High sensitivity and resolution.	Equipment is costly, sample preparation is complex, and prone to biofilm interference.
	Hyperspectral Imaging	High-resolution polymer type and size detection using short-wave infrared (SWIR).	Expensive and time-intensive. Requires precise optical alignment and dried samples.
	Multispectral Imaging	Faster and more portable than hyperspectral imaging. Suitable for field applications.	Less effective in the presence of biofilm or organic contaminants.
	Fluorescent Dye	Low-cost and accessible. Visualizes MPs using selective dyes like Nile Red under fluorescence microscopy.	Potential for false positives due to organic matter. Requires further development of environmentally friendly dyes.
Mechanical	Laser Optical Trapping	Enables precise size-based separation and chemical identification when combined with spectroscopy.	Requires controlled environments and advanced calibration. Limited field applicability.

Category	Method	Advantages	Limitations
	Photonic Optical Trapping	Allows high-precision manipulation and imaging of MPs in microfluidic systems.	Relies on advanced optical equipment and controlled laboratory conditions.
	Field Flow Fractionation	Separates MPs based on size and density. Works well with chemical analysis techniques for polymer identification.	Requires specialized equipment and operational complexity.
	Acoustophoresis	Portable and suitable for field studies. Effective separation of MPs from biological or organic particles.	Influenced by sample properties and acoustic interference in complex environments.
	Ultrasound	Relatively low-cost and portable. Minimizes sample preparation time and enables precise particle manipulation.	Accuracy may vary depending on particle size and acoustic properties of the matrix.
Electrical	Impedance Spectroscopy	Measures electrical properties for polymer type identification. Non-invasive and versatile.	Sensitive to environmental noise. Requires advanced signal processing and calibration.
	Dielectrophoresis	Separates and classifies MPs based on dielectric properties. Suitable for integration into portable systems.	Limited effectiveness in highly conductive environments or with biofilm-coated particles.
	Dielectric Spectroscopy	Provides frequency-dependent dielectric properties for polymer classification. Complements other methods.	Requires careful calibration and is less effective in complex sample matrices.

Category	Method	Advantages	Limitations
	Microwave Sensing	Non-invasive, high sensitivity, and capable of analyzing biofilm-coated MPs. Integrates with microfluidic systems for high-throughput analysis.	Equipment cost and complexity may limit widespread adoption in the field.

2.4 Microwave Sensing

2.4.1 History of Microwave Sensing

The development of microwave sensors began in the 1950s, driven by efforts to create better methods for measuring permittivity and understanding its relationship with the physical properties of materials and mixtures. However, the foundation of modern microwave engineering can be traced back to the late 19th century when James Clerk Maxwell formulated the electromagnetic theory, which Heinrich Hertz later validated through experiments in the 1880s. This theoretical groundwork laid the basis for practical applications such as guided waves and transmission lines, enabling innovations like radar, radio, and wireless communication systems. Significant progress in microwave technology occurred during World War II, particularly at the Massachusetts Institute of Technology’s Radiation Laboratory. Here, scientists like I. I. Rabi and H. A. Bethe advanced waveguide components, small-aperture coupling theory, and microwave antenna designs. This period marked the beginning of modern microwave engineering, with developments that remain influential today [141].

Despite the theoretical advancements, early microwave sensors in the 1950s were limited in application due to their high cost, large size, and the lack of advanced signal processing capabilities. The 1970s brought significant improvements with the introduction of solid-state components, and by the early 1980s, microprocessors enabled the development of simple, cost-effective, yet sophisticated microwave sensors. These innovations catalyzed a rapid expansion of microwave applications and commercial products. Today, microwave technology is widely used for sensing in challenging environments, inaccessible real-time monitoring, bio-implants, telesurgery, lab-on-a-chip systems, drug delivery, air-quality monitoring, and pipeline integrity monitoring under high temperature and pressure conditions [142, 143].

2.4.2 Microwave Applications

Microwave engineering has found extensive applications across diverse fields due to the unique properties of microwave frequencies, such as high bandwidth, non-invasive interaction with materials, and line-of-sight propagation. In communications, microwave technology is essential for systems like mobile broadband, satellite communications, and wireless LAN protocols [144]. Its high frequencies enable increased bandwidth and higher data rates, making it integral to modern telecommunication infrastructure [145]. Additionally, the line-of-sight nature of microwave signals is ideal for satellite and terrestrial links, ensuring reliable communication over long distances [146].

In remote sensing, microwaves are used in radar systems, radiometry, and radio astronomy [147]. These applications benefit from the strong reflective properties of microwaves, which enable high-precision measurements. In biomedical applications, microwaves are utilized for diagnostic imaging and therapeutic procedures like cancer thermotherapy, offering non-invasive solutions for healthcare advancements [148].

Microwave technology also plays a significant role in industrial processes, including food processing, semiconductor manufacturing, and moisture control [149, 150, 147]. Its ability to selectively and efficiently heat materials makes it ideal for drying, reheating, and chemical processing [151]. This thesis focuses specifically on microwave sensing, leveraging its high sensitivity and adaptability to develop innovative solutions for detecting microplastics and other environmental pollutants.

2.4.3 Microwave Basic Principles

Generally, microwave sensors use electromagnetic waves in the range of 300 MHz to 300 GHz. The wavelength of electromagnetic waves is defined by the equation:

$$\lambda = \frac{c}{f}$$

where f [Hz] is the operating frequency and c [m/s] is the speed of light in vacuum, or less in other media. Consequently, microwaves have wavelengths ranging from 1 m to 1 mm, corresponding to their frequency range of 300 MHz to 300 GHz. For most practical applications, microwave wavelengths are neither small enough to be modeled as rays nor large enough to be treated as electrostatic or magnetostatic fields. As a result, full-wave analysis, which accounts for both electrical and magnetic fields, is necessary to accurately model microwaves and their interactions with matter [141].

The interaction of microwaves with materials is governed by the material's complex permittivity, $\varepsilon = \varepsilon' - i\varepsilon''$, and complex permeability, $\mu = \mu' - i\mu''$, assuming an $\exp(+j\omega t)$ time dependence. The real part of the complex permittivity, ε' , also known as the dielectric constant, represents the material's ability to align its electric dipoles when exposed to an external electric field. The imaginary part, ε'' , is related to the material's conductivity and is expressed as:

$$\varepsilon'' = \frac{\sigma}{\omega}$$

where σ [S/m] is the electrical conductivity and ω [rad/s] is the angular frequency of the wave. The permeability of the medium is similarly defined for magnetic fields and dipoles. In the microwave spectrum, materials are classified as insulators or conductors based on their permittivity. If $\varepsilon' \gg \varepsilon''$, the material behaves as an insulator, whereas if $\varepsilon' \ll \varepsilon''$, it behaves as a conductor [141].

2.4.4 Microwave Structures

In the design of microwave sensors, transmission lines and planar structures play a crucial role. Transmission lines are typically used in microwave circuits to transmit energy or information from one point to another or to couple energy between components. Early microwave systems primarily relied on waveguides and coaxial cables. Although waveguides are effective for high-power applications and minimizing losses, they are bulky and expensive. Planar transmission lines, introduced later, emerged as an alternative, offering configurations that are not only more suitable for microwave sensors and circuits but also easier and more cost-effective to fabricate. Examples of planar transmission lines include microstrip, stripline, coplanar, and slot lines. These structures provide several advantages, such as ease of integration, compactness, and low cost [152].

Transmission structures can be categorized as open, side-open, or closed. Open configurations are more susceptible to perturbations, while closed configurations are resistant to external fields. For applications requiring miniaturization, integrated planar structures are preferred. Conversely, conventional designs are better suited for high-power and high-Q-factor requirements. The development of integrated sensors and circuits has also been accelerated by advances in fabrication technologies, such as micro- and nanofabrication [153].

Each planar structure offers unique characteristics. For instance, microstrip lines generally have a higher Q-factor, suspended microstrip lines exhibit lower conductivity losses,

and coplanar waveguides (CPWs) provide superior integration capabilities and design flexibility. In microfluidic applications, planar structures such as coplanar strips, CPWs, and microstrip designs are frequently employed [154].

Microwave sensors operate through electromagnetic field interactions, which enable the extraction and analysis of material properties such as permittivity, permeability, and conductivity. For example, microwave sensing systems can convert the presence of analytes (e.g., biomolecules, cells, or viruses) into electronic signals. Over time, various microwave topologies and sensor structures have been developed to address specific objectives, each with its advantages and limitations [152].

Resonant structures are critical components of many microwave sensing devices because they enable the localization of high-field regions. At specific locations in a resonator, the electric and magnetic energy densities increase significantly at the resonance frequency, enhancing sensitivity and performance. These structures are particularly efficient within their designed frequency bands, as the signal-to-noise ratio improves with the resonator’s Q-factor. However, this increase in sensitivity is accompanied by a narrower frequency band, with the drop in amplitude depending on the Q-factor, which results from shifts in the resonance frequency caused by changes in the surrounding environment [155].

Microwave devices often employ resonant structures such as stripline and microstrip resonators, coaxial cavities, and coaxial lines. To achieve high sensitivity, a resonator sensor must exhibit a sharp frequency response, and the electric field must be confined to the region of the sensor where the sample substance is placed. While resonance features are preferred for such applications, resonators may not be ideal for wideband applications. Resonance-based methods, such as resonant dielectric spectroscopy, are widely used to obtain permittivity data at microwave frequencies, particularly for polar liquids. Resonators can be excited in multiple distinct modes to enable measurements at discrete frequencies across the permittivity spectrum. Compared to broadband techniques, this approach offers greater accuracy and reduced uncertainty [156].

Dielectric spectroscopy has proven valuable across various scientific and engineering disciplines [157]. It provides a straightforward electronic means of collecting information about the subject under investigation without requiring labeling, chemical modification, or physical intrusion. The permittivity of a material is a frequency-dependent property that describes its interaction with an electric field. The real part of permittivity indicates the material’s ability to polarize, while the imaginary part reflects the power dissipated by molecular alignment in the field. At microwave frequencies, the specific dipolar relaxations of molecules can reveal changes in liquid composition or environmental conditions [158]. Resonators allow measurements at discrete frequencies, offering improved accuracy

compared to broadband techniques.

Dielectric measurement techniques have been applied in various fields, demonstrating their versatility [159], [160]. Resonant techniques, in particular, have been extensively developed for microwave applications [161]. The ability to sense dielectric properties is especially significant in systems involving biomolecules, which exhibit distinct dielectric properties and can significantly alter the ionic conductivity of water. For example, tumor cells have been shown to have higher electrical conductivity and permittivity than normal cells, enabling label-free detection of target molecules in their native environment [162].

A prominent type of microwave sensor is the metamaterial-based split-ring resonator (SRR), first introduced by Pendry et al. in 1999 [163], [164]. SRRs made from artificially constructed materials have gained interest due to their unique electromagnetic properties. The resonance frequency of SRRs is highly sensitive to changes in capacitance, making them particularly well-suited for microwave sensing applications [165]. Research on SRRs has focused on topics such as flexible metamaterial structures [166], applications in biomolecule sensing [167], and their magnetic response properties [168].

2.4.5 Microwave Sensing Parameters

Several parameters are utilized in the microwave sensing method, including the resonance frequency, the amplitude of the scattering parameter at the resonance frequency, and the quality factor (Q-factor) of the resonance profile. At microwave frequencies, the complex dielectric constant of a material can be determined by measuring the shift in the resonance frequency and Q-factor of a microwave resonator when it is in contact with the material under test. These shifts are compared to the baseline measurements of the resonator itself. The difference between the measured resonance frequencies and Q-factors provides information about the real and imaginary parts of the material’s dielectric constant, respectively. Thus, accurate measurements of the resonance frequency and Q-factor are crucial. The resonance frequency can be identified from the scattering parameter diagram, where it corresponds to the extremum point [141].

Every resonant structure has a Q-factor, which characterizes its performance. The Q-factor is defined as the ratio of the energy stored to the energy lost per cycle under sinusoidal excitation. It is expressed as:

$$Q = 2\pi \frac{\text{Energy stored}}{\text{Energy lost per cycle}} = 2\pi \frac{E_{\text{mag,max}} - E_{\text{elec,max}}}{P_{\text{avg}}}$$

where $E_{\text{mag,max}}$ [V/m] and $E_{\text{elec,max}}$ [V/m] are the maximum magnetic and electric energies, respectively, and P_{avg} is the average power dissipated [141].

The Q-factor is highly dependent on the energy lost per cycle. As energy loss increases, the Q-factor decreases, necessitating the implementation of Q-enhancement mechanisms to improve resonator performance.

Microwave sensors offer several distinct advantages. They do not require physical contact with the object being measured, allowing for online measurements to be performed from a distance without interfering with the process. Additionally, microwaves penetrate all materials except metals, enabling volumetric measurements that account for the entire material rather than just the surface. The high contrast between water and most other materials makes microwave sensors particularly well-suited for water content measurements. Their stability is another key advantage, as the resonant frequency of microwave resonator sensors is directly related to their physical dimensions [169]. Furthermore, microwave sensors are less sensitive to environmental factors such as water vapor, dust (unlike infrared sensors), and high temperatures (unlike semiconductor sensors). At lower frequencies, the electrical properties of materials are often dominated by direct current (DC) conductivity, which strongly depends on temperature and ion content. In contrast, at microwave frequencies, the influence of DC conductivity is minimized, providing more reliable measurements. Moreover, the non-ionizing nature of microwaves makes them safe for use, and they do not alter the material being tested in any way. Microwave sensors also offer fast response times, making them suitable for dynamic applications [154].

Despite these advantages, there are limitations to consider. Higher frequency operation generally increases the cost of electronic components, except for specific frequency bands used in radar and other high-volume applications. Additionally, microwave sensors require separate calibration for different materials, limiting their universal applicability. For instance, a moisture sensor calibrated for one material cannot necessarily measure moisture in another material without recalibration. The sensors are often customized for specific applications, which reduces their versatility. Furthermore, they are sensitive to multiple variables, meaning additional sensors may be needed to compensate for factors that influence measurements. Finally, due to their relatively long wavelengths, microwave sensors have limited spatial resolution, which may restrict their use in applications requiring high precision [170].

These factors, both advantages and disadvantages, will be carefully considered in the context of the proposed thesis projects, which aim to optimize the use of microwave sensors for material measurements and sensing applications.

2.4.6 Microplastic Detection Using Microwave Sensing

The limitations of existing methods for MP detection, as discussed in the previous section, underscore the need for a solution that is accurate, fast, cost-effective, and capable of real-time on-site monitoring. Traditional methods such as FTIR and Raman spectroscopy are highly precise but are constrained by the need for centralized laboratory settings, extensive sample preparation, and susceptibility to environmental contaminants. Similarly, mechanical methods, while effective for pre-analysis separation, often lack scalability and field applicability. Electrical sensing methods, including impedance spectroscopy and dielectric spectroscopy, offer significant advantages in compactness, sensitivity, and versatility but still face challenges in scalability and handling the heterogeneous nature of MP samples.

Microwave sensing emerges as a promising alternative to overcome these challenges. This method exploits the dielectric and conductive properties of MPs, which differ significantly from those of natural environmental materials such as water and sediments. Compared to existing electrical sensing techniques, microwave sensors operate over a broader frequency range (typically 0.1 to 100 GHz), enabling more versatile and precise detection of MPs. For instance, resonance-based microwave spectroscopy provides high sensitivity to subtle changes in material properties, allowing for effective differentiation of MPs based on size, concentration, and type. Additionally, microwave sensing systems are cost-effective, with field-deployable devices like palm-sized nano-VNAs available at approximately \$80, making them accessible for widespread use. These devices also offer multiplexing capabilities and detect capacitance changes on the order of femtofarads, surpassing the sensitivity of conventional impedance and capacitance sensing methods.

Microwave sensing also addresses some of the limitations of mechanical methods. By eliminating the need for physical filters to sort particles, microwave sensors simplify the detection process and enhance scalability. Integration with planar technology enables the development of compact, lightweight, and robust systems that are easy to fabricate and operate. Moreover, the ability to separate the sensing tag from the reader reduces cost and operational complexity, providing flexibility for various practical applications.

2.4.7 Frequency Dispersion and Temperature Dependence of Polymer Dielectric Properties

The dielectric properties of polymers such as polyethylene (PE) and polystyrene (PS)—widely used as reference MP materials in this thesis—are known to exhibit weak frequency dispersion over the microwave frequency range (1–10 GHz), particularly in dry and low-loss

conditions. For example, the relative permittivity (ϵ') of PE typically ranges from 2.25 to 2.35, and that of PS from 2.4 to 2.6 in this range, with minimal change across frequency bands due to their non-polar molecular structure [171, 172].

Temperature, however, does induce mild changes in dielectric properties. As temperature increases, both ϵ' and dielectric loss ($\tan \delta$) may rise slightly due to enhanced molecular mobility, although this effect is generally small for rigid polymers in the 10–40°C range relevant to this study [173]. These baseline characteristics underpin the frequency shifts observed in experimental data and simulations presented in Chapters 4 through 6.

2.5 Microfluidic Systems for Environmental Monitoring and Microplastic Detection

2.5.1 Introduction to Microfluidic Systems

Microfluidic systems are devices that manipulate small volumes of fluid within microchannels, typically ranging from tens to hundreds of micrometers in diameter [174]. These systems offer significant advantages, including precise control over fluid dynamics, reduced sample consumption, and high throughput [175]. The ability to handle small fluid volumes allows for efficient processing, which is critical for applications across a wide range of fields, from chemical analysis to biomedical research [176, 177]. The integration of microfluidic technologies has enabled advancements in areas such as drug testing, diagnostics, and chemical synthesis, where control over fluid movement and mixing at small scales is essential [178, 179, 180].

A key feature of microfluidic systems is their ability to manage continuous flow, a process that enables efficient real-time monitoring and analysis. Continuous flow allows for the rapid transportation of fluids through the system, enabling the quick processing of samples without the need for complex sample preparation. This capability is particularly advantageous for applications requiring high precision and fast analysis [181]. Additionally, continuous flow in microfluidic devices facilitates the separation, concentration, and detection of various substances, improving the accuracy and sensitivity of these systems [182, 183, 184]. These features make microfluidic platforms well-suited for a broad spectrum of applications where time and sample efficiency are critical.

The compact design and integration of multiple functions in microfluidic systems also provide significant advantages for various practical applications. For instance, these systems can combine processes like sample pre-treatment, filtration, and analysis within a

single device [185, 186]. This reduces the complexity of traditional laboratory techniques, offering a streamlined and scalable approach for many fields. Their portability and low-cost production make microfluidic systems ideal for settings where traditional laboratory infrastructure may be unavailable or impractical, enabling their use in point-of-care diagnostics and field testing, among other areas [187].

2.5.2 Applications of Microfluidic Systems in Environmental Monitoring

Microfluidic systems have gained widespread adoption in environmental monitoring due to their ability to provide precise, efficient, and real-time analysis of contaminants [80]. These systems are particularly advantageous for detecting pollutants that are present in trace amounts or are difficult to analyze using traditional methods. Their compact size, low sample volume requirements, and ability to integrate multiple analytical functions into a single platform make them ideal for on-site and continuous monitoring. By enabling real-time, high-throughput analysis, microfluidics significantly improves the speed and accuracy of environmental monitoring, especially in complex and dynamic environments [188, 189, 190, 191, 192].

A key strength of microfluidic systems in environmental applications is their ability to integrate various sensing technologies, such as optical, electrochemical, and microwave sensors [193, 194, 195]. These sensors allow for the detection of a wide range of environmental pollutants, including organic compounds, heavy metals, and particulate matter. For example, electrochemical sensors integrated into microfluidic platforms can detect trace amounts of pollutants by measuring changes in electrical properties, while optical sensors provide data on the concentration of specific compounds by detecting their absorption or fluorescence characteristics. The combination of these diverse sensing methods within a microfluidic device offers a comprehensive approach for environmental monitoring, enabling the simultaneous detection of multiple pollutants from a single sample [80].

Beyond their ability to detect a variety of environmental pollutants, microfluidic systems also excel in providing non-invasive, low-cost, and scalable solutions [196]. The non-contact nature of certain sensing techniques, such as microwave and optical sensing, reduces the risk of sample contamination, making microfluidic devices suitable for on-field applications [107]. Moreover, the integration of multiple analytical processes—such as filtration, pre-concentration, and detection—into a single microfluidic platform reduces the need for complex laboratory procedures and enhances overall efficiency [187]. These features make microfluidic systems a promising technology for widespread deployment in environmental

monitoring, providing an effective and adaptable solution to address the growing need for real-time, on-field pollution detection.

2.5.3 Integration of Microfluidic Devices with Microwave Sensors

As discussed earlier, microwave sensing has proven to be an effective method for in situ MP detection, offering rapid, sensitive, and non-invasive analysis. The integration of microwave sensors with microfluidic devices enhances both the efficiency and accuracy of detection. Microfluidic platforms allow for the precise manipulation of small fluid volumes, enabling efficient concentration, confinement, and continuous flow processing of samples. The non-contact nature of microwave sensing also minimizes cross-contamination, making it ideal for sensitive applications in environmental monitoring [197].

Microwave-microfluidic systems have demonstrated significant versatility across various fields, including environmental and biomedical applications. These systems exploit the dielectric properties of MPs, which are distinct from the surrounding medium, such as water, allowing for precise detection. Studies have shown the feasibility of detecting and quantifying MPs in water using microwave-microfluidic systems, with particle sizes ranging from μm to 0.5 mm. This highlights the high sensitivity and reproducibility of such systems. Moreover, the non-destructive nature of microwave sensing, coupled with its integration into microfluidic devices, enables real-time, high-throughput analysis, which is critical for large-scale environmental monitoring [198, 199].

Recent advancements have focused on using resonant microwave sensors integrated with microfluidic devices for enhanced liquid characterization. These systems measure the dielectric properties of materials, enabling precise identification of the complex permittivity of materials under test (MUTs). Resonant structures, such as split-ring resonators, are often used due to their high sensitivity and compact design. However, challenges remain, such as temperature fluctuations that may affect measurement accuracy. These environmental factors necessitate robust calibration techniques to ensure reliable results. Despite these challenges, microwave-microfluidic systems continue to evolve, with innovations such as broadband sensors for wide-frequency dielectric analysis and enhanced sensitivity through interdigital capacitor structures [200].

Despite the significant progress in developing microwave-microfluidic systems, there are still hurdles to overcome before these platforms can be fully commercialized for real-world MP detection. Key challenges include scaling for larger sample volumes and optimizing sensitivity for trace-level detection of MPs. However, the potential of combining microwave

sensing with microfluidic devices remains substantial, offering scalable, cost-effective, and field-deployable solutions for diverse applications in environmental monitoring and biomedical diagnostics [140, 184, 195].

Chapter 3

Methodology: Design, Fabrication, and Experimental Setup for Microwave-Microfluidic Sensors

This chapter outlines the systematic approach undertaken for the development and characterization of the proposed microwave-microfluidic sensor for plastic detection in environmental samples. The methodology is divided into three key parts. First, we present the numerical simulation procedures implemented in Ansys HFSS to design and optimize the microwave sensor structure. This includes defining the problem, applying boundary conditions, and generating mesh grids to ensure accurate modeling of electromagnetic responses. Second, the fabrication process for both the microwave sensor and the microfluidic chip is described, detailing material selection and manufacturing techniques. Lastly, the experimental setup used for sensor characterization and testing is introduced; together, these sections establish a comprehensive framework for sensor design, fabrication, and performance evaluation.

3.1 Numerical Simulation in Ansys HFSS

This section introduces a numerical model that integrates both the physical components of the device and its operating parameters.

3.1.1 Problem Definition

Full-wave simulations are conducted using the commercial software package, Ansys HFSS. The microwave signal is introduced through a planar microwave reader, connected to a coaxial cable via an SMA connector (one-port) or a pair of SMA connectors (two-port configuration). To perform the simulations, the entire device is enclosed within a radiation boundary.

Energy dissipation typically occurs through three mechanisms: conductive, dielectric, and radiative losses. By analyzing the resonance frequency shift and power absorption within the material under test (MUT), key parameters are evaluated, including the dielectric properties of the microfluidic chip components (e.g., the permittivity of chip and substrate materials), passivation layer thickness, the presence of the MUT, microchannel geometry effects, and coupling effects within the sensor structure.

To optimize sensor performance, the reflection and transmission coefficients are analyzed, ensuring minimal conductive and dielectric losses while accounting for radiation losses, particularly due to the sensor's dimensions being comparable to the wavelength. For a highly sensitive sensor, a measurable resonance frequency shift is essential to detect subtle variations caused by the presence of the MUT.

3.1.2 Solution Process

Maxwell's equations govern the entire electromagnetic phenomenon, including boundary conditions and effects related to capacitance and inductance. These equations describe the relationship between the electric field ($\mathbf{E}$) and the magnetic field ($\mathbf{H}$) in the presence of materials characterized by electric permittivity (ϵ), conductivity (σ), and magnetic permeability (μ). The differential form of Maxwell's equations in the frequency domain is expressed as follows:

$$\nabla \times \mathbf{E} = -j\omega\mu\mathbf{H} \quad (3.1)$$

$$\nabla \times \mathbf{H} = (\sigma + j\omega\epsilon)\mathbf{E} \quad (3.2)$$

$$\nabla \cdot (\epsilon\mathbf{E}) = \rho \quad (3.3)$$

$$\nabla \cdot (\mu \mathbf{H}) = 0 \quad (3.4)$$

where ρ represents the density of free charges, j is the imaginary unit, and $\omega = 2\pi f$ is the angular frequency. As demonstrated by Maxwell's equations, electric and magnetic fields are coupled, meaning that electric fields can act as sources of magnetic fields and vice versa. This two-way coupling results in several effects, including radiation, wave propagation, and phase delays.

3.1.3 Boundary Conditions

Boundary conditions define the behavior of fields at the edges of problem regions and object interfaces. In HFSS, boundaries are used to construct both open and closed electromagnetic models, as well as to simplify the electromagnetic or geometric complexity of these models [201]. In a closed model, energy can escape only through the applied port, whereas in an open system, electromagnetic energy can radiate away. The model includes commonly used electromagnetic boundary conditions: Radiation (absorbing) boundary conditions to simulate open-space environments, Impedance boundaries to account for finite conductivity, and Perfect Electric Conductor (PEC) boundaries to represent ideal conductors.

In radiation boundary conditions, an open and absorbing boundary allows waves to radiate outward into space while simultaneously absorbing the outgoing waves. An absorbing boundary condition (ABC) is applied around the sensor domain to emulate an open region and minimize reflections from the edges of the simulation domain. This ensures proper modeling of radiated or scattered fields in open-space environments. This boundary region should be placed at least $\frac{1}{4}$ of the wavelength away from the radiation source, which, in this case, is the microwave sensor structure. Due to the radiation boundary, unphysical reflections are prevented, and the numerical solution appropriately truncates calculations at the radiation boundary domain for radiating and scattering structures. To clarify, outgoing waves are absorbed at the boundary, preventing their return and influence on the solution. Impedance boundaries are used to model imperfect (finite-conductivity) conductors, allowing the field solver to account for surface currents and skin depth effects without explicitly resolving the conductor volume. This boundary condition is valid when the skin depth of the microwave sensor electrodes is sufficiently high at the operating frequency band. The Perfect Electric Conductor (PEC) boundary condition represents an ideal, lossless conductor, where the tangential component of the electric field is zero.

Any structure defined as perfectly conducting is treated as a Perfect E boundary. Perfect E boundary conditions are applied to the microwave excitation through the SMA connector and coaxial cable.

3.1.4 Excitation

As a result of excitation, electromagnetic fields are introduced into the structure, where they interact with the components of the device. Various types of excitations can be applied, including wave ports, lumped ports, terminals, incident waves, voltage sources, and current sources. In this work, a wave port excitation is used, representing the external surface where microwave signals are introduced into the system.

Wave ports are suitable for implementing planar microwave structures. The cross-sectional surface of the coaxial cable is designated as the excitation port, with its size defined by the inner and outer radii of the shield. Simulations are performed with a source power of 1 W in all cases.

3.1.5 Mesh Generation and Operations

Ansys HFSS divides the geometric model into tetrahedral mesh elements as shown in Figure 3.1, forming a general mesh structure. Using a mathematical model based on Maxwell's equations, which govern electromagnetic phenomena, the equations are discretized in three-dimensional space within the problem region. The partial differential equations are then solved using the finite element method.

The HFSS approach divides the mesh into small three-dimensional tetrahedral and two-dimensional triangular elements. A tetrahedron, a solid with four triangular faces, forms a simplex in $\mathbb{R}^3$. This property allows any arbitrary three-dimensional (3D) domain to be decomposed into tetrahedrons. Consequently, the tetrahedral mesh elements can be stretched or adjusted to fill and decompose the 3D geometric model, offering flexibility and precise control over the mesh structure.

Adaptive mesh refinement is a key feature of this process and distinguishes HFSS from other electromagnetic simulators. After defining boundary conditions, excitations, and material properties, the geometry is iteratively decomposed using adaptive meshing. The process begins with an initial mesh, which is solved to estimate the error in the solution. Higher-error regions are then refined with finer meshes, and a new solution is generated to recalculate the error. This process is repeated until convergence is achieved. Each

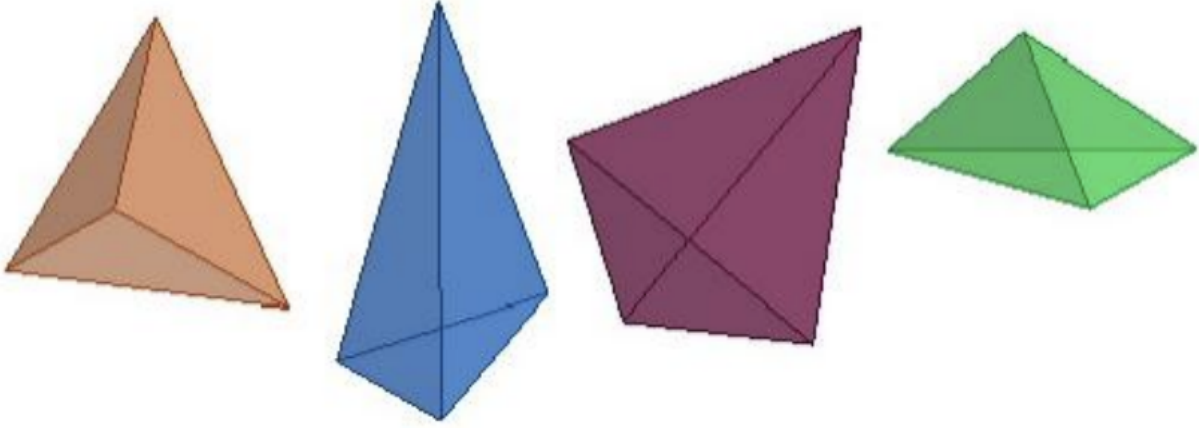


Figure 3.1: Different types of tetrahedral meshing elements.

iteration of this adaptive meshing process is referred to as an 'adaptive pass.' Adaptive mesh refinement ensures that regions with strong electromagnetic fields have finer meshes, while areas with weaker fields are represented by coarser meshes, resulting in improved computational efficiency.

A mesh refinement operation, known as seeding mesh, is an optional but valuable technique that allows engineering expertise to guide mesh construction in critical areas where precise electromagnetic field calculations are essential. Performing this operation before the adaptive passes begin provides the solver with an initial framework for refinement. This approach can significantly reduce the number of passes required for convergence and minimize the total number of tetrahedral mesh elements. As a result, more accurate results are obtained with less computational effort. While adaptive pass analysis can independently converge and identify critical electromagnetic fields, incorporating seeding mesh accelerates the process and improves accuracy in capturing these fields.

3.2 Microwave Sensor Fabrication

To fabricate the microwave sensor, the design must first be drawn using a commercial software package. In this work, QCAD was used to create the final design for fabrication. Once the design is completed, it should be printed on glossy paper. The printed design is then attached to a copper sheet and fused to it using heat lamination for 15–20 minutes.

Next, the process proceeds to the wet etching step, which is used to remove any un-

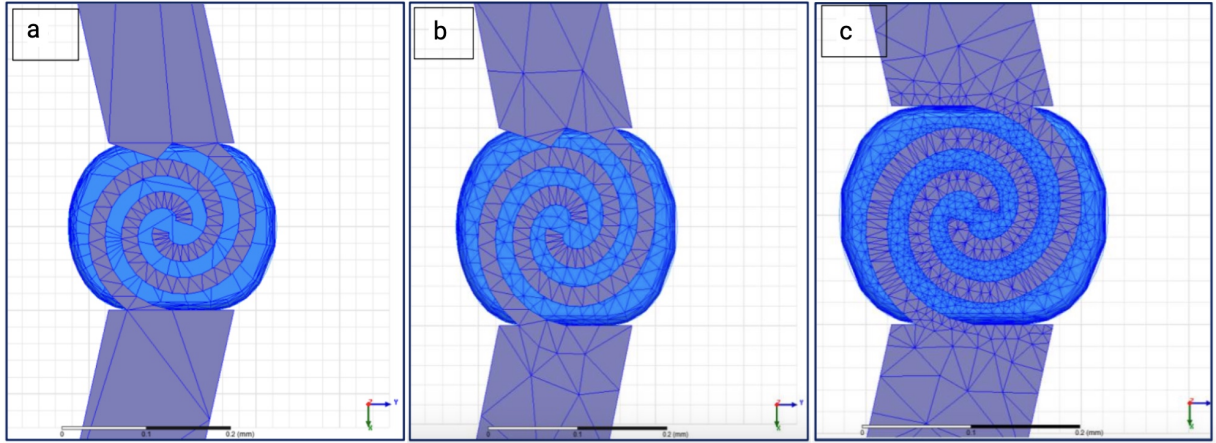


Figure 3.2: Mesh refinement across adaptive passes: (a) Coarse initial mesh without seeding; (b) Finer initial mesh with seeding; (c) Enhanced mesh refinement in high-field regions for improved convergence.

wanted copper elements from the sheet. According to the sensor's design, the "unwanted" copper elements are the regions not utilized in the final pattern. Wet etching ensures that only the desired design pattern remains on the copper sheet. Wet etching involves immersing the copper sheet in a chemical solution to dissolve excess copper. The etching rate during this process is influenced by the following factors:

- Etching chemical concentration
- Etching solution temperature
- Stirring of the etching solution

To improve the etching rate, the temperature of the etching solution should be increased, and stirring should be applied during the process. Copper wet etching can be performed using several chemical solutions. In this work, APS-100 Copper Etchant was used. APS-100 provides fine-line definition suitable for thin-layer copper sheets and is compatible with tin-lead solders.

The etching rate with APS-100 is approximately 8 nanometers per second when using the solution at 40 °C. For every 10 °C increase in temperature, the etching rate approximately doubles. Conversely, mixing one part deionized water with two parts etchant reduces the etching rate by approximately 50%. While temperatures between 20 °C and 80 °C are suitable, the most common operating range is 30 °C to 40 °C.

APS-100 Copper Etchant is composed of the following:

- 15–20 wt.% Ammonium Persulfate
- 80–85 wt.% Deionized (DI) Water

Wet etching offers several advantages over dry etching, making it a preferred method for many applications. It is more cost-effective and provides excellent surface uniformity, ensuring precise pattern transfer. The process is highly adaptable to various designs and demonstrates superior selectivity, which is particularly useful for precision etching of inner surfaces. Additionally, wet etching achieves higher etching rates compared to dry methods, while requiring simpler and easier-to-maintain equipment. Another key benefit is that it can be carried out under normal atmospheric conditions, further simplifying operational requirements.

After completing the wet etching process and transferring the final design onto the copper sheet, the SMA connector(s) should be soldered to the port(s) to enable external connections to the microwave circuitry. At this stage, the sensor is ready for experimental testing [202, 203].

3.3 Microfluidic Chip Fabrication

Two different fabrication approaches were employed for constructing the microfluidic channels, depending on the required channel dimensions. For microchannels larger than 50 μm , 3D-printed molds were used, while channels smaller than 50 μm were fabricated using a soft-lithography technique. Both methods utilize PDMS due to its flexibility, biocompatibility, and optical transparency, making it an ideal material for microfluidic applications.

3.3.1 Fabrication with 3D-Printed Molds

For microchannels larger than 50 μm , 3D-printed molds were utilized. The molds were designed using CAD software and fabricated with a Formlabs Form 3 resin-based 3D printer, employing Clear Resin (Formlabs). After printing, the molds were cleaned and post-cured to ensure dimensional accuracy and structural stability. PDMS was prepared by mixing it with a curing agent in a 10:1 ratio, degassed for 30 minutes, and poured over the 3D-printed molds. The molds were then placed on a hot plate and cured overnight at 85°C to allow complete polymerization. Once cured, the PDMS structures were carefully peeled

off the molds, and fluidic access holes were created using a biopsy punch. This method provides a scalable and flexible approach for fabricating larger microchannels.

3.3.2 Fabrication with Soft Lithography

A soft-lithography technique was employed to fabricate microchannels smaller than 50 μm . First, PDMS was mixed with a curing agent in a 10:1 ratio, degassed, and then molded against SU-8/silicon masters. The molds were cured for two hours at 95°C, cut out from the masters, and fluidic access holes were created using a biopsy punch. Next, the thin glass slide and PDMS chip were treated with oxygen plasma at 29.7 W and 500 mTorr for 30 seconds to ensure a leak-proof seal. A schematic representation of the fabrication steps for both approaches is provided in Figure 3.3 [2].

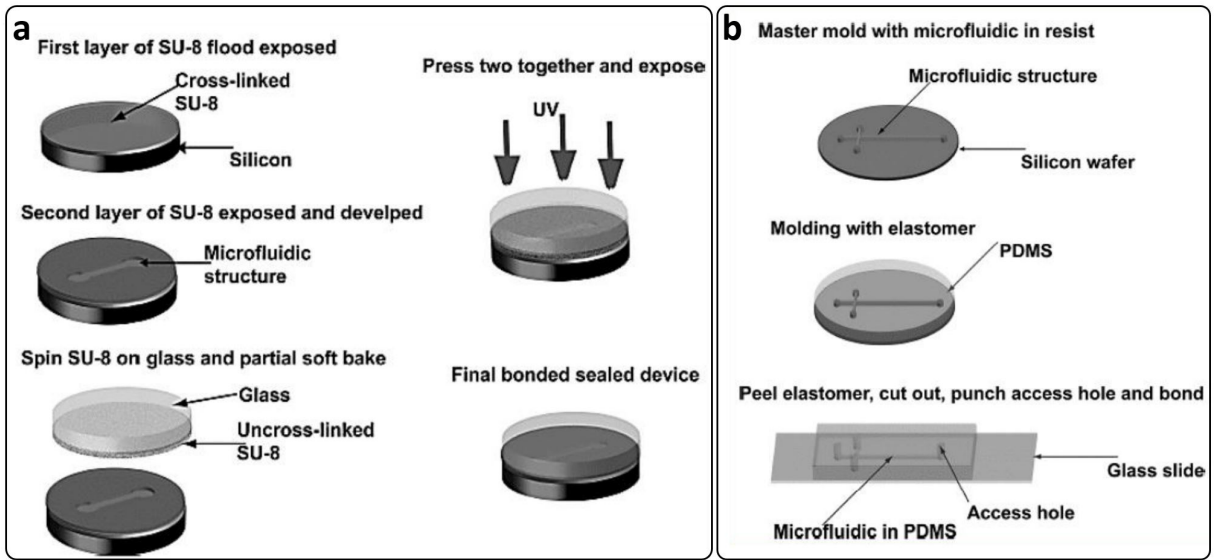


Figure 3.3: Fabrication process of microfluidic networks in PDMS elastomer using the soft-lithography technique. (a) Steps for fabricating SU-8 molds, including exposure, development, and bonding processes to create sealed microfluidic devices. (b) Replication of the microfluidic structures in PDMS from the SU-8 master mold, followed by elastomer curing, hole punching, and bonding to complete the device assembly [2].

3.4 Experimental for Characterization

The experimental setup comprises several integrated components designed to facilitate fluid handling, particle visualization, and frequency response monitoring. At its core, the system includes a microfluidic chip or micro-reservoir coupled with a microwave sensor, a syringe pump, and a vector network analyzer (VNA). Fluid flow is precisely controlled using a high-precision syringe pump (Harvard Apparatus, 33 Twin), with connections established through ETFE (ethyltrifluoroethylene) tubing (Tefzel, Upchurch Scientific).

For particle visualization, an digital microscope (ShenZhen Andonstar Technology Co., Ltd) equipped with a camera is employed. A Python-based program is implemented to manage system controls and provide a user-friendly computer interface. Frequency response monitoring is performed using two vector network analyzers: the MS2028C (Anritsu) and the M5090 (Copper Mountain Technologies).

Chapter 4

Detection of Microplastics by Microfluidic Microwave Sensing: An Exploratory Study

4.1 Overview

This chapter includes work that has been published in the following peer-reviewed article. The paper is the result of a collaboration between the Waterloo Microfluidics Lab and the Waterloo Ecohydrology Group. The Ecohydrology Group primarily contributed to the environmental aspects of the project. Maziar ShafieiDarabi and Pei Zhao are co-first authors of the paper. Dr. Zhao, a former postdoctoral researcher in the Waterloo Microfluidics Lab, initiated the project. Maziar later took the lead, carrying out the simulations, experiments, writing, figure updates, and manuscript revisions.

Maziar ShafieiDarabi, Pei Zhao, Xinyao Wang, Stephanie Slowinski, Shuhuan Li, Zahra Abbasi, Fereidoun Rezanezhad, Phillippe Van Cappellen, Carolyn L. Ren, "Detection of Microplastics by Microfluidic Microwave Sensing: An Exploratory Study," *Sensors and Actuators A: Physical*, Volume 383, 2025, Article 116154, ISSN 0924-4247.

Detection of microplastics in water environments and consumables is essential to evaluate the abundance, sources, transport pathways, degradation, and exposure risks of these emergent contaminants. In this study, we explore the potential of detecting microplastics in aqueous samples using a microwave sensor integrated in a microfluidic platform. The principle relies on the change in permittivity of the water due to the presence of microplastics that, in turn, leads to a resonance frequency shift recorded by the microwave sensor.

The method is tested using 20 and 70 μm polyethylene microspheres. Both the experimental data and the numerical simulations show consistent dependencies of the frequency shift on the size and concentration of the microspheres, as well as on temperature. However, the experimental trends in resonance frequency are not as pronounced as predicted by the numerical simulations. In addition, the limits of detection of the current microwave-microfluidic device are much higher than the typical particle abundances encountered in most freshwaters. Based on these preliminary results, we outline potential directions for the further development of microfluidic microwave sensing of microplastics in water samples.

4.2 Introduction

Concept of microplastics was first reported by Thompson et al. [3] in 2004 with evidence showing the widespread distribution of small plastic fragments and fibers in the oceans. Since then, microplastics have drawn ever-increasing attention from academia, industry, and government agencies [4]. Microplastics are roughly defined as tiny plastic particles with a nominal size of less than 5 mm that are ubiquitous in oceans, freshwaters, wastewaters, biota, and soils [3, 4, 5, 6]. Microplastics originate from textiles, tires, city dust, cleaning, or personal care products and from the breakdown of larger single use plastic products. Due to their small size, microplastics can easily enter the food chain through various means, including uptake by aquatic organisms and plant roots. Microplastics are recognized as a major threat to aquatic life and could pose risks to human health as well, because people may ingest and inhale more and more microplastics from consumables and ambient air [11, 12]. Evidence shows microplastic pollution has become a global environmental problem due to increasing plastic production and the associated amounts of mismanaged plastic waste [204, 205]. In the Sustainable Development Goals (SDGs) of the United Nations' 2030 Agenda, reducing the amount of plastic and microplastic waste is an important indicator for controlling global pollution [206].

Detection and identification of microplastics is a critical step in managing the growing issue of microplastic pollution because it enables to: i) delineate the spatial and temporal distributions of microplastics in the environment, biota, air, food, and source waters; ii) inform risk assessments of exposure to microplastics, iii) identify the sources and reconstruct the transport pathways of microplastic pollution; and iv) propose strategies to monitor the effectiveness of mitigation efforts [28, 29, 30, 31]. A number of detection methods have been applied to the analysis of microplastics, including visual counting, Raman spectroscopy, hyperspectral imaging, X-ray photoelectron spectroscopy, Fourier transform infrared spectroscopy (FTIR), digital holography, scanning electron microscopy (SEM),

and thermal analysis [29, 30, 34, 207, 208, 209, 210, 211]. Each of these methods has advantages and limitations. For microplastics smaller than 200 μm , visual observation by optical microscopy is not always reliable and may cause inaccurate microplastic quantification [34]. Methods such as FTIR, X-ray, and Raman spectroscopy have been used to determine microplastics abundances down to parts per million (ppm) resolution [212]. However, these techniques are time consuming and laborious, sensitive to human subjectivity, and mainly operated in centralized labs due to the need for advanced equipment, complex sample preparation procedures, and complicated data analyses, which are also expensive [53, 54]. Thermal analysis-based techniques are typically considered destructive involving removing organic and inorganic particles from the sample to isolate the microplastics.

The size and morphology of microplastics cover wide ranges and include microbeads, synthetic fibers, fragments of plastic waste and tires, and many more. The degradation of macro- and micro-plastic material in the environment is influenced by many factors as well, which challenges the quantitative analysis of microplastics and obtain statistically meaningful information. While advancements in machine learning are increasingly used in the automatic identification of microplastics [213, 214], they are based on the data obtained using sophisticated equipment, e.g., Raman and FTIR spectroscopy methods, and may need quite long computing times (e.g., days) [44]. Hence, efforts are underway towards developing new approaches that combine automation and high-throughput screening, in order to address the above-mentioned limitations and advance microplastics detection technology [54, 215]. Recent advancements in sensing technology have enabled the development of a novel platform capable of distinguishing between microplastics and other environmental particles, such as organic or biological matter, by integrating impedance cytometry and microwave sensing [134]. In this study, we investigate the feasibility of employing microwave sensing integrated in a microfluidic platform as a potential rapid, easy, and labor-saving microplastic detection method in aqueous samples. Microwave sensing is based on the interaction between an applied electromagnetic field and the permittivity plus conductivity of a given sample, here water with suspended microplastics particles. Microfluidics involves the manipulation of small quantities of fluids at the microscale, typically nanoliters to microliters. The proposed technique, which combines microwave sensing and microfluidics, offers unique advantages such as high sensitivity, custom fabrication, low cost, multiplexing, high-throughput and portability, and has been proven to be a reliable approach for characterizing liquid samples including biochemical solutions, dairy products and heavy metal ions in water [68, 69, 70, 71, 72, 216, 217, 218, 219, 220, 221, 222, 73, 74, 75]. However, this technique has rarely been applied for the detection of microplastics. Here, we present results of a first exploratory assessment of combining microwave sensing and microfluidics for the rapid and quantitative detection of microplastics in water.

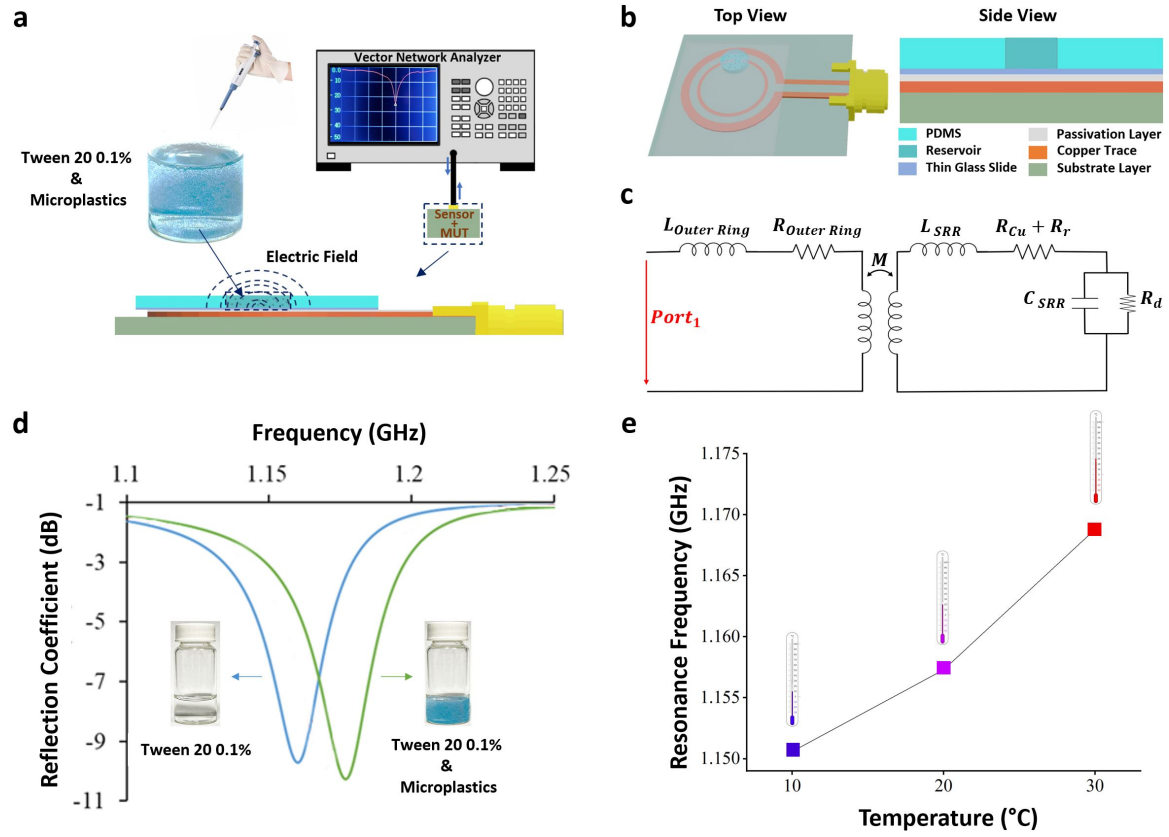


Figure 4.1: Microfluidic-microwave sensing system for microplastics detection: (a) schematic representation of the sensing system including the microchip integrated with a micro-reservoir and a microwave sensor and connected to the vector network analyzer (VNA); (b) top and side view of the microfluidic chip; (c) lumped element model of the structure, where $L_{Outer\ Ring}$ and $R_{Outer\ Ring}$ represent inductance and total resistance (including conductive and radiative losses of the excitation loop), the inner split ring resonator (SRR) exhibits an inductance L_{SRR} , a resistance R_{Cu} (conductive losses), a resistance R_r (radiation), a capacitance C_{SRR} (gap), and a resistance R_d (dielectric loss), and M represents the electromagnetic coupling between the outer ring and the inner SRR [223, 224, 225]; (d) schematic showing the frequency shift in response to the presence of microplastics; and (e) example of results showing the effect of temperature on the sensing performance.

4.3 Materials and Methods

Microplastic suspensions were prepared with two diameter sizes of blue polyethylene microspheres (Cospheric LLC), 10-27 μm (average 20 μm) and 63-75 μm (average 70 μm). These microspheres were selected because (1) their color allows for easy visual monitoring of spillage and homogeneity of the suspensions, and (2) the uniform spherical shape simplifies their representation in model simulations. The 20-70 μm size range also overlaps with the dominant size range of microplastic particles found in bottled water, an area of heightened public concern [226]. For each particle size (70 and 20 μm), three concentrations were used: 10^5 (100k), 10^6 (1000k) and 10^7 (10000k) particles L^{-1} . While these concentrations are much higher than those typically observed in most aquatic environments, they enabled us to verify the proof of concept for microwave sensing of microplastics. Future efforts will focus on lowering the limit of detection (LOD) of the method (see section Where do we go from here?). To further prevent aggregation of the microspheres, a surfactant, Tween 20, was added to boiling deionized water to make a 0.1% Tween 20 solution [227]. This solution was poured into a glass vial containing the microspheres, which was then centrifuged for 5 min to ensure a uniform suspension. Sensing experiments were carried out in a temperature-controlled environmental chamber at 10°C, 20°C, and 30°C.

Initially, we used the AI-powered QWave system (QuantWave Technology, see Figure 4.7), which integrates microfluidic, high-frequency microwave, and machine learning technologies to capture unique liquid fingerprints. Originally developed for monitoring manufacturing processes in industries such as water/wastewater treatment and food production, the QWave system enables real-time detection and data collection. This innovative technology was adapted for our research to explore its potential in detecting microplastics in environmental samples. The latter has been used to reliably monitor wastewater quality properties, including conductivity and biochemical plus chemical oxygen demand, with microwave sensing integrated with a microfluidic chip [228, 229, 230]. Our preliminary results with the microspheres were not repeatable, however, because of the tendency of the Tween 20 surfactant to adhere to the microchannel and the tubing used for automated sample transport. This problem persisted even after extensive cleaning with the recommended combination of isopropyl alcohol (IPA), acetone, and water. In addition, accumulation on the tubing wall also altered the concentration of Tween 20 in the suspensions.

To address the above problem, we developed a new microchip that incorporates a disposable micro-reservoir positioned atop the microwave sensor, as shown in Figure 4.1 (also Figure 4.8 in the supplemental material). The microwave sensor design is the same as that in the QWave system, consisting of two concentric rings with the radius of the outer and inner rings being 9.75 mm and 7.25 mm, respectively. The micro-reservoir design is

simple, low-cost, and easy to replace. It comprises two substrates that are bonded via oxygen plasma treatment (29.7 W, 500 mTorr for 2 min). The top substrate is a 40mm-thick layer of Polydimethylsiloxane (PDMS) with a punch-through hole of 2mm in diameter resulting in a volume of around 125 μL that serves as the reservoir filled with the sample suspension prior to sensing. A sample volume of 100 μL was used for all the tests. The bottom substrate is a thin glass slide acting as solid support for the reservoir. The reservoir is aligned with the microwave sensor located beneath the glass slide and affixed to the glass slide with double-sided tape. The microchip and the microwave sensor are easily separated by removing the double-sided tape as shown in FigS2. In an experimental series with a given particle size and concentration, the reservoir was washed with IPA and water for three times between each test. When switching to another concentration or particle size, the reservoir was replaced by a new one.

The microwave sensor is fabricated by a wet chemical etching process. First, the microwave design printed on a glossy photo paper is laminated on a copper-coated polymer slide using a thermal laminator for 15 min to transfer the design to the copper-coated slide. The patterned slide is then immersed in a copper etching solution with agitation for 15 min to remove the coated copper except for the patterned region which is covered by the printed ink. Next, the ink is washed away using acetone resulting in the designed microwave sensor made of copper. Finally, a SubMiniature version A (SMA) connector (yellow component in Figure 4.1 a and b) is soldered to the microwave-microfluidic platform, so that the sensor can be linked to a vector network analyzer (VNA, MS2028C, Anritsu). The sensing principle is based on the changes in the permittivity and conductivity of the microplastics suspension due to changes in the microplastics concentration and size plus the temperature. These changes in the permittivity and conductivity of the suspensions in turn cause shifts in the resonance frequency and the magnitude of the resonance frequency, respectively. For the detection of microplastics, the permittivity is a more sensitive variable than the conductivity because the conductivity of the microspheres is low compared to that of the Tween 20 0.1% background solution. That is, the imposed changes in microsphere concentration and size did not cause detectable changes in conductivity and, hence, in the magnitude of the resonance frequency. In contrast, we were hopeful to observe detectable shifts in the resonance frequency due to the difference between the dielectric constants of the microplastics ($\sim 2\text{-}3$ at room temperature) and the Tween 20 0.1% solution (~ 80 at room temperature).

The resonance frequency spectra of the aqueous suspensions of microspheres were obtained by measuring the reflection coefficient (S11) over a range of frequencies (Figure 4.1 c) using a vector network analyzer (VNA) (MS2028C, Anritsu). S11 represents the amount of energy that is reflected back toward the source when a signal is sent through the device.

It is expressed as a complex number with magnitude and phase and is typically measured in decibels (dB). A high S11 value indicates that a significant amount of energy is being reflected back, while a low S11 value indicates that most of the energy is being transmitted through the device. For all the tests, the microsphere-free Tween 20 0.1% solution was used as the baseline against which shifts of the resonance frequency of the microsphere suspensions were determined.

The VNA was configured with a frequency resolution of 100 kHz, which represents the smallest detectable resonance frequency shift in our measurements. This resolution imposes a detection threshold, meaning that frequency shifts smaller than 100 kHz cannot be reliably measured. The sensitivity of the sensor, which describes the change in resonance frequency, (Δf) corresponding to the change in microplastic concentration (ΔC), can be defined as $S = \frac{\Delta f}{\Delta C}$ and is inherently limited by the VNA's resolution. In our study, for 70 μm particles, the sensitivity is approximately $S = \frac{390\text{Hz}}{1\text{kparticlesL}^{-1}}$. For the same concentration of larger particles, the sensitivity increases due to their stronger interaction with the electromagnetic field.

4.4 Numerical Simulations

To determine whether microfluidic microwave sensing has the potential to detect microplastics, we carried out full-wave electromagnetic simulations using the commercial software Ansys HFSS [231]. The system layout in the simulations was that of a sensor consisting of two planar concentric ring-shaped electrodes and a PDMS-made micro-reservoir of 100 μL , as in Figure 4.1 b. The outer ring was connected to a microwave source via a coaxial cable, and the inner ring, which was a split ring resonator, was electromagnetically coupled to the outer ring. The dimensions were kept the same as those in the QuantWave system. The PDMS micro-reservoir was located on top of the gap of the split ring resonator. The latter served as a capacitive discontinuity (1 mm) located below the PDMS micro-reservoir.

The dielectric constant (relative permittivity) of a microplastic suspension depends on the dielectric constants and volume ratios of both the microspheres and the Tween 20 0.1% solution:

$$\varepsilon_{\text{mixture}} = \frac{(\varepsilon_{\text{Microplastic}} \times V_{\text{Microplastic}} + \varepsilon_{\text{Tween 0.1\%}} \times V_{\text{Tween 0.1\%}})}{V_{\text{suspension}}} \quad (4.1)$$

where ε stands for the dielectric constant (-) and V for volume (L). This equation serves as a rough estimation to predict the direction of the resonance frequency shift when

microplastics are added to the host medium. However, for more accurate modeling of heterogeneous mixtures, the Maxwell-Garnett effective medium approximation [232] can be used:

$$\varepsilon_{\text{mix}} = \varepsilon_h \left(1 + 3f \frac{\varepsilon_p - \varepsilon_h}{\varepsilon_p + 2\varepsilon_h - f(\varepsilon_p - \varepsilon_h)} \right) \quad (4.2)$$

Here, ε_{mix} is the effective permittivity of the suspension, ε_h is the permittivity of the host medium (e.g., Tween 20 solution), ε_p is the permittivity of the microplastic particles, and f is the volume fraction of the dispersed particles.

The dielectric constant of the Tween 20 0.1% solution at 10, 20 and 30°C (from low to high temperatures) was measured directly with a coaxial dielectric probe kit from Keysight (N1501A). Based on the density of the microplastics (1 gcm^{-3} , Cospheric LLC) the dielectric constant of the microplastic spheres was estimated to be in the range 2.3-2.5 which is within the values reported for High-Density Polyethylene (HDPE) [233]. The average value of 2.4 was used in the simulations. The dielectric constants of the microsphere suspensions calculated with Equation (1) for 10°C are presented in Table 1 (for 20°C and 30°C are presented in Table S1 and Table S2, respectively). The numerically simulated resonance frequencies for the suspensions at 10°C are listed in Table 2 (the results at 20°C, and 30°C are listed in Table S3, and Table S4, respectively). The frequency shifts relative to the Tween 20 0.1% background solution at 10°C can be found in Table 3 (the results for 20°C and 30°C can be found in Table S5 and Table S6, respectively).

The limit of detection (LOD) is a critical parameter in evaluating the sensor detection capability. According to the numerical simulations shown in Figure 4.2, at 10°C the sensor should be able to detect 70 μm microsphere suspensions with concentrations $\geq 1000k$ particles L^{-1} , that is, concentrations generating frequency shifts exceeding 400 kHz. At lower particle concentrations, suspension frequencies should barely be distinguishable from the pure Tween 20 0.1% solution.

The simulation results for frequency shifts of microspheres ranging in size from 20 to 300 μm , concentrations from 0 to 10000k particles L^{-1} , and at temperatures of 10, 20 and 30°C are shown in Figure 4.3. As expected, the plots imply that the frequency shift increases with increasing concentration of the microspheres because this would result in a decrease in the dielectric constant of the suspension (Table 1). When the microsphere size increases, the dielectric constant of the sample solution also decreases, resulting in a larger frequency shift and a lower LOD. For example, at 10°C, the LOD for 300 μm microspheres is 10k particles L^{-1} , compared to 10000k particles L^{-1} for the 20 μm microspheres at the same temperature.

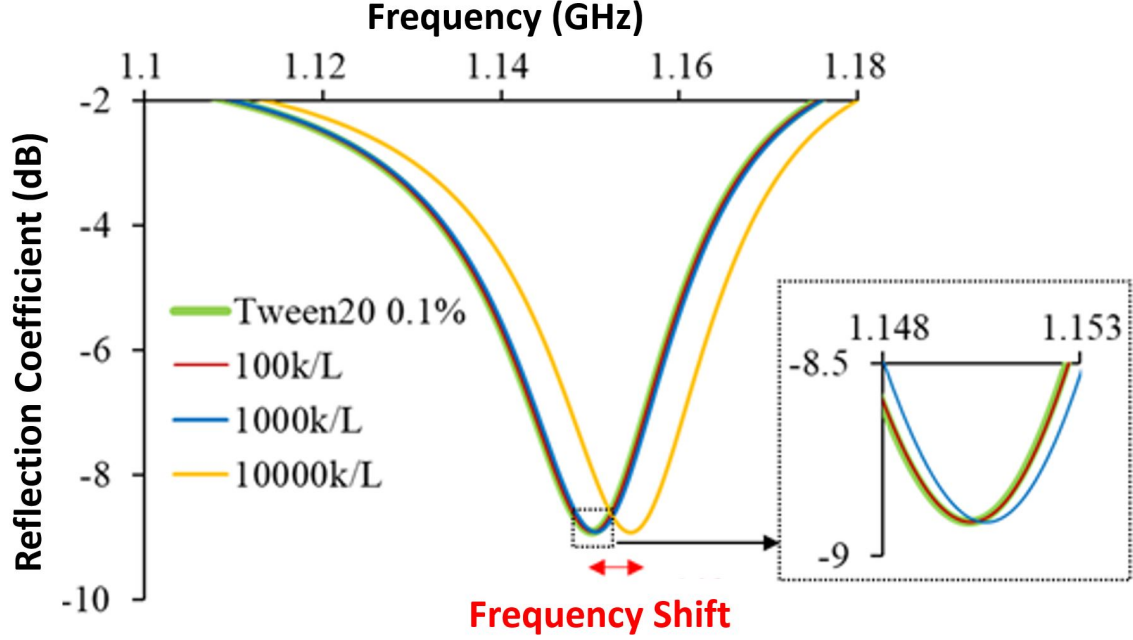


Figure 4.2: Numerically simulated S11-frequency plots for the samples with particle size of $70 \mu\text{m}$ at 10°C , and for concentrations of 0, 100k, 1000k, and 10000k particles L^{-1} .

Table 4.1: Dielectric constants of the microsphere suspensions with varying particle size and concentration calculated with Equation (1). The table provides the values at a temperature of 10°C . The values at 20 and 30°C can be found in Tables 4.6 and 4.9.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
Dielectric constant at 10°C						
0	83.0540	83.0540	83.0540	83.0540	83.0540	83.0540
100	83.0540	83.0540	83.0540	83.0540	83.0537	83.0531
1k	83.0540	83.0540	83.0539	83.0537	83.0514	83.0452
10k	83.0540	83.0537	83.0529	83.0507	83.0279	82.9661
100k	83.0537	83.0510	83.0428	83.0214	82.7934	82.1746
1000k	83.0514	83.0243	82.9423	82.7283	80.4484	74.2602
10000k	83.0279	82.7572	81.9369	79.7970	56.9984	-

Table 4.2: Magnitudes of resonance frequencies simulated with HFSS for microsphere suspensions with varying sizes and concentrations at 10°C. Corresponding values at 20 and 30°C can be found in Tables 4.7 and 4.10.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Resonance frequency at 10°C (GHz)					
0	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
100	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
1k	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
10k	1.1506	1.1506	1.1506	1.1506	1.1507	1.1509
100k	1.1506	1.1506	1.1506	1.1507	1.1515	1.1537
1000k	1.1506	1.1507	1.1510	1.1517	1.1598	1.1838
10000k	1.1507	1.1516	1.1545	1.1621	1.2702	-

Table 4.3: Frequency shifts of the microsphere suspensions with varying sizes and concentrations at 10°C simulated with HFSS. Corresponding values at 20 and 30°C can be found in Tables 4.8 and 4.11.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Frequency shift at 10°C (MHz)					
0	0	0	0	0	0	0
100	0	0	0	0	0	0
1k	0	0	0	0	0	0
10k	0	0	0	0	0.1	0.3
100k	0	0	0	0.1	0.9	3.1
1000k	0	0.1	0.4	1.1	9.2	33.2
10000k	0.1	1	3.9	11.5	119.6	-

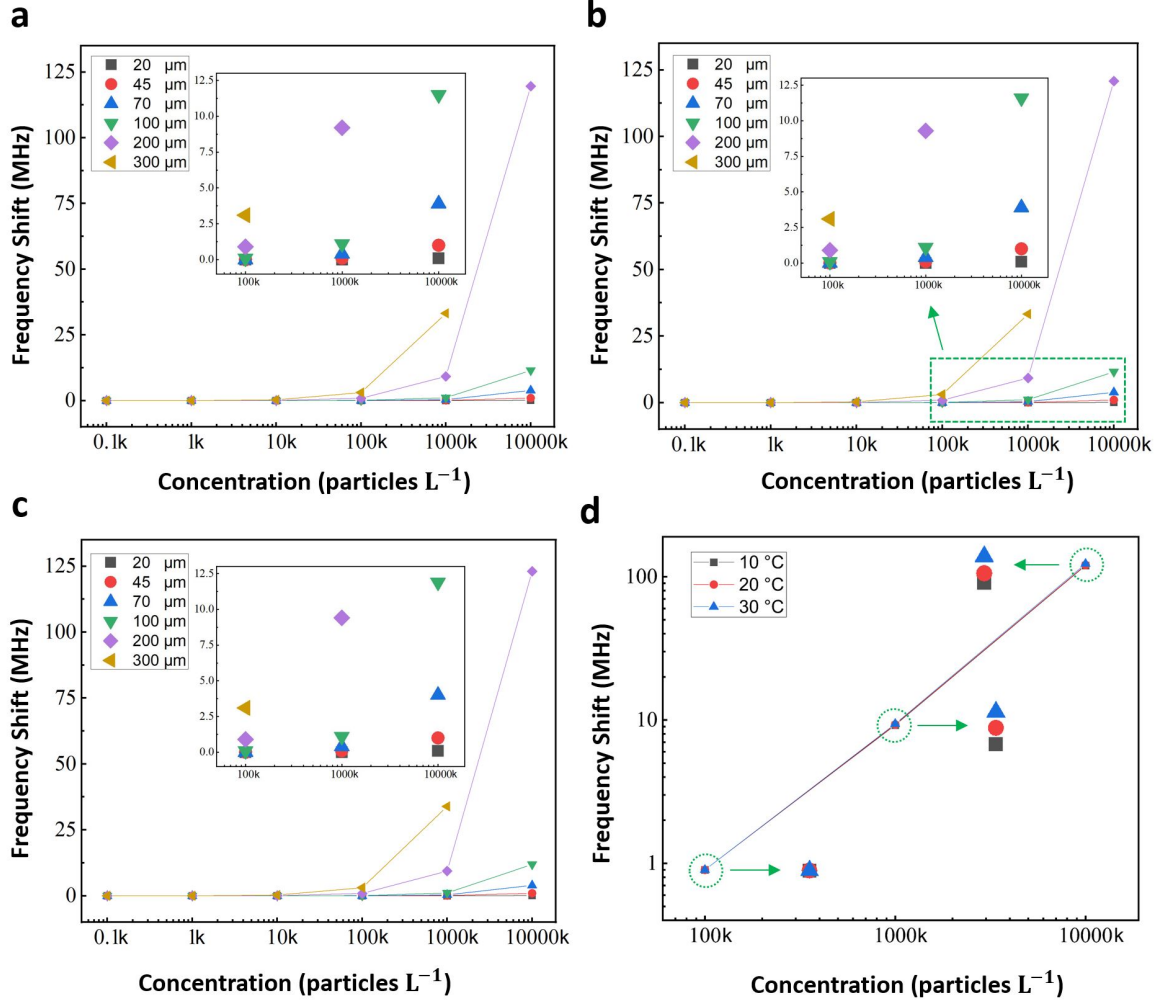


Figure 4.3: Model-predicted frequency shifts as a function of particle concentration for suspensions with particle size of 20-300 μm and concentration of 0-10000k particles L^{-1} at a temperature of 10°C (a), 20°C (b), and 30°C (c). Panel (d) compares the results for the 200 μm particles at the temperatures of 10, 20 and 30°C. (Note: The frequency shifts for particles larger than 200 μm at a concentration of 10,000k particles L^{-1} are significantly larger compared to other data points and they have been excluded from the plots due to their exceptionally large magnitudes, which would make it challenging to effectively visualize them alongside other data points.)

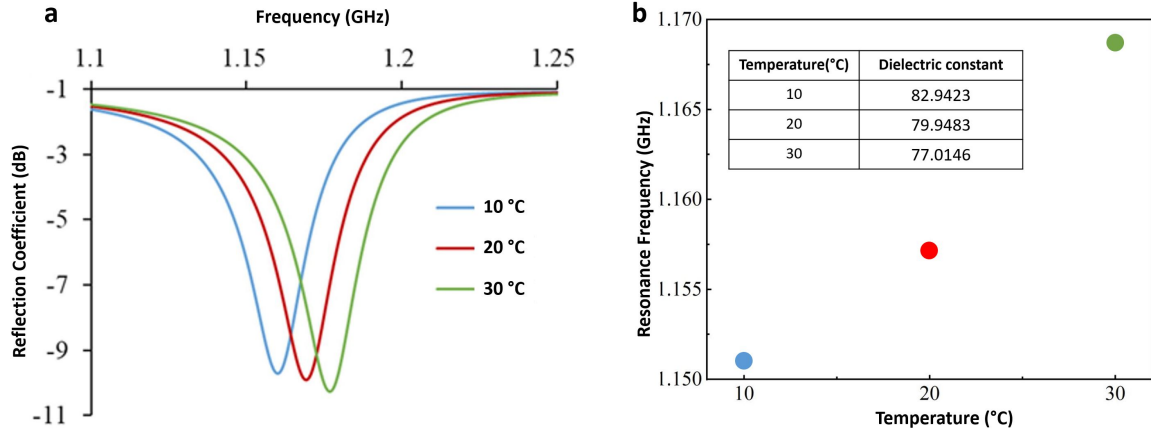


Figure 4.4: (a) The model-generated S11 curves for the 70 μm particles at a concentration of 1000k particles L^{-1} over three different frequency and (b) the effect of temperature on the resonance frequency.

The predicted effect of temperature on the sensing performance can be seen in Figure 4.3 d, for the 200 μm microplastic particles: detectability increases with increasing temperature. For example, for 10000k particles L^{-1} , the simulated frequency shifts at 10, 20, and 30 °C are 119.6, 120.8, and 123 MHz, respectively. However, as shown in Table 4.3, Table 4.8, and Table 4.11, for a suspension of 200 μm microspheres at a lower concentration of 100k particles L^{-1} , the frequency shifts at the three temperatures would be around the same, at 0.9 MHz. Therefore, the effect of temperature is relatively limited for the smaller particles and, especially, at the lower concentrations tested. The simulated effect of temperature on the S11 curve of the suspension of 70 μm microspheres at a concentration of 1000k particles L^{-1} is shown in Figure 4.4. When the suspension temperature increases, the dielectric constant decreases, resulting in an increase in the resonance frequency (see inset in Figure 4.4 b). Similar upward trends are seen for the other suspensions with varying particle sizes and concentrations indicating the importance of controlling or recording temperature when carrying out the measurements. Furthermore, for ex situ analyses, the sensitivity of the method could, in principle, be tuned by adjusting the temperature of the measurements. Nonetheless, the model-predicted LODs of the method are much higher than microplastics number concentrations reported for most freshwaters and tap water [234], hence, limiting the direct in-field use of the microwave-microfluidic platform at this stage of development.

4.5 Experimental Results and Discussion

The measurement process for detecting microplastics involved filling the reservoir of the microfluidic chip with the prepared suspension and then measuring the reflection coefficient (S11) using the VNA. After each measurement, the microfluidic chip was thoroughly cleaned with IPA and deionized water to prevent cross-contamination between samples. The cleaning procedure was repeated three times to ensure accuracy. The experiments were conducted in a temperature-controlled chamber, with measurements performed at 10°C, 20°C, and 30°C to assess the impact of temperature on resonance frequency. Proper temperature control was maintained to minimize its effect on the measurement results. The microwave sensor's resonance frequency was determined by analyzing the reflection coefficient (S11) spectra across a range of frequencies. The Tween 20 0.1% solution used in the suspensions was essential for preventing particle aggregation, ensuring that the microspheres remained uniformly distributed throughout the suspension. These steps were meticulously followed to ensure consistency and reproducibility in the measurements.

The reflection coefficient (S11) data obtained experimentally in the microfluidic platform were compared to the simulation-predicted resonance frequency shifts presented in the previous section. For example, the measured S11 spectra for the 70 μm particles at a concentration of 1000k particles L^{-1} in Figure 4.5 show that, as expected, the resonance frequency shifts to the right (i.e., shifts to higher values) with increasing temperature, because of the decreased dielectric constant. The same trend was observed for the suspensions with other concentrations and when 20 μm microspheres were used.

Based on the experimentally measured S11 spectra, resonance frequencies and frequency shifts were determined, and used thereafter to differentiate suspensions with different concentrations and sizes of microplastics. In this study, microsphere suspensions with diameters of 20 μm and 70 μm were measured by microwave sensing over a concentration range of 0-10000k particles L^{-1} and at 10, 20, and 30 °C (Figure 4.6). The VNA can reliably detect a frequency shift above 0.1 MHz as indicated by the dotted lines in Figure 4.6. Unsurprisingly, it was easier to detect the larger particles (i.e., 70 μm) than the smaller ones (i.e., 20 μm). For example, a LOD of 1000k particles L^{-1} is observed for the 70 μm particles over the temperature range of 10°C - 30°C. The LOD, however, increases to 10000k particles L^{-1} for the 20 μm microplastics, as seen in Figure 4.6 (a-c).

Both the experimental and simulation results imply a positive correlation of the frequency shift with temperature. However, they also indicate that this temperature effect is relatively small between 10 and 30°C. For example, the experimental results in Figure 4.6 d show that the effect is almost undiscernible for concentrations below 10000k particles

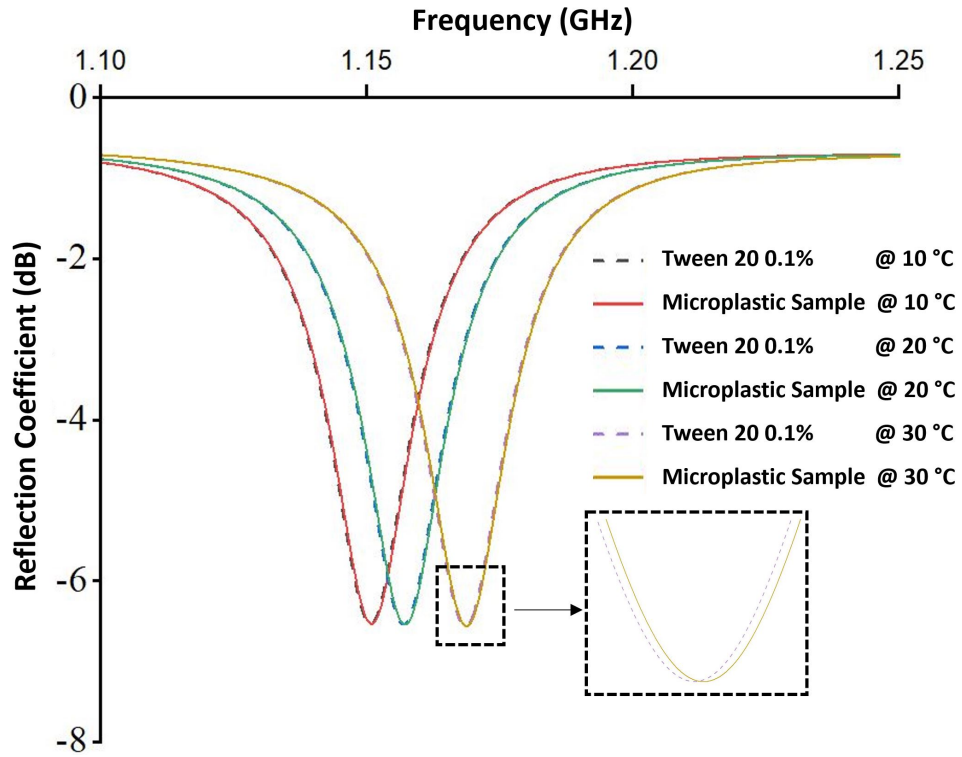


Figure 4.5: Experimental frequency spectra of the Tween 20 0.1% solution and the 70 μm microsphere suspensions of 1000k particles L^{-1} at 10, 20, and 30°C.

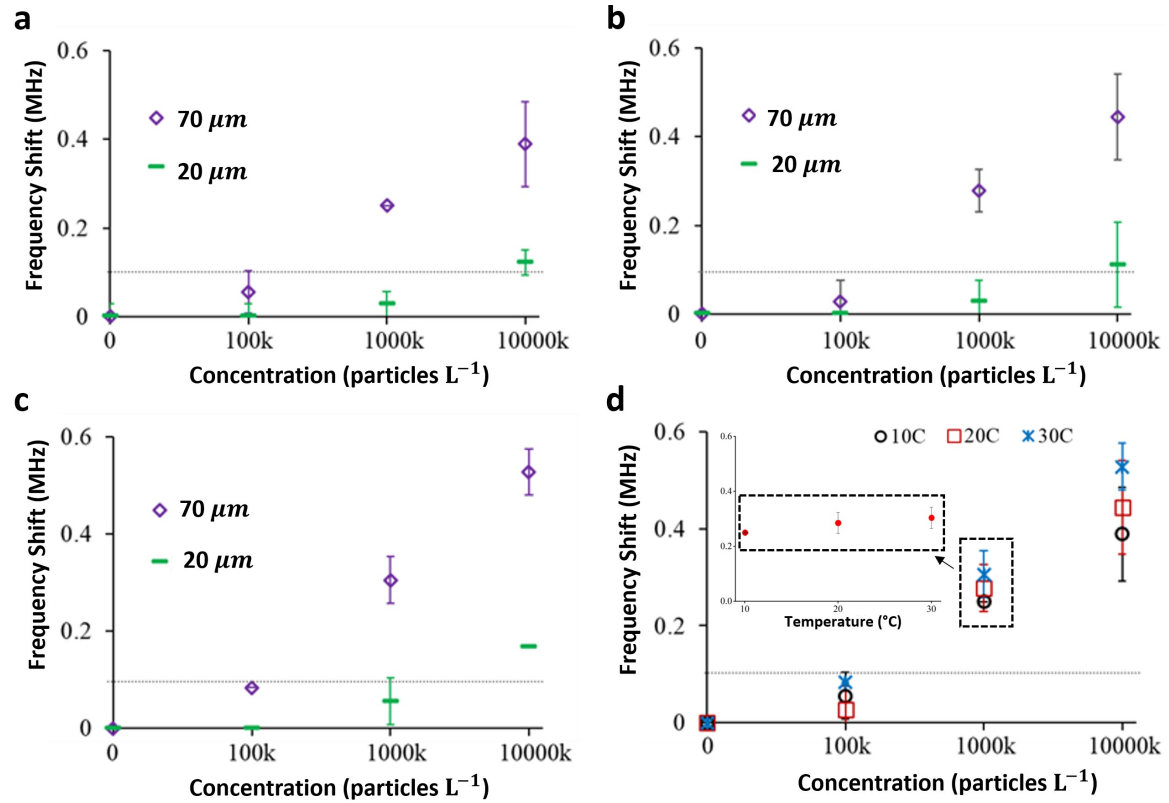


Figure 4.6: Experimentally measured frequency shifts over the concentration range 0-10000k particles L^{-1} for suspensions with particle sizes of 20 and 70 μm , at (a) 10 $^{\circ}C$, (b) 20 $^{\circ}C$ and (c) 30 $^{\circ}C$, and (d) for the 70 μm microsphere suspensions at 10, 20, and 30 $^{\circ}C$.

L^{-1} . For 10000k particles L^{-1} , the frequency shift is 0.39, 0.44 and 0.53 MHz for 10, 20, and 30°C, respectively. The reason for this weak temperature dependence is the high dielectric constant of the background Tween 20 0.01% solution, which masks changes in the dielectric constant when microplastics are added.

The directions of change of the frequency shift with changing microsphere concentration and size are the same for the experimental and numerical results. That is, the frequency shift increases with increasing particle size and with increasing concentration. Larger microplastics should therefore be easier to detect because of lower LODs. For example, as shown in Table 4.4, at 30°C, the 70 μm and 20 μm microspheres can be detected (i.e., they yield a frequency shift larger than 0.1 MHz) when the concentrations exceed 1000k and 10000k particles L^{-1} , respectively. Nevertheless, the frequency shifts predicted by the simulations generally exceed those obtained experimentally. For example, the predicted frequency shift for 70 μm microspheres at a concentration of 10000k particles L^{-1} is 4 MHz, compared to 0.5 MHz observed in the experiments.

The discrepancy between the numerically predicted and experimentally measured resonance frequency shifts is contributed by several factors. First, the VNA-cable-sensor system in the simulation is assumed to be ideal, which does not reflect the conditions of the actual experiments. The resolution of the VNA is limited to 100 kHz, constraining the smallest detectable resonance frequency shifts and introducing system noise into the measurements. Second, the idealized model configuration in the simulations may not fully capture the actual (i.e., physical) microwave-microfluidic system used in the experiments. For instance, the dielectric constant of the chip materials, such as PDMS, glass, the sensor substrate and the passivation layer, considered in the simulations are best estimates rather than directly measured values. Additionally, the estimated dielectric constants of the microplastic suspensions may not be accurate. While the dielectric constant of the background Tween 20 0.1% solution was measured with high precision using a VNA and probe, however, the dielectric constant of the microplastic particles was not measured because the measurement requires a large volume and the microplastic particle solutions are very expensive, instead, was estimated based on the density provided by the supplier and values reported in the literature. Lastly, temperature fluctuations (ranging from 10°C to 30°C) also caused detectable shifts in resonance frequency, further contributing to the overall uncertainty in the results.

Table 4.4: Simulated and experimental frequency shifts of microsphere suspensions of 20 and 70 μm and concentrations ranging from 0 to 10000k particles L^{-1} at 30°C.

Particle diameter (μm)	20 μm		70 μm	
	Frequency shift (MHz) at 30°C			
	Simulation	Experiment	Simulation	Experiment
0	0	0	0	0
100k	0	0	0	0.083333
1000k	0	0.027778	0.4	0.305556
10000k	0.4	0.122222	4	0.527778

4.6 Where Do We Go from Here?

In this study, the feasibility of using a microwave sensor integrated with a microfluidic platform for the quantitative detection of microplastics is explored using numerical simulation and experimental measurements. The method relies on the resonance frequency shift between a suspension containing plastic microspheres and that of the background solution (Tween 20 0.1%). The experimental trends are consistent with those of the numerical simulations with respect to the effects of particle size, concentration, and temperature. Larger particles at a higher concentration and higher temperature can be detected with greater confidence.

At the present stage of development, however, the method’s LOD remains too high for practical applications. Future research will therefore focus on reducing the LOD. Several avenues will be pursued to increase the performance of microfluidic microwave sensing of microplastics, including further improvements in the microwave structure and microfluidic platform, upgrading the sensor material, pre-concentrating the microplastic particles, and enhancing the contrast in dielectric constant in the presence of microplastic particles. Ultimately, the sensor system could be combined with portable measurement circuitry to create a multi-purpose field-deployable sensing system suitable for various applications.

One crucial determinant affecting the method’s LOD is the measurement equipment employed in the experiments. The Anritsu MS2028C utilized here is limited to 4000 data points for the full frequency range (5 kHz to 20 GHz) and to 1500 data points within a narrower frequency range of 150 MHz (as done in this study), resulting in a resolution of 100 kHz. Given that the resolution within a specific frequency range span is contingent upon

Table 4.5: Comparative analysis of sensing technologies based on size range, size and concentration estimation capabilities, and environmental factor consideration.

Applied Design Method	Size Range	Estimation Capability		Environmental Factors
		Size	Conc.	
NEMS Mass Spectrometry [135]	100 nm	Yes	Yes	None
Nano Electrode Arrays [64]	50-500 nm	Yes	Yes	None
Impedance Sensor Array [133]	2.8 μm	Yes	Yes	None
Impedance Cytometry & Microwave Resonators [134]	20 μm	Yes	Yes	None
Microfluidic Microwave Sensors [138]	10-20 μm	Yes	Yes	None
MEMS Resonator [139]	25 μm	No	Yes	None
Spiral Resonator [235]	20-70 μm	Yes	Yes	None
C-Split Ring Resonator [63]	30-80 μm	Yes	Yes	None
LC Resonator [66]	50-500 μm	No	Yes	None
Conventional Transmission Line [65]	N/A	No	Yes	None
Flow-Through Cavity [236]	300-1000 μm	Yes	Yes	None
This Work	10-75 μm	No	Yes	Temperature

the number of data points, in forthcoming experiments we plan to use a more advanced VNA system, specifically the Copper Mountain M5090. The latter boasts 200,000 data points for the full frequency range (300 kHz to 8.5 GHz), representing a more than 50 times increase in the number of data points compared to the equipment employed in this study. Additionally, environmental noise also plays a critical role in influencing the LOD, particularly at enhanced resolution levels. In future work, we will focus on developing strategies to mitigate the impact of environmental noise, ensuring that our measurements are both accurate and reliable under varied conditions.

Furthermore, the current sensing system relies on a split-ring resonator (SRR)-based sensor featuring a 1 mm capacitive gap in the inner ring, which is notably larger than the microspheres in our experiments. The 1 mm gap was inherited from our initial trials with the AI-powered QuantWave system, that is, before introducing the replaceable reservoir to address the adhesion of Tween 20 to the microfluidic chip and tubing (section Materials and Methods). The literature suggests that aligning the gap size in a SRR with the characteristic dimension of the particles being tested enhances detection sensitivity and specificity [237]. In that case, electromagnetic interactions between the SRR and the material intensify, resulting in stronger coupling and more distinct changes in the SRR’s response in the presence of the target material. Future method development will therefore emphasize increasing the system’s performance by maximizing the signal-to-noise ratio and ensuring the SRR is most sensitive to microplastic particles while being less affected by other matrix effects. Integrating impedance cytometry and microwave sensing also shows a promising pathway to enhance specificity and accuracy in microplastic detection applications [134].

Pre-concentration methods could also be used to augment the concentration of microparticles in samples [238, 239]. These methods include external filtering performed on-site on the water samples or via microfluidic approaches to increase the on-chip concentration, such as capturing the microparticles in a reservoir and detecting them in flow-through mode [240, 235, 236]. In addition, the potential of manipulating the dielectric constant of the microplastics suspension by adding organic and non-organic solutes to aqueous samples deserves further consideration [241]. Overall, the flexibility of the microfluidic microwave detection approach could be leveraged by developing sensing platforms that incorporate multiple sensors, each designed to monitor specific microplastic particles, sizes, concentrations, or variable surface properties due to degradation processes.

To contextualize the performance and applicability of our microwave-microfluidic sensing approach, Table 4.5 presents a comparative overview of various sensing technologies reported in recent literature. The table highlights the size ranges, concentration estimation capabilities, and environmental factors addressed by each method. The papers were selected for their focus on plastic particles at either the micron or nano scale, ensuring relevance to the detection challenges associated with microplastics. This comparative analysis emphasizes the diversity of methodologies employed in the field and highlights how our approach aligns with or differs from existing technologies.

4.7 Uncertainty Analysis

The accuracy and reliability of the results presented in this study are subject to several sources of uncertainty, which can be broadly categorized into systematic and operational errors. Identifying and understanding these factors is essential for assessing the limitations of the current platform and guiding improvements in future iterations.

Systematic errors stem from the limitations of the instruments and materials employed throughout the experimental process. A primary source of such error is the Vector Network Analyzer (VNA), specifically the Anritsu MS2028C used in this work, which has a resolution limit of 100 kHz. Although the device is capable of displaying frequency shifts below this value, such readings fall within its uncertainty range and cannot be reliably interpreted, particularly in low-concentration conditions where signal shifts are subtle. Additionally, while a temperature-controlled chamber was used to maintain the environment between 10°C and 30°C, small fluctuations in ambient conditions can still influence the dielectric properties of the samples and thus affect the measured resonances.

Another contributing factor is the variability in the micro-reservoir fabrication. The PDMS layer and the bonding process using oxygen plasma can introduce inconsistencies in

reservoir geometry and alignment. These affect the distribution of the electromagnetic field and, consequently, the resonance response of the sensor. Furthermore, although the particle sizes (20 μm and 70 μm) and densities are nominally known, manufacturing tolerances and batch variation can result in discrepancies that alter the effective volume fraction and permittivity of the suspension. Signal inconsistencies may also arise from imperfect soldering of SMA connectors and variability in cable quality, which can introduce parasitic losses or impedance mismatches that deviate from simulation assumptions.

In contrast, operational errors arise from aspects of manual handling and experimental procedures. For instance, fabrication steps such as the alignment of the concentric ring structures or quality of etching can vary slightly between devices, influencing the baseline resonance behavior. Sample preparation is another potential source of error. Errors in pipetting, incomplete mixing, or non-uniform vortexing can lead to inaccurate particle concentrations or aggregation of particles, which affects the local dielectric profile of the sample. Despite the use of a surfactant (Tween 20) to aid in dispersion, full homogenization of the suspension is not guaranteed, and aggregation or settling can occur.

Additionally, variation in surface charge across different batches of microplastic particles may impact their distribution within the static reservoir and interaction with the microwave field. Finally, unavoidable human factors, such as minor misalignments in placing the reservoir on the sensor, or inconsistencies in cleaning and refilling procedures between tests, can contribute to cumulative deviations across measurements.

4.8 Summary

This exploratory study demonstrates the proof of concept for detecting microplastics in aqueous samples using a microwave sensor integrated into a microfluidic platform. The sensor works by detecting shifts in resonance frequency caused by changes in the permittivity of water when microplastics are present. The study focused on polyethylene microspheres of two sizes (20 μm and 70 μm) and examined how their concentration and temperature affected detection. The results show that larger particles and higher concentrations lead to more significant frequency shifts, with temperature also influencing the measurements. While the method showed promise, the detection limits (LOD) are still higher than typical microplastic concentrations in most freshwaters, suggesting the need for further development to improve sensitivity and reduce the LOD.

4.9 Supplementary Information

In addition to the main findings presented in this chapter, supplementary information is provided to support and expand upon the methodologies, data analysis, and results discussed. These supplementary details offer additional context and technical insights, ensuring a comprehensive understanding of the work and its contributions to the field.

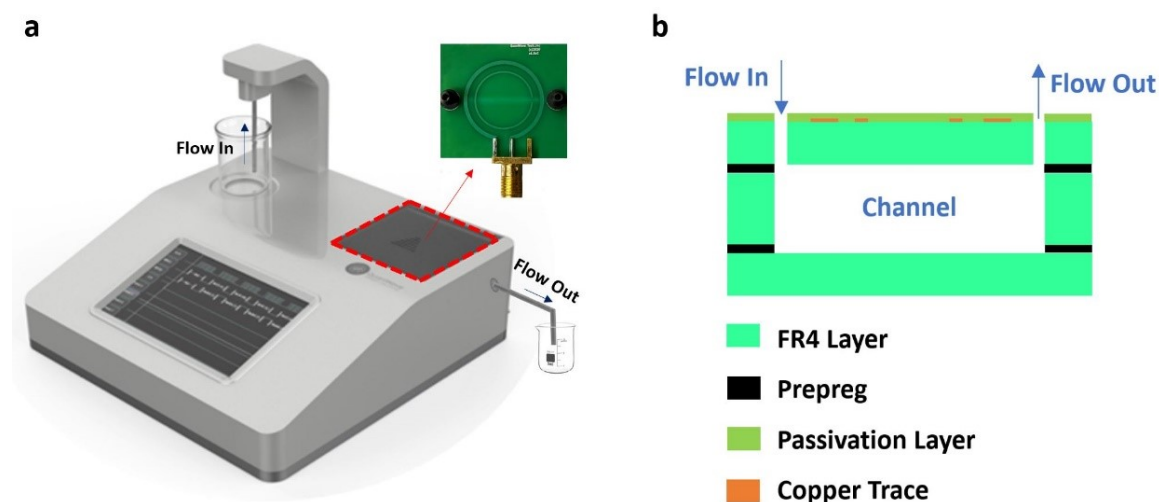


Figure 4.7: (a) Schematic of the AI-powered QWave system (QuantWave Technology) with detailed microwave sensor design, and (b) cross-sectional view of the microwave sensor highlighting its components.

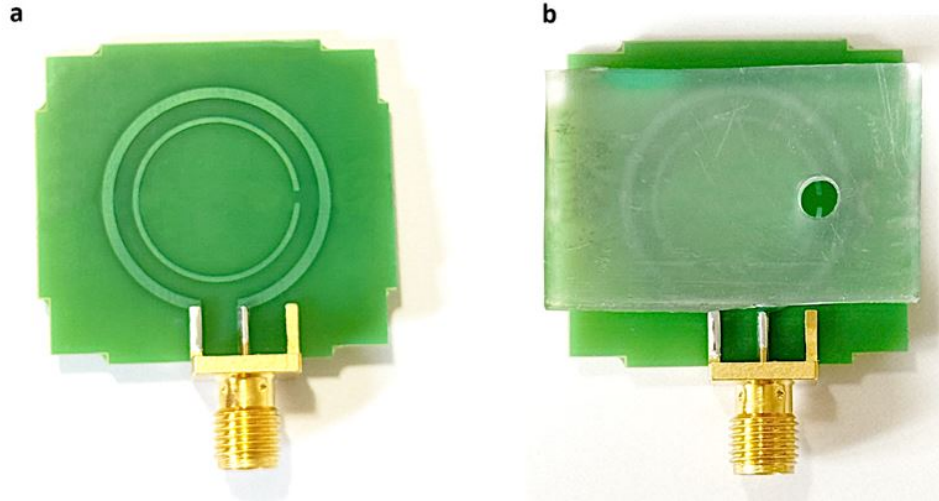


Figure 4.8: (a) Schematic of the fabricated sensor featuring two planar concentric ring-shaped electrodes with outer and inner ring radius of 9.75 mm and 7.25 mm, respectively. (b) The PDMS-made micro-reservoir, with a volume of 100 μL , positioned atop the sensor.

Table 4.6: Dielectric constants of the microsphere suspensions with varying particle size and concentration calculated with Equation (1). The table provides the values at a temperature of 20°C.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Dielectric constant at 10°C					
0	83.0540	83.0540	83.0540	83.0540	83.0540	83.0540
100	83.0540	83.0540	83.0540	83.0540	83.0537	83.0531
1k	83.0540	83.0540	83.0539	83.0537	83.0514	83.0452
10k	83.0540	83.0537	83.0529	83.0507	83.0279	82.9661
100k	83.0537	83.0510	83.0428	83.0214	82.7934	82.1746
1000k	83.0514	83.0243	82.9423	82.7283	80.4484	74.2602
10000k	83.0279	82.7572	81.9369	79.7970	56.9984	-

Table 4.8: Frequency shifts of the microsphere suspensions with varying sizes and concentrations at 20°C simulated with HFSS.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Frequency shift at 10°C (MHz)					
0	0	0	0	0	0	0
100	0	0	0	0	0	0
1k	0	0	0	0	0	0
10k	0	0	0	0	0.1	0.3
100k	0	0	0	0.1	0.9	3.1
1000k	0	0.1	0.4	1.1	9.2	33.2
10000k	0.1	1	3.9	11.5	119.6	-

Table 4.7: Magnitudes of resonance frequencies simulated with HFSS for microsphere suspensions with varying sizes and concentrations at 20°C.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Resonance frequency at 10°C (GHz)					
0	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
100	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
1k	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
10k	1.1506	1.1506	1.1506	1.1506	1.1507	1.1509
100k	1.1506	1.1506	1.1506	1.1507	1.1515	1.1537
1000k	1.1506	1.1507	1.1510	1.1517	1.1598	1.1838
10000k	1.1507	1.1516	1.1545	1.1621	1.2702	-

Table 4.9: Dielectric constants of the microsphere suspensions with varying particle size and concentration calculated with Equation (1). The table provides the values at a temperature of 30°C.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Dielectric constant at 10°C					
0	83.0540	83.0540	83.0540	83.0540	83.0540	83.0540
100	83.0540	83.0540	83.0540	83.0540	83.0537	83.0531
1k	83.0540	83.0540	83.0539	83.0537	83.0514	83.0452
10k	83.0540	83.0537	83.0529	83.0507	83.0279	82.9661
100k	83.0537	83.0510	83.0428	83.0214	82.7934	82.1746
1000k	83.0514	83.0243	82.9423	82.7283	80.4484	74.2602
10000k	83.0279	82.7572	81.9369	79.7970	56.9984	-

Table 4.10: Magnitudes of resonance frequencies simulated with HFSS for microsphere suspensions with varying sizes and concentrations at 30°C.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Resonance frequency at 10°C (GHz)					
0	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
100	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
1k	1.1506	1.1506	1.1506	1.1506	1.1506	1.1506
10k	1.1506	1.1506	1.1506	1.1506	1.1507	1.1509
100k	1.1506	1.1506	1.1506	1.1507	1.1515	1.1537
1000k	1.1506	1.1507	1.1510	1.1517	1.1598	1.1838
10000k	1.1507	1.1516	1.1545	1.1621	1.2702	-

Table 4.11: Frequency shifts of the microsphere suspensions with varying sizes and concentrations at 30°C simulated with HFSS.

Particle Size (μm)/ Concentration (particles L^{-1})	20	45	70	100	200	300
	Frequency shift at 10°C (MHz)					
0	0	0	0	0	0	0
100	0	0	0	0	0	0
1k	0	0	0	0	0	0
10k	0	0	0	0	0.1	0.3
100k	0	0	0	0.1	0.9	3.1
1000k	0	0.1	0.4	1.1	9.2	33.2
10000k	0.1	1	3.9	11.5	119.6	-

Chapter 5

Size and Concentration Characterization of Microplastic Particles in Aqueous Samples Using Sensitivity-enhanced Coupled Planar Microwave Resonators

5.1 Overview

This chapter presents the progression of work on microwave-microfluidic sensors for detecting microplastic particles in aqueous samples. The preliminary results established the feasibility of using a passive disposable microwave sensor for online microplastic contamination monitoring, which was accepted and published in *2022 IEEE/MTT-S International Microwave Symposium*. Building on these findings, the research advanced to include sensitivity-enhanced coupled planar microwave resonators capable of size and concentration characterization of microplastic particles. The comprehensive results from this work have been submitted for publication in *Elsevier Journal of Hazardous Materials* and are currently under review.

Maziar ShafieiDarabi, Zahra Abbasi, and Carolyn L. Ren, "Passive Disposable Microwave Sensor for Online Microplastic Contamination Monitoring," 2022 IEEE/MTT-S International Microwave Symposium - IMS 2022, Denver, CO, USA, 2022, pp. 263-266.

Maziar ShafieiDarabi, Zahra Abbasi, and Carolyn L. Ren "Size and concentration characterization of microplastic particles in aqueous samples using sensitivity-enhanced coupled planar microwave resonators" *Journal of Hazardous Materials* (2024).

This study presents a simple yet novel microwave sensing platform for real-time, on-site monitoring of microplastic (MP) particle size and concentration in water, with its sensitivity enhanced through the integration of an interdigital capacitor (IDC) with the traditional split-ring resonator (SRR) structure. The platform utilizes a disposable sample holder ($< \$1$), enabling multiplex testing without cross-contamination. The sensing principle relies on electromagnetic interactions between the suspended MP particles and the microwave resonator that is electromagnetically coupled with a planar microwave reader. Initially, the MP particles are homogeneously distributed in the reservoir, but they gradually settle toward the bottom over time, resulting in a dynamic shift in the resonance frequency that depends on MP size. The resonance frequency shift plateaus once all particles have settled, providing a measure of MP concentration. The sensor was designed and optimized using HFSS Simulations and tested across various temperatures (10–30 °C) and host mediums (DI water, tap water, NaCl and urea solutions). Its performance was evaluated by detecting MPs of different sizes (20, 70, and 275 μm) at concentrations of 100k, 1,000k, and 10,000k particles/L. The average detection slopes were 8.64 kHz for 20 μm , 38.52 kHz for 70 μm , and 110.78 kHz for 275 μm . This novel sensor demonstrates significant potential for on-site monitoring of both the size and concentration of MP particles, offering a cost-effective solution for environmental monitoring.

5.2 Introduction

In recent years, there has been growing interest in understanding the impacts of microplastics (MPs), commonly defined as plastic particles smaller than 5 mm, on the natural environment and human health [242], [243], [244], [245]. MPs are ubiquitous pollutants found in diverse ecosystems, including seawater, freshwater, sediments, soil, and even within nearly every organ of the human body [246], [247], [248], [14], [249], [250], [251], [252]. These pollutants originate from diverse sources such as synthetic textiles, tires, personal care products, landfills, construction projects, factories, and agriculture [7], [8], [9], [10]. Their small size and persistence make them difficult to remove from the environment, allowing them to spread widely across various ecosystems. MPs are present in many different forms, such as spherical, fiber-like, and irregular shapes, which adds complexity to their characterization [7]. While the exact effects of MPs on the natural environment and human health remain unclear, it is widely recommended to prevent and control MP pollution by reducing

their production and consumption, promoting recycling, and finding biodegradable alternatives [253], [254], [255]. Concurrently, significant attention has been directed toward understanding the distribution, abundance, and characterization of MPs. Addressing this issue is crucial to mitigating their environmental and health impacts, particularly given their ability to interact with other pollutants and bioaccumulate in food chains. To tackle these challenges, rapid detection and monitoring of MPs, with the potential for on-field monitoring, are urgently needed to better understand their distribution, abundance, and characterization.

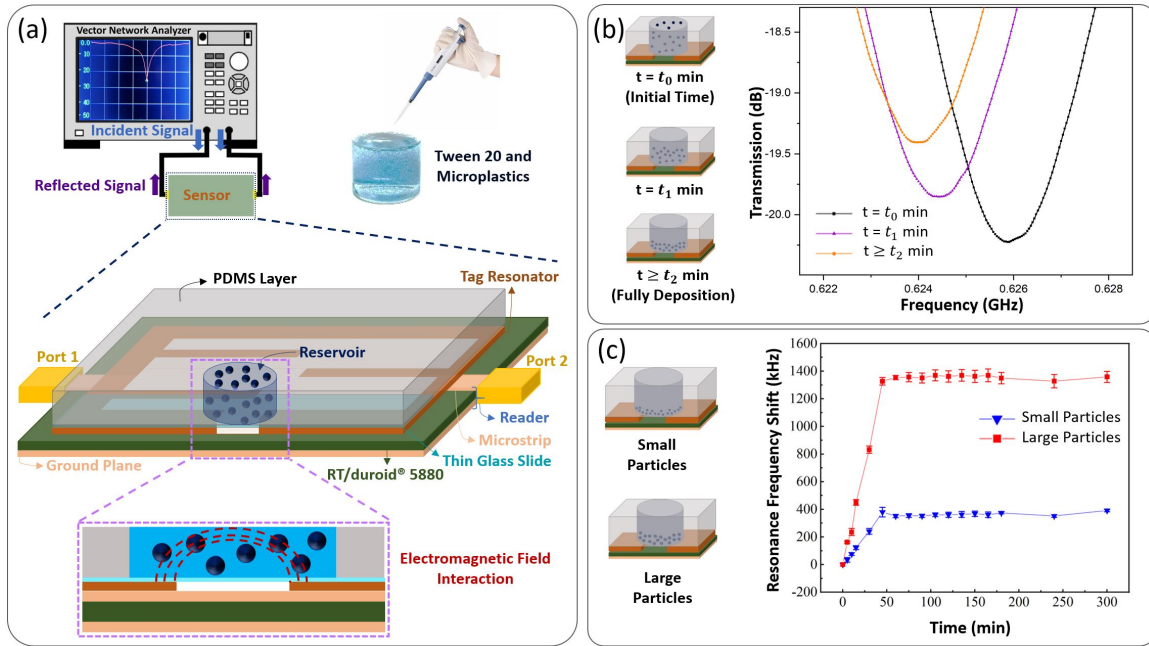


Figure 5.1: Microwave sensing mechanism for monitoring the concentration and size of microplastics: (a) Schematic of the system consisting of a microwave sensor with a reservoir hosting the sample under test which is a mixture of Tween 20 0.1% and microplastics and a vector network analyzer (VNA) used to monitor the transmission coefficient; (b) Numerically simulated transmission coefficient (S_{21}) as a function of the deposition rate of microplastics; and (c) Demonstration of the dependence of the experimentally measured resonance frequency shift as a function of the size of microplastics.

Several well-established methods have been developed for detecting and monitoring MPs in aqueous samples [32]. Fourier-transform infrared (FTIR) and Raman spectroscopy

are among the most widely used techniques for accurately determining MP contamination due to their unique ability to identify chemical compositions at the molecular level [256], [257], [258], [13], [259], [260], [261], [262], [263]. However, these methods generally require bulky, expensive equipment and well-trained personnel to operate. Additionally, they are offline techniques that involve time-consuming and laborious sampling processes, often taking several hours to complete [33]. As a result, there is a growing demand for affordable and user-friendly MP sensing techniques suitable for on-site applications.

Electrical sensing methods offer unique advantages for on-site detection of MPs due to their compact detection systems and scalability for multi-location testing. Several approaches have been explored as summarized in Table 5.1, including resonance microwave spectroscopy (RMS), dielectric spectroscopy (DS), high-frequency impedance spectroscopy (HFIS), and electrical impedance spectroscopy (EIS). Each method has a unique interaction with the electrical properties of the material, providing diverse capabilities for MP detection [62], [63], [64], [65].

Although most electrical sensing methods share similar working principles, RMS stands out as a promising solution for on-site MP detection due to its broader frequency range (typically 0.1 to 100 GHz), enabling more versatile and precise detection of various particle types. Microwave sensing differentiates materials based on their permittivity, making it highly sensitive for detecting MPs, which typically exhibit permittivity values (~ 2 to 3.5) distinct from their natural surrounding materials, such as water (~ 80), wet sediments (~ 10 to 30) and blood (~ 50 to 60) [66], [67]. Furthermore, microwave sensors can be integrated with planar technology, such as printed circuit boards (PCBs) and microstrip antennas, to create compact, lightweight, durable, and cost-effective systems [264], [265], [266], [267], offering a practical solution for continuous measurements in real-world applications [268], [269].

Most existing RMS methods primarily focus on determining the concentration of MPs, which cannot assess particle size or type. A few methods, such as spiral resonator and multi-complimentary split ring resonator (CSRR), can estimate particle size within a certain range with the cost of complicating the system due to the need to incorporate physical filters [81], [63]. While single-particle monitoring methods like HFIS and EIS [62], [64] are capable of determining particle size, concentration, and type, they require complex systems to measure both low- and high-frequency responses and to precisely control flow rates, ensuring that only one particle passes through the channel at any given time. In contrast, the novelty of this work lies in its ability to simultaneously monitor both the size and concentration of MPs using a microwave resonance-based bulk monitoring method, as illustrated in Figure 5.1, without the need for physical filters or single-particle monitoring. By leveraging the natural relationship between particle size and deposition rate,

our method eliminates the complexities of precise flow control or multi-frequency response measurements required in single-particle methods. Furthermore, the proposed sensor can effectively monitor MPs across a wide size range, from 20 μm to 300 μm , and at concentrations spanning three orders of magnitude. This approach not only simplifies the detection process but also significantly expands the versatility and practicality of MP monitoring systems for real-world applications.

5.2.1 Measuring principle

The measuring principle can be understood as follows. The deposition rate of MPs in water depends on their size, resulting in size-dependent displacements of MPs over a given time period. Equ (5.1) describes this displacement for a single particle, derived considering ideal assumptions, including perfectly spherical particles and negligible colloidal interactions. While Equ (5.1) may not precisely represent the behavior of MPs in water, it highlights that this displacement is inherently size-dependent. As a result, over a given time period, the number of MPs settling within the sensing region - and thus electromagnetically interacting with the microwave sensor - varies with size. This interaction results in a size-dependent shift in the resonance frequency from that corresponding to the original sample where MPs are homogeneously distributed. As MPs gradually settle toward the bottom, the frequency shift evolves over time, eventually plateauing once all MPs have settled. The slope of the resonance frequency shift over time can be used to determine the size of the MPs while the plateau value serves as an indicator of MP concentration. Therefore, by monitoring the time-varying resonance frequency shift, this method is capable of determining both the MP size and concentration using a simple system shown in Figure 5.1. Aggregation of MPs, which can affect measurement accuracy, is mitigated by adding a surfactant, Tween 20, as demonstrated in our previous study [140].

5.3 Sensor Design and Theory of Operation

5.3.1 Sensor Design and Simulation

The sensing platform consists of two major components: a sensing chip and a vector network analyzer (VNA) for monitoring resonance frequency. The sensing chip is a multilayer structure comprising three layers: a sample holder as the top layer (5mm thick), an interdigital capacitor (IDC)-inspired tag resonator as the middle layer (252 μm thick), and a

passive planar microwave reader as the bottom layer (1.575mm thick). The sample holder is fabricated from a Polydimethylsiloxane (PDMS) substrate featuring a punched-through hole of 100 μ L, serving as the sample reservoir. A 150 μ m-thick 1"x3" glass slide is bonded to the PDMS via oxygen plasma treatment, forming the sample reservoir. The reservoir is precisely aligned with the primary gap of the tag resonator to ensure maximum electromagnetic coupling between the sample solution and the resonator. The tag resonator and the reader are separated by two 0.5mm-thick small spacers of Polymethylmethacrylate (PMMA) with the remaining areas separated by air. A larger air gap allows more flexibility to place the tag resonator, however, too large an air gap would compromise the damping of the electromagnetic (EM) wave. The current air gap of 0.5mm was optimized by ensuring the highest transmission coefficient (S_{21}) value from port 1 to port 2 without compromising the damping of the EM wave. The three layers are assembled together using adhesive tapes in this study, which can be refined for practical, on-site applications. This modular structure allows the relatively expensive tag and reader components to be reused, while the low-cost sample holder (< \$1) is disposable, enabling multiplex testing without cross-contamination.

Split ring resonator (SRR) structures are widely used in microwave sensing applications due to their simplicity and well-documented performance in the literature. However, they failed in detecting the deposition rate of MPs even with high concentrations. A logic improvement is to increase the sensing region, as demonstrated by the dual SRR structure employed in our previous study for MP detection [269]. This structure however requires precise alignment of the two SRRs, which is difficult to achieve in experiments resulting in low reproducibility. To address these challenges, we adopted a trending approach in this study by integrating an IDC structure with the traditional SRR [270, 271]. As illustrated in the lumped-element equivalent circuit model (Figure 5.2 (b)), the SRR+IDC structure provides additional coupling paths ($C_{\text{Leg-SRR}}$ and $C_{\text{Leg-Reader}}$) compared to the traditional SRR (limited to $C_{\text{crt}} - R_{\text{crt}}$). These additional paths arise from interactions between the IDC electrode legs and the strip line in the reader, as well as between the IDC legs and the outer part of the tag resonator. The increased coupling paths generate a stronger electric field in the sensing region, particularly in the primary gap of the resonator that aligns with the sample reservoir. The number and width of the IDC legs were optimized to achieve characteristic impedance matching between the reader and the tag resonator, ensuring maximum electromagnetic coupling.

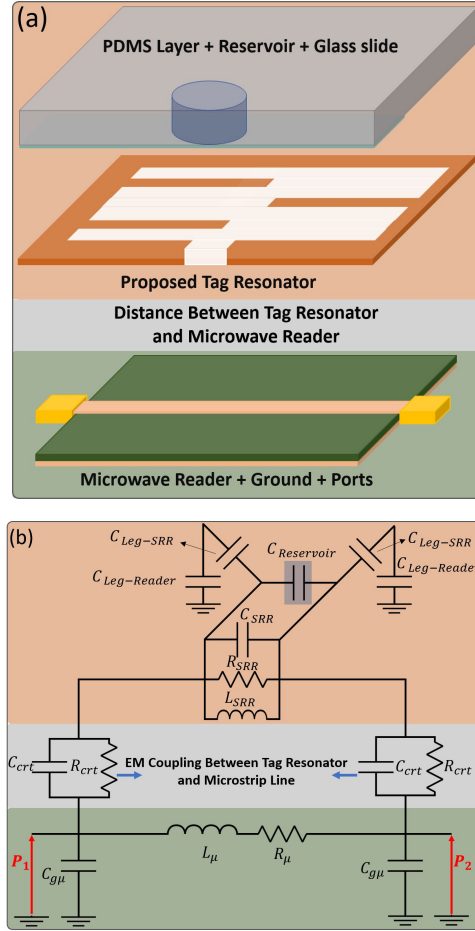


Figure 5.2: (a) The sensing platform schematic: the planar microwave reader and the chipless tag resonator. The planar microwave reader is responsible for transmitting and receiving microwave signals, while the chipless tag resonator interacts with the transmitted signals. The schematic diagram provides an overview of the arrangement and connection of these components. In addition, (b) presents a lumped-element equivalent circuit, which represents the electrical behavior and characteristics of the sensing platform. This circuit model helps in understanding the interactions and dynamics of the components within the platform.

To further confirm the enhanced sensing performance of the proposed SRR+IDC structure compared to a simple SRR, a comparative analysis of their resonance profiles was conducted using HFSS simulations. As shown in Figure 5.3 (a), the SRR+IDC sensor

outperformed the simple SRR in terms of the amplitude of the reflection coefficient at the resonance frequency, achieving a quality factor of 89.2142 compared to 79.9477 for the simple SRR. The sensitivity of microwave sensing is primarily influenced by the electric field intensity near the primary gap. As shown in Figure 5.3 (b), the SRR+IDC sensor exhibits significantly higher electric field intensity than the simple SRR sensor. This improvement is attributed to the coupling effect between the IDC legs. The stronger electric field not only enhances the interaction between the sensor and the sample within the reservoir but also improves the sensor's ability to detect lower MP concentrations or a wider range of concentrations. Moreover, the heightened electric field intensity improves the sensor's signal-to-noise ratio, enabling more reliable and accurate outputs. It also allows for faster response times to changes in the sample, which is crucial for applications requiring real-time or rapid detection. In summary, the heightened electric field intensity of the SRR+IDC structure enhances its sensitivity and overall performance, making it well-suited for a wide range of sensing applications.

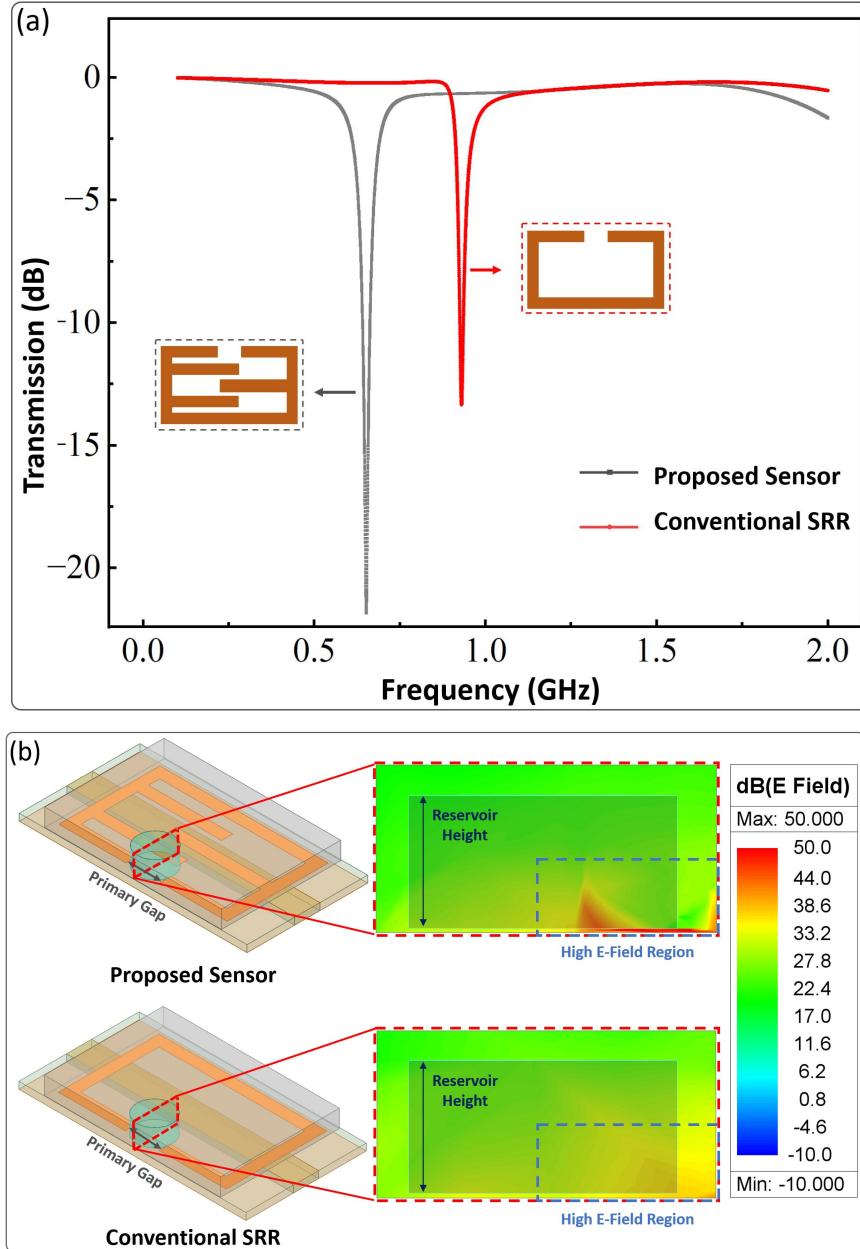


Figure 5.3: (a) An analysis of the S_{21} response of the proposed sensor, a combination of the Interdigital Capacitor (IDC) and Split Ring Resonator (SRR) structure and that of the conventional SRR, (b) the electric field intensity surrounding the reservoir, which holds the sample under test and is positioned on top of the resonator, observed from the lateral perspective.

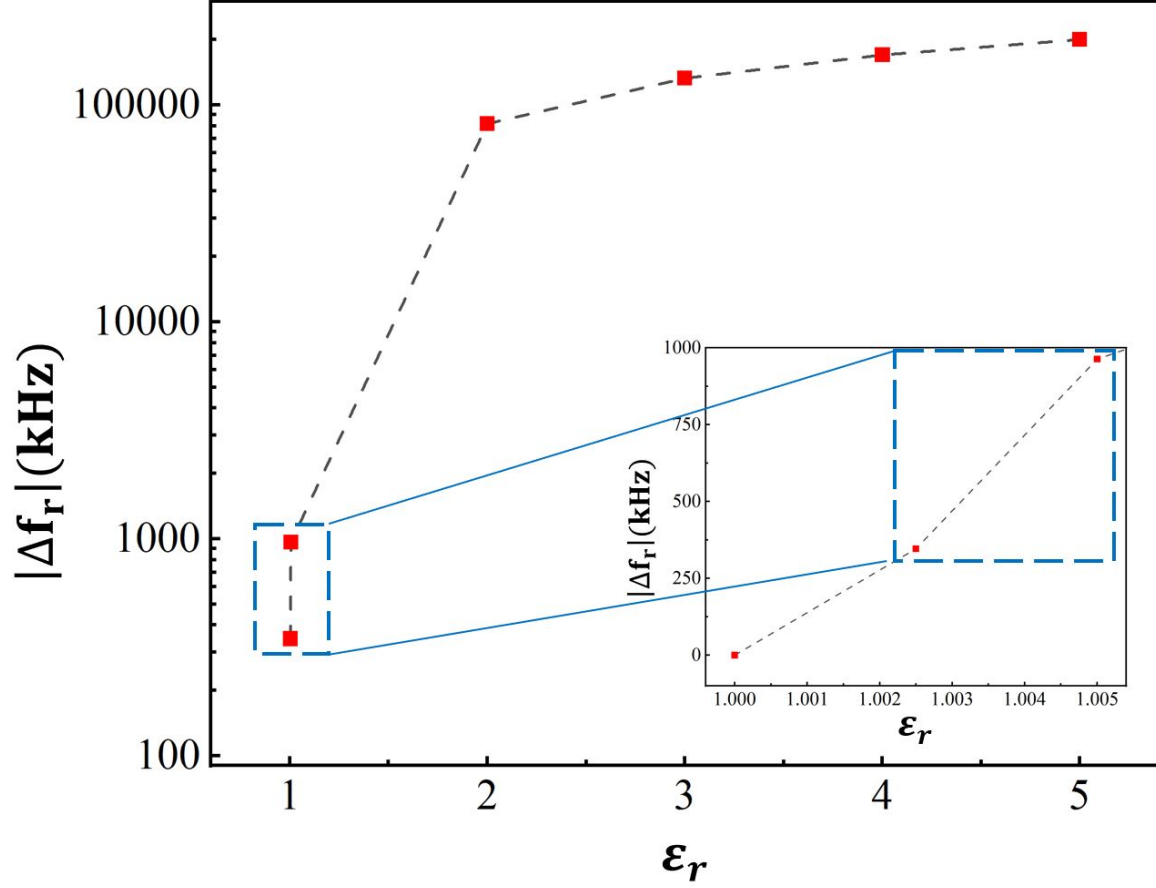


Figure 5.4: The absolute value of resonance frequency shifts across a permittivity range of 1 to 5, with an inset highlighting shifts at specific permittivity values (1, 1.0025, and 1.005). Since the resonance frequency at a permittivity of 1 is used as the reference, the resonance frequency shift at this permittivity equals zero and cannot be represented on the logarithmic scale of the main plot. However, it is included in the inset for clarity.

The permittivity of commonly observed MPs ranges from 1 to 5. While experimental studies are constrained by the availability of MPs, the sensing potential of the SRR+IDC sensor can be evaluated using HFSS simulations. Figure 5.4 presents the numerically simulated frequency shifts corresponding to different relative permittivity values ranging from 1 to 5. The results clearly demonstrate that the proposed sensor is capable of detecting a change as small as 0.005 in relative permittivity, resulting in a frequency shift of nearly 1

MHz. This underscores the design's exceptional sensitivity, even when accounting for the presence of an air gap between the tag and the reader.

5.3.2 Correlation between Microsphere Size and Deposition Speed in Aqueous Samples

We experimentally observed that larger MPs settle toward the bottom more rapidly than smaller ones, which inspired the idea of measuring both the size and concentration of MPs by monitoring the resonance frequency shift over time. In principle, larger MPs deposit more quickly into the sensing region than smaller ones within the same time period, which result in distinct differences in time-dependent resonance frequency shifts. The unique slope of the time-dependent frequency shift can be used to determine MP size while the plateaued value corresponding to the scenario where all MPs settled at the bottom indicates the MP concentration. To validate this concept, we conducted a theoretical analysis of the displacement of a settling MP particle under idealized assumptions, including perfectly spherical particles and negligible colloidal interactions. The primary forces acting upon the particle include gravity, viscous drag force and buoyancy force [272], [273], [274]. Solving the governing equations yielded a closed-form equation for the particle displacement within a viscous aqueous sample:

$$\begin{aligned}
y(t) = & \frac{2\rho_{\text{particle}}r^2}{9\mu}g\left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right)t \\
& - \frac{4\rho_{\text{particle}}^2r^4}{81\mu^2}g\left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right)e^{-\frac{9\mu}{2\rho_{\text{particle}}r^2}t} \\
& - \frac{4\rho_{\text{particle}}^2r^4}{81\mu^2}g\left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right)
\end{aligned} \tag{5.1}$$

where ρ_{particle} [kg/m³] represents the density of the spherical particle, ρ_{fluid} [kg/m³] is the density of the fluid, r [m] stands for the radius of the spherical particle, g [m/s²] denotes the gravitational acceleration, and μ [kg/(m · s)] signifies the dynamic viscosity. Equ (1) shows that the particle displacement is size dependent and larger particles have a larger displacement, meaning that falling more rapidly than smaller ones. This confirms our idea of measuring the deposition rate to monitor MP size. More detailed derivations can be found in the supplemental file.

5.3.3 Behavior of Electromagnetic Waves in Multi-Layer Medium with Varying Permittivity

As previously explained, the gradual settling of MPs toward the sensor surface causes the number of MP particles near the sensing region to vary with time, resulting in a time-dependent resonance frequency as shown in Figure 5.1 (b). A downward shift in the resonance frequency was observed in our experiments for all MP sizes. This phenomenon occurs because the sample solution transforms into a two-layer structure during the deposition process. The bottom, thin layer is primarily composed of MPs with low permittivity (2.4 in this study), while the upper, thick layer consists of the high-permittivity (~ 80) host medium, a Tween 20 (0.1%) solution with suspended MPs. As the deposition progresses, the thickness of the bottom MP layer increases with the accumulation of MPs until all MPs have settled. At this stage, the upper layer consists of almost entirely of pure Tween20 (0.1%) solution. To better understand the downward shift in resonance frequency during this deposition process, we examine the behavior of EM wave in a multi-layer medium, where each layer possesses a distinct permittivity.

In a medium composed of two layers with distinct permittivity values, EM waves encountering the boundary between these layers undergo reflection and transmission processes because changes in permittivity affect the characteristics of the incident wave, including its amplitude and phase. These phenomena can be quantified using the reflection and transmission coefficients, which represent the proportion of the incident wave that is reflected back into the first layer and that which propagates into the second layer. Consider an EM wave initially present in the first layer, characterized by permeability μ_1 and permittivity ϵ_1 , exhibits an electric field in the y-direction and a magnetic field in the x-direction while propagating along the z-direction. As this incident wave reaches the interface with the second layer, characterized by permeability of μ_2 and permittivity ϵ_2 , the incident, transmitted, and reflected electric and magnetic fields can be expressed as follows:

$$E_{y,i}(z, t) = A_i e^{j(\omega t - k_1 z)}, \quad (5.2)$$

$$E_{y,r}(z, t) = B_r e^{j(\omega t + k_1 z)}, \quad (5.3)$$

$$E_{y,t}(z, t) = A_t e^{j(\omega t - k_2 z)}. \quad (5.4)$$

Given the knowledge of the characteristic impedance of the two layers, denoted as Z_{c1} and Z_{c2} , the magnetic field can also be expressed as follows:

$$H_{x,i}(z, t) = -\frac{A_i}{Z_{c1}} e^{j(\omega t - k_1 z)}, \quad (5.5)$$

$$H_{x,r}(z, t) = +\frac{B_r}{Z_{c1}} e^{j(\omega t + k_1 z)}, \quad (5.6)$$

$$H_{x,t}(z, t) = -\frac{A_t}{Z_{c2}} e^{j(\omega t - k_2 z)}. \quad (5.7)$$

The tangential component of the electric field in the first layer combines the incident and reflected waves, while the tangential component of the electric field in the second layer solely comprises of the transmitted wave. Similarly, the tangential magnetic component in the first layer results from the sum of the incident and reflected waves, whereas, the tangential magnetic field component in the second layer is comprised of the transmitted component. Moreover, the characteristic impedance can be expressed in terms of the permittivity and permeability of the layers [275], [276]. Subsequently, the transmission and reflection coefficients can be defined as follows:

$$\gamma = \frac{\sqrt{\epsilon_1} - \sqrt{\epsilon_2}}{\sqrt{\epsilon_1} + \sqrt{\epsilon_2}} \quad (5.8)$$

$$T = \frac{2\sqrt{\epsilon_1}}{\sqrt{\epsilon_1} + \sqrt{\epsilon_2}} \quad (5.9)$$

Equs. (5.8- 5.9) demonstrate how the amplitude of transmitted waves increases as the permittivity contrast between the layers grows. In our experiments, the observed downward shift in resonance frequency is a direct result of the heightened amplitude of the transmitted waves under the two-layer configuration. This arises due to the significant permittivity difference between the MPs ($\epsilon_1 = 2.4\epsilon_0$) and the Tween 20 solution ($\epsilon_2 \sim 80\epsilon_0$). As the MPs settle at the reservoir bottom, forming a distinct layer, this change in permittivity distribution enhances the transmitted wave amplitude compared to a uniform distribution scenario. Once all MPs have settled at the bottom, the increased wave amplitude leads to a longer effective propagation path for the transmitted EM waves, extending the propagation distance and increasing the system's capacitance. It is worth noting that the resonance frequency f_r is inversely proportional to the square root of the capacitance, meaning that a higher capacitance results in a lower resonance frequency. This change in EM wave behavior highlights the intricate interaction between the MPs and the host medium, shedding light on how they jointly affect the system's EM characteristics. These findings underscore the

potential of EM wave-based sensing as a powerful tool for monitoring and quantifying the dynamic behavior of MPs within the sample.

Although the intrinsic permittivity jump between the microparticle layer and the surrounding Tween-20 solution is large, the particle layer is only tens of micrometres thick—several hundredths of a wavelength at our operating frequencies. The relevant perturbation parameter, therefore remains well below unity, so a first-order, normal-incidence plane-wave treatment captures the dominant change in stored electric energy and predicts the observed resonance shift to within experimental uncertainty while keeping the analysis fully analytic and transparent. A rigorous lateral-wave (evanescent-field) model, which would require the full spatial field distribution and a numerical solution of the surface-wave dispersion relation, could refine the absolute capacitance values and will be pursued in future work aimed at high-precision permittivity retrieval for near-field sensors.

5.4 Materials and Methods

The sample solutions consisted of a 0.1% Tween 20 solution with suspended MP particles. The MP particles used in this study were blue polyethylene microspheres obtained from Cospheric LLC, categorized into three size ranges: 10-27 μm (average diameter 20 μm), 63-75 μm (average diameter 70 μm), and 250-300 μm (average diameter 275 μm). The preparation began with by creating a 0.1% Tween 20 solution, which was made by dissolving Tween 20 in boiling deionized water. To ensure even dispersion of the surfactant, a magnetic stir bar was used. The prepared Tween 20 solution was added to a vial containing the MP particles. The vial was then centrifuged for 5 minutes to achieve a homogeneous distribution of the MP particles within the suspension. Three different concentrations - 100k, 1,000k, and 10,000k Particles/L - were prepared for each size category.

Also, the effects of host medium, temperature, and particle type were investigated. The host media included deionized water, tap water, 0.1 M sodium chloride (NaCl) solution, and 0.05 M urea solution, all prepared with Tween 20 using the same process mentioned in the last paragraph for deionized water, with PE particles of 275 μm and a concentration of 100k Particles/L. The effect of temperature was studied by performing experiments at three different temperatures: 10°C, 20°C, and 30°C, using PE particles of 275 μm and a concentration of 100k Particles/L. For the particle type comparison, Soda Lime Solid Glass particles from Cospheric LLC, with a size range of 250-300 μm (average diameter 275 μm), at a concentration of 100k Particles/L, were prepared and compared with polyethylene particles of the same size and concentration.

The microwave tag resonator and reader were fabricated using a wet chemical etching process. First, the design was printed on a glossy photo paper and then laminated onto a copper-coated polymer slide using a thermal laminator, which patterned the design onto the copper-coated slide. The patterned slide was then subjected to copper etching and subsequent acetone washing to complete the fabrication process. The fabricated tag resonator and reader, shown in Figure 5.5(c), are separated by two small PMMA spacers. The sample holder consists of a PDMS mold featuring a punch-through hole as the sample reservoir and a 0.15mm-thick glass slide. These components were bonded together via oxygen plasma treatment. The PDMS mold was made by mixing the base and curing agent in a 10:1 ratio, degassing for 30 mins and baking on a hotplate at 85°C overnight. The 100 μ L reservoir was then made by punching a hole through the PDMS mold before bonding with the glass slide. The microwave sensor was then assembled with the sample holder using 50 μ m thick double-sided tape. An SMA connector was soldered to the reader, enabling the connection of the sensor to the VNA (MS2028C Anritsu). The transmission coefficient (S_{21}) was recorded over a frequency range of 600 MHz to 750 MHz with 4001 data points. Calibration was performed using the SOLT method with a standard kit to ensure high accuracy of the measurements.

To ensure measurement accuracy and mitigate potential sources of error, several precautions were taken during the experimental setup. Electromagnetic interference was minimized by conducting all measurements in an environment free from active electromagnetic-emitting devices. For real-world applications, potential shielding solutions, such as electromagnetic enclosures or filters, could be employed to further isolate the sensor from external noise. Additionally, to account for environmental variations, the resonance frequency shift was always determined relative to an initial baseline measurement of each sample. This approach helped correct for minor fluctuations in the permittivity of the host medium, ensuring consistency across measurements. Calibration was regularly performed, despite the fixed hardware setup, to maintain consistent performance across all tests.

5.5 Experimental Results and Discussions

The optimized sensor was used to monitor the size and concentration of MP particles in Tween20 solutions using the experimental setup shown in Figure 5.5. Each experiment began with calibrating the VNA, followed by connecting it to the sensor chip and securing the entire setup to the testing table to prevent any movement, which could affect measurement accuracy. Next, 100 μ L of the MP sample solution was pipetted into the reservoir, and the VNA started recording the S_{21} parameter at specific time intervals. After each

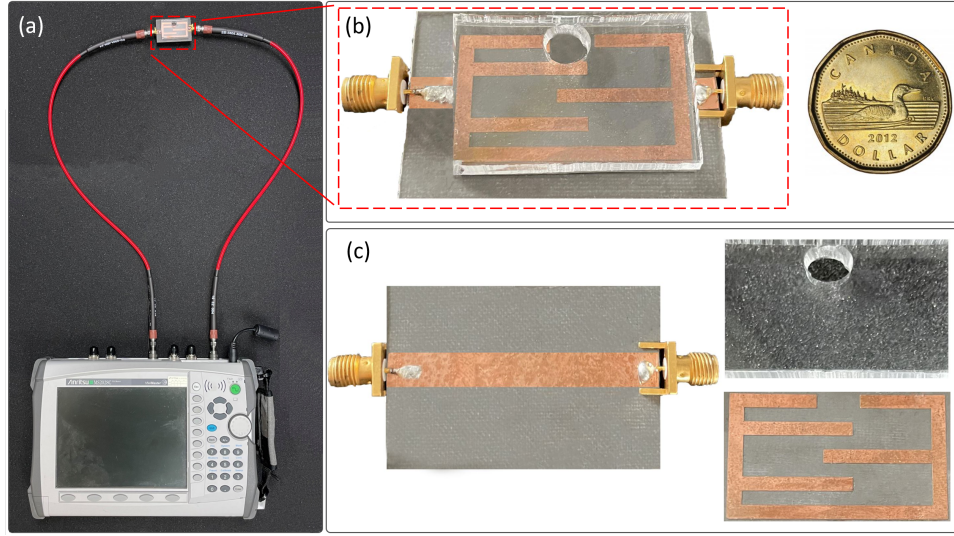


Figure 5.5: (a) Experimental setup for microplastic deposition monitoring. The characterization process of the sensor involves using a VNA, a device that measures and analyzes the electrical properties of the sensor, (b) zoomed view of the sensor with a Canadian dollar coin included for scale, (c) detailed view of the fabricated microwave reader, tag resonator, and PDMS container.

measurement, the reservoir (a hybrid structure of PDMS and glass) is replaced with a new one, and the experiment was repeated. For each set of conditions, the experiment was repeated at least three times to ensure repeatability.

To validate that the deposition of MPs dynamically influences the resonance frequency, three sample solutions containing 1,000k Particles/L of MP particles with three sizes of MP particles, 20 μm , 70 μm , and 275 μm , respectively, were monitored continuously for a period of 5 hours. The resonance frequency shift for each size of MP particles is shown in Figure 5.6. The deposition of MP particles was completed within one hour, which was used as the measurement period used for the rest of the experiments at two other concentrations including 100k Particles/L and 10,000k Particles/L.

To quantitatively describe the deposition trend, the data for each MP size were fitted to an exponential model, as detailed in the supplementary file. The extracted growth rates from the fitted models indicate that the 20 μm particles had the smallest slope of 0.03000, the 70 μm particles had slope of 0.03503, and the 275 μm particles had the largest slope of 0.03690. Additionally, the early linear phase duration and its slope were analyzed.

The linear phase was defined as the period until the deposition process reached 90% of its maximum resonance frequency shift. The results show that the 20 μm particles had a linear phase duration of 76.75 min, the 70 μm particles had 65.73 min, and the 275 μm particles had 62.39 min. The average slopes in this phase confirmed the size-dependent deposition trend, with the 20 μm particles at 6.08 kHz/min, the 70 μm particles at 23.68 kHz/min, and the 275 μm particles at 120.80 kHz/min.

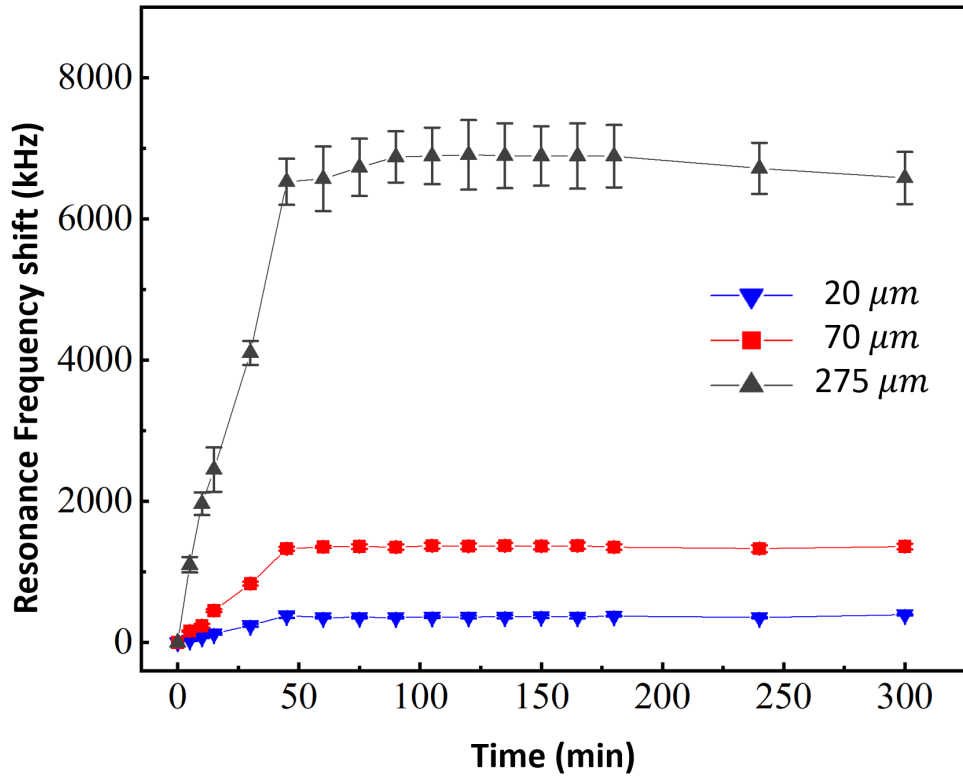


Figure 5.6: Deposition monitoring of microplastic samples with varying sizes and constant concentration of 1000 k Particles/L, over 5 hours with 17 time intervals, demonstrating size-dependent slope variation. The findings reveal that the samples consisting of larger microplastics exhibited steeper slopes in comparison to those containing smaller microplastics.

After finding a correlation between size and growth rate, further investigation was conducted on different concentrations for each size to assess their impact on deposition behavior. First, linear fitting was applied to the data for each MP size, as detailed in the

supplementary file. The statistical results indicate that for the 20 μm MP particles, the linear slope increased from 6.54 kHz/min at 100k Particles/L to 8.33 kHz/min at 1000k Particles/L and reached 11.04 kHz/min at 10,000k Particles/L. For the 70 μm MP particles, the slopes were measured at 20.81 kHz/min for 100k Particles/L, 28.87 kHz/min for 1000k Particles/L, and significantly increased to 65.87 kHz/min for 10,000k Particles/L. The largest 275 μm particles showed the highest deposition rates, with slopes of 75.38 kHz/min at 100k Particles/L and a remarkable increase to 146.18 kHz/min at 1000k Particles/L. The variation in error among the three types of curves observed in Fig. 6 can be attributed to differences in particle interactions within the sensing region. For smaller particles, interactions remain minimal, leading to more consistent and repeatable measurements. However, as particle size increases, the range of particle sizes is wider (e.g., 250 to 300 μm), making variability more pronounced. Additionally, since larger particles occupy a greater proportion of the reservoir volume, the chances of particle-particle interactions increase, leading to higher variability in deposition dynamics and, consequently, resonance frequency shifts. This effect is reflected in the larger error bars associated with larger particles.

Building on these statistical findings, the qualitative trends in resonance frequency shift further illustrate the impact of particle size on deposition behavior. The resonance frequency shift of the 20 μm MP particles exhibited a notably shallower slope on average compared to the larger 70 μm and 275 μm particles. It is worth noting that even the steepest slope observed among the 20 μm particle samples did not surpass the lowest slope found in the 70 μm particle samples, clearly distinguishing these two size categories.

Moving on to the 70 μm MP samples, they exhibited a steeper slope in the resonance frequency shift compared to the 20 μm particle samples, but still fell short of the 275 μm samples in this aspect. Furthermore, the steepest slope observed among the 70 μm particle samples did not exceed the mildest slope seen in the 275 μm particle samples, further reinforcing the consistent trend that larger MPs result in a more pronounced slope in their resonance frequency shift over time.

Lastly, the resonance frequency shift of the 275 μm MP samples exhibited the steepest average slope among all the size categories. This supports the trend observed in our study, where larger MPs consistently showed a greater slope in their resonance frequency shift over time. It is important to note that the results for 10,000k particles/L concentration with 275 μm size particles have been excluded from Figure 5.7, as the excessive volume of the MPs relative to the reservoir size rendered the data impractical.

As shown in Figure 5.7, the system is capable of determining the concentration of 100k Particles/L for all three sizes of MP particles. In this study, we considered three size

categories, with one (275 μm) significantly larger than the other two (20 μm and 70 μm), mainly due to the cost considerations. As shown in Fig. 6, the system can distinguish the two small sizes of MP particles, indicating its ability to differentiate particles within the range of 70 μm to 275 μm . The choice of 275 μm particles as the upper size limit is based on the studies by Colson et al. [62], which explored particles ranging from 300 μm to 1000 μm . Furthermore, the current reservoir volume limits the evaluation of particles larger than this size.

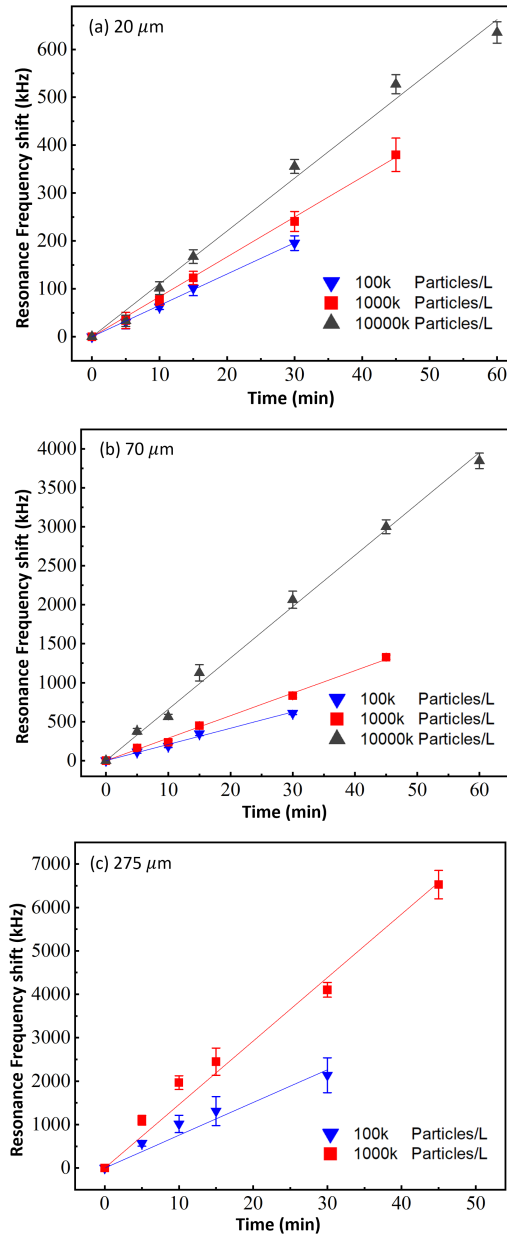


Figure 5.7: Resonance Frequency Shift of Microplastic Samples with Varied Concentrations and Sizes: The resonance frequency shift of the samples was measured for three different concentrations: 100k Particles/L, 1,000k Particles/L, and 10,000k Particles/L. The samples contained microplastics of three different sizes: 20 μm , 70 μm , and 275 μm . Specifically: The resonance frequency shift of the sample containing microplastics with a size of (a) 20 μm , (b) 70 μm , and (c) 275 μm .

As shown in Figure 5.8(a), temperature-dependent experiments were conducted at 10°C, 20°C, and 30°C using polyethylene particles within the 250–300 μm size range (average 275 μm) at a concentration of 100k Particles/L. The deposition process was monitored over a 30-minute period at 5 time intervals: 0 min (initial), 5 min, 10 min, 15 min, and 30 min. The experiments were performed in Tween 20 and deionized (DI) water with a 0.1% concentration to evaluate the effect of temperature on particle accumulation.

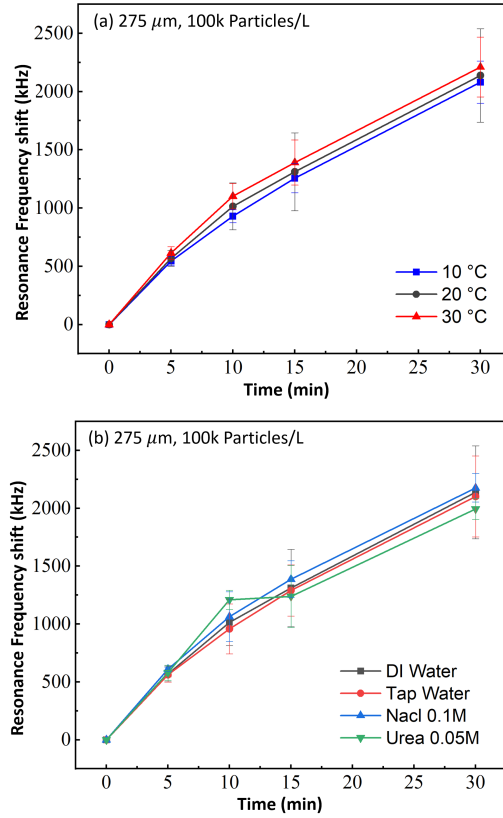


Figure 5.8: Resonance frequency shift as a function of time for 275 μm polyethylene (PE) microplastic particles at a concentration of 100k particles/L. (a) Effect of temperature (10°C, 20°C, and 30°C) on resonance frequency shift, showing higher shifts at elevated temperatures. (b) Influence of different host mediums (DI water, tap water, 0.1 M NaCl solution, and 0.05 M urea solution) on resonance frequency shift, demonstrating minimal variations across different mediums.

The results showed that temperature had a moderate effect on the resonance frequency shift. At the final measurement time of 30 minutes, the average resonance frequency shift

was 2080.1 ± 181.9 kHz at 10°C , 2137.7 ± 401.6 kHz at 20°C , and 2210.4 ± 256.6 kHz at 30°C . To reduce the temperature-induced variations in resonance frequency, a multiplexed temperature sensor can be incorporated into the system. By recording and compensating for real-time temperature fluctuations, the system can eliminate thermal effects, ensuring more accurate microplastic detection across different environmental conditions. The effect of different host mediums on the resonance frequency shift was investigated to assess whether variations in water composition influence microplastic deposition, as illustrated in Figure 5.8(b). The tested host mediums included deionized (DI) water, tap water, NaCl solution (0.1 M), and urea solution (0.05 M). The experiments were conducted using polyethylene particles within the 250–300 μm size range (average 275 μm) at a concentration of 100k Particles/L. The deposition process was monitored over a 30-minute period, with measurements recorded at 5 time intervals: 0 min (initial), 5 min, 10 min, 15 min, and 30 min. The experiments were performed in Tween 20 and DI water with a 0.1% concentration. The results indicate that the choice of host medium had a negligible effect on the resonance frequency shift. At 30 minutes, the measured frequency shift was 2137.7 ± 401.6 kHz in DI water, 2101.5 ± 349.3 kHz in tap water, 2175.5 ± 122.8 kHz in NaCl solution, and 1993.5 ± 88.8 kHz in urea solution. While minor differences were observed, they remained within the expected range of standard deviation, indicating that the host medium composition does not significantly alter the deposition behavior of microplastics. This observation is further reinforced by the fact that the resonance frequency shift is defined as the difference between the initial time and the measured value at each interval for each sample separately. This means that any variations in the host medium do not introduce a systematic bias in the results, as the measurement is always relative to the initial state of the same sample. As a result, the detection method remains robust and reliable across different environmental conditions, ensuring that microplastic accumulation can be effectively monitored regardless of the water composition.

To further investigate the ability of the system to differentiate between particle types, an experiment was conducted using polyethylene and soda lime glass particles, as shown in Figure 5.9. Both particle types had a size range of 250–300 μm (average 275 μm) and were tested at a concentration of 1000k Particles/L. The deposition process was monitored over a 300-minute period, with measurements recorded at 17 time intervals, ranging from 0 min (initial) to 300 min. The results indicate that the slope of the linear phase is nearly identical for both polyethylene and soda lime glass, suggesting that their early accumulation behavior follows a similar trend. Specifically, the slope of the linear phase for polyethylene was 146.68 kHz/min, while for soda lime glass, it was 143.83 kHz/min. However, after reaching saturation, the resonance frequency shift differed between the two materials, with polyethylene reaching 6579.57 kHz, whereas soda lime glass reached 6458.56 kHz. This

variation is attributed to the difference in dielectric constants between polyethylene and soda lime glass, affecting their final resonance behavior. While these results confirm that the system can differentiate between the two particle types, it is important to emphasize that this study focuses on bulk monitoring rather than individual particle identification. By referencing a calibration table of different particle types, the system can assess which particle type and size the overall sample most closely resembles. This approach allows for a general classification of microplastic contamination, making it a practical and effective tool for large-scale environmental assessments, where precise identification of individual particles is not necessary.

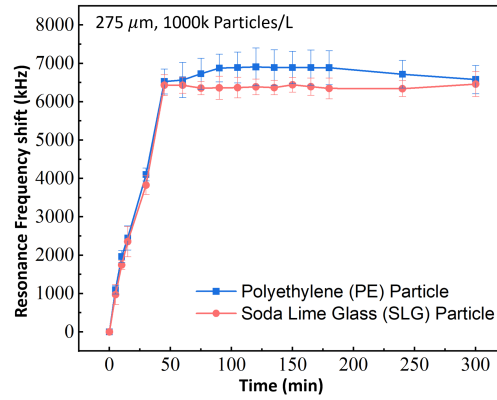


Figure 5.9: Resonance frequency shift as a function of time for 275 μm polyethylene (PE) and soda lime glass (SLG) particles at a concentration of 1000k particles/L. The PE particles exhibit a higher final resonance frequency shift compared to SLG particles, indicating a material-dependent response.

5.6 Limitations and Future Outlook

Electrical sensing methods show great promise for on-field MP detection, offering advantages such as compact design, scalability, and real-time monitoring capabilities. However, these technologies are still in their early stages, and ongoing advancements are working to overcome the existing challenges. Many current methods provide valuable capabilities for MP detection, and future developments are expected to enhance their ability to simultaneously determine particle size and concentration. Additionally, efforts are underway to simplify sample preparation, optimize flow control, and reduce instrumentation costs,

Table 5.1: Performance Comparison with State-of-the-Art

	Measurement Principle	Applied Design Method	Size Range	Size Estimation	Concentration Estimation
[135]	HFIS ¹	NEMS Mass Spectrometry	100 nm	N	Y
[64]	HFIS ¹	Nano Electrode Arrays	50-500 nm	Y	Y
[133]	EIS ¹	Impedance Sensor Array	2.8 μm	Y	Y
[134]	EIS ²	Concentric Circular SRR	20 μm	Y	Y
[138]	RMS ³	CPW and SRR	10-20 μm	Y	Y
[139]	RMS ³	Mems Resonator	25 μm	N	Y
[81]	RMS ³	Spiral Resonator	20-70 μm	Y	Y
[140]	RMS ³	Circular SRR	20-70 μm	N	Y
[63]	RMS ³	Multi CSRR	30-80 μm	Y	Y
[66]	RMS ³	LC Resonator	50-500 μm	N	Y
[65]	DS ⁴	Transmission Line	N.A	N	Y
[62]	EIS ²	Flow-Through Cavity	300-1000 μm	Y	Y
This Work	RMS ³	Sensitivity-enhanced Split Ring Resonator	20-300 μm	Y	Y

* 1. High Frequency Impedance Spectroscopy, 2. Electrical Impedance Spectroscopy, 3. Resonance Microwave Spectroscopy, 4. Dielectric Spectroscopy

making these approaches increasingly viable for large-scale environmental monitoring. The proposed method in this work is also not an exception and has its own limitations. While the proposed microwave sensing platform provides a low-cost, real-time approach for MP size and concentration estimation, it primarily performs bulk monitoring and does not differentiate individual polymer types with high specificity. Environmental factors such as temperature fluctuations may introduce measurement uncertainties. The detection method relies on the gravitational settling of MPs, which may prolong measurement times, particularly for smaller particles, and the shape of the particles might also affect the deposition. Additionally, while the sensor covers a broad range of MP sizes (20–300 μm), further improvements are needed to extend detection capabilities to particles smaller than 20 μm , which are increasingly relevant in environmental studies. A cost-benefit analysis highlights the affordability and practicality of novel sensors with the potential of field deployable compared to conventional spectroscopic methods, such as FTIR and Raman spectroscopy, which typically cost between 500 to 1000 CAD for the analysis of just 500 mL of a sample, depending on the technique used [277]. While these spectroscopic methods offer high accuracy in polymer identification, their high operational costs and extensive sample preparation requirements make them less feasible for large-scale and routine environmental monitoring. In contrast, our microwave-based approach provides a cost-effective alternative with

significantly lower operational expenses, enabling broader accessibility for environmental studies and routine monitoring applications. Future research should focus on enhancing the specificity of plastic type identification by integrating complementary detection techniques or leveraging machine learning for advanced classification. Conducting field studies in diverse water environments will help refine calibration models and improve accuracy under real-world conditions. Optimizing the sensor structure and signal processing techniques could enable detection of smaller MPs and nanoplastics. Developing a flow-based sensing system would enhance measurement efficiency, enabling continuous monitoring in dynamic aquatic environments. Additionally, a customized resonator-reservoir design tailored for specific size categories could significantly improve the limit of detection and sensitivity of the sensor. This approach would allow for optimized measurement conditions for different particle size ranges, enhancing overall performance and applicability. Lastly, integrating automated data processing and remote sensing technologies could support large-scale, real-time MP pollution assessments, making the sensor a more versatile tool for environmental monitoring. Both bulk monitoring and single-particle monitoring approaches offer distinct advantages and drawbacks and can be used in a complementary manner. Bulk monitoring enables faster analysis and requires simpler equipment, making it suitable for large-scale environmental assessments where rapid detection is crucial. However, it lacks the resolution needed for precise particle characterization. On the other hand, single-particle monitoring provides detailed insights into individual MPs, including size, concentration, and type, but requires precise flow control and more complex instrumentation. By integrating both techniques, a comprehensive monitoring strategy can be established, leveraging the speed and simplicity of bulk monitoring with the detailed characterization capabilities of single-particle methods.

5.7 Uncertainty Analysis

The reliability of the proposed sensing platform and its experimental results is subject to various sources of uncertainty, which can be broadly categorized into systematic and operational errors. This section provides an overview of these factors to clarify the limitations of the current system and offer a foundation for future refinement.

Systematic errors originate from the instrumentation and fixed design characteristics of the sensor platform. A major contributor is the resolution and calibration accuracy of the vector network analyzer (VNA), which, although capable of fine measurements, has a practical sensitivity limit around 100 kHz. This introduces uncertainty when interpreting small frequency shifts, especially for low-concentration or small-size microplastic (MP)

particles. Temperature fluctuations are another source of systematic error. Despite efforts to test under controlled temperatures (10–30°C), minor environmental variations can still influence the permittivity of both the host medium and the MPs, subtly altering the resonance behavior. Additionally, the fixed structure of the sensor, particularly the thickness and alignment of the PDMS-glass reservoir, the spacers, and the air gaps between layers, can impact electromagnetic field distribution and sensitivity. Fabrication tolerances, such as slight variations in etching or bonding quality, also affect repeatability and introduce inherent deviations across test cycles. Furthermore, SMA connector quality, soldering consistency, and cable shielding influence signal transmission and can introduce parasitic noise, further affecting measurement accuracy.

Operational errors, on the other hand, are introduced during sample preparation, handling, and experimental execution. The homogeneity of the particle suspension is a critical factor; despite the use of Tween 20 as a surfactant, MPs may still aggregate or settle unevenly during preparation, leading to non-uniform distributions within the sensing region. Pipetting inaccuracies, especially during dilution and sample loading, can lead to unintended concentration deviations. Human error is also a consideration in assembling the sensor, aligning the reservoir, replacing components, and conducting repeat measurements. Density values provided by the particle supplier are used as fixed inputs for theoretical calculations, but actual variations in MP density may differ slightly, contributing to uncertainty in the deposition rate and size estimation. Additionally, the gravitational settling behavior of MPs is influenced not only by their size but also by their surface charge and hydrodynamic properties, which are difficult to control and may vary between batches.

Taken together, these sources of error highlight both the challenges and the opportunities for improving the platform’s performance. While the current sensor demonstrates promising sensitivity for simultaneous MP size and concentration detection, further improvements in fabrication precision, temperature compensation, and sample handling protocols will be essential to enhance its robustness and reliability in real-world applications.

5.8 Summary

This study presents a novel, cost-effective sensor designed for on-site monitoring of both the size and concentration of MP particles. The sensor features a sensitivity-enhanced planar microwave design and operates as a tag-reader coupled structure, with the tag resonator energized through electromagnetic coupling with the planar microwave reader. To enhance coupling strength and sensitivity, an IDC was integrated with the traditional SRR tag

resonator. The sensor consists of a disposable reservoir holder which enables multiplex testing without cross-contamination, a critical need for on-site applications.

The sensor performance is demonstrated through the monitoring of three MP particle sizes - 20 μm , 70 μm , and 275 μm - at concentrations of 100k, 1000k and 10,000k Particles/L. It successfully detects 20 μm MP particles at a concentration of 100k Particles/L and differentiates between 20 μm and 70 μm particle sizes. This study represents a significant step forward in addressing the critical issue of MP pollution and advancing sustainable environmental solutions. Furthermore, it paves the way for the development of disposable microwave sensors, offering substantial opportunities in smart environmental monitoring and water quality assessment.

5.9 Supplementary Information

Supplementary information is included in this section to complement the main findings, providing additional details on methodologies. This supplemental content offers deeper context and technical insights, enhancing the overall understanding of the work.

5.9.1 Forces Acting on a Sphere Moving Through a Viscous Fluid

The predominant forces acting on a microsphere moving through a viscous fluid include gravitational force, buoyant force, and viscous drag force [272], [273], [274]. These forces dictate the time-dependent displacement of microspheres in an aqueous environment. Figure 5.10 illustrates these forces:

Weight of the Sphere (F_w): The gravitational force acting downward, calculated as:

$$F_w = mg$$

where m is the mass of the sphere and g is the acceleration due to gravity.

Buoyant Force (F_b): The upward force exerted by the fluid, proportional to the displaced fluid's weight:

$$F_b = mg \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}$$

Viscous Drag Force (F_v): The resistive force caused by the fluid's viscosity as the sphere moves through it, given by Stokes' law:

$$F_d = 6\pi\mu Rv$$

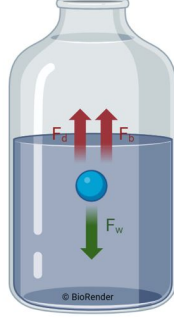


Figure 5.10: Forces on a sphere in a viscous fluid: Gravitational (F_w), buoyant (F_b), and viscous drag (F_d) forces acting during motion.

where:

- μ : Dynamic viscosity of the fluid
- R : Radius of the sphere
- v : Velocity of the sphere relative to the fluid

The net force acting on the sphere can be expressed as:

$$F = ma = m \frac{dv}{dt} = F_w - F_b - F_d$$

or equivalently:

$$F = mg - mg \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}} - 6\pi\mu Rv$$

Figure 5.11 extends this analysis to derive the time-dependent velocity and displacement of the sphere. The derived equations are:

1. Velocity of the Sphere ($v(t)$)

$$ve^{\frac{6\pi\mu R}{m}t} = \frac{mg}{6\pi\mu R} \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}} \right) e^{\frac{6\pi\mu R}{m}t} + A$$

$$\text{At } t = 0, \text{ we have } v(0) = 0 \Rightarrow A = -\frac{mg}{6\pi\mu R} \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}} \right).$$

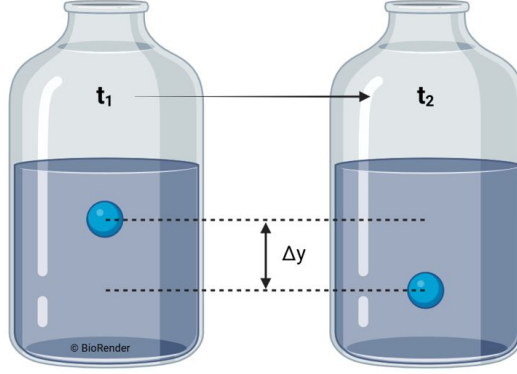


Figure 5.11: Displacement of a microsphere (Δy) in a viscous fluid over time (t_1 to t_2).

$$v(t) = \frac{mg}{6\pi\mu R} \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right) - \frac{mg}{6\pi\mu R} \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right) e^{-\frac{6\pi\mu R}{m}t}$$

2. Displacement of the Sphere ($y(t)$)

$$y(t) = \int_0^t v(t) dt$$

$$y(t) = \frac{mg}{6\pi\mu R} \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right) t - \left(\frac{m}{6\pi\mu R}\right)^2 g \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right) e^{-\frac{6\pi\mu R}{m}t} - \left(\frac{m}{6\pi\mu R}\right)^2 g \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right)$$

$$y(t) = \frac{2\rho_{\text{particle}}r^2}{9\mu} g \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right) t$$

$$- \frac{4\rho_{\text{particle}}^2r^4}{81\mu^2} g \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right) e^{-\frac{9\mu}{\rho_{\text{particle}}r^2}t}$$

$$- \frac{4\rho_{\text{particle}}^2r^4}{81\mu^2} g \left(1 - \frac{\rho_{\text{fluid}}}{\rho_{\text{particle}}}\right)$$

where:

- m : Mass of the particle [kg]
- r : Radius of the sphere particle [m]
- g : Acceleration due to gravity [m/s²]
- μ : Dynamic viscosity [kg·m⁻¹·s⁻¹, or Pa·s]
- ρ_{fluid} : Density of the fluid [kg/m³]
- ρ_{particle} : Density of the particle [kg/m³]

These equations demonstrate that the displacement of the particle depends on its size, with larger particles moving more rapidly than smaller ones. This theoretical analysis supports the experimental observation that deposition rates vary with particle size, as described in the main text. The PE particles used in this study have a density of 1 g/cm³, while the host medium, consisting of 0.1% Tween 20 in deionized water, has an approximate density of 0.997 g/cm³ and a dynamic viscosity of about 0.9 mPa·s at room temperature.

5.9.2 Exponential Fitting Analysis of Figure 6 in the Manuscript

For the fitting analysis of the deposition data, we applied an exponential growth function of the form:

$$f(t) = A(1 - e^{-kt})$$

where A represents the saturation limit of the resonance frequency shift, and k is the growth rate constant that characterizes the speed of deposition.

The extracted fitting parameters for each particle size are as follows:

Size	A	k
20 μm	383.75702011693494	0.0299999999999999943
70 μm	1394.4843002348325	0.035032497219834875
275 μm	6964.095149010965	0.03690467212983636

These values confirm the size-dependent deposition behavior, where larger particles exhibit a steeper initial deposition slope and a higher overall resonance frequency shift. The fitted function accurately represents the experimental data and provides a quantitative description of the deposition dynamics for different microplastic sizes.

5.9.3 Linear Fitting Analysis of Figure 7 in the Manuscript

To evaluate the deposition behavior of microplastics under different conditions, a linear fitting model of the form:

$$y = a + b \cdot x$$

was applied to the data in Figure 7, where y represents the resonance frequency shift and x is the deposition time. Since all plots pass through the origin (0,0), the intercept a was set to zero, leaving only the slope b , which represents the rate of frequency shift per unit time.

The calculated slope values for each condition are presented in the table below:

Particle Size	Concentration (Particles/L)	Slope (kHz/min)
20 μm	100k	6.54324
	1000k	8.33226
	10000k	11.03769
70 μm	100k	20.81361
	1000k	28.86893
	10000k	65.87392
275 μm	100k	75.38342
	1000k	146.17598

The results demonstrate a clear size-dependent trend, where larger microplastics exhibit steeper deposition slopes across all tested concentrations. Additionally, higher particle concentrations lead to an increased slope, indicating a faster accumulation rate. This confirms that both particle size and concentration significantly influence the deposition process, supporting the sensitivity of the detection system in distinguishing variations in microplastic accumulation dynamics.

5.9.4 Sensor Dimensions

The dimensions of the sensor are illustrated to provide a detailed overview of its structural design:

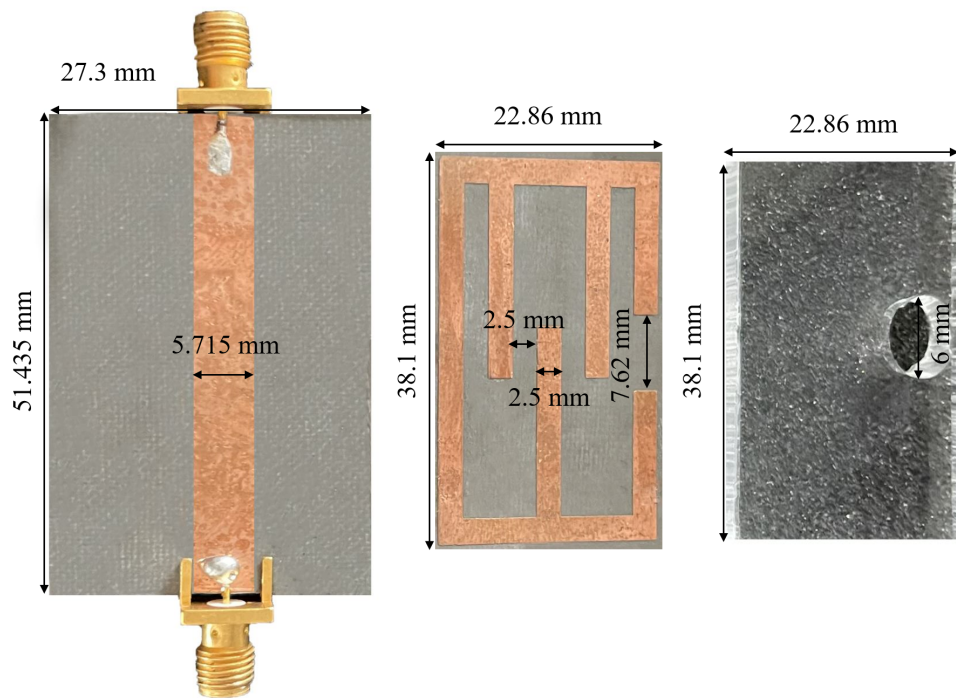


Figure 5.12: The fabricated sensor including the microwave reader, resonator, and reservoir with labeled dimensions.

Chapter 6

An AI-Powered Microwave-Microfluidics Platform for Comprehensive Microplastic Detection: Multi-variable Analysis of Size, Concentration, and Type

This chapter presents a microwave-microfluidic sensor with a single-particle monitoring feature for detecting microplastic particles in aqueous environmental samples. The preliminary results of this work have been accepted for presentation at the *IEEE International Symposium on Antennas & Propagation Conference*. Additionally, the comprehensive findings from this study will be submitted for publication in *ACS Environmental Science & Technology*, contributing to the ongoing efforts in advancing environmental monitoring technologies for microplastic detection.

Maziar ShafieiDarabi, Amirhossein YazdaniCherati, Zahra Abbasi, and Carolyn L. Ren, "SRR-Based Disposable Microwave-Microfluidic Sensor for Assessing Liquid Carrier Influence on Microplastic Detection," 2025 IEEE International Symposium on Antennas & Propagation Conference - APS 2025, Ottawa, ON, CA, 2025.

Maziar ShafieiDarabi, Amirhossein YazdaniCherati, Nikhil V. Giri, Samarpreet Bhatti, Zahra Abbasi, and Carolyn L. Ren "AI-Powered Microwave-Microfluidic Platform for Microplastic Detection" will be submitted to *ACS Environmental Science & Technology*, 2025.

Microplastic (MP) contamination is a growing environmental concern, with significant

implications for ecosystems and human health. Traditional methods for detecting and quantifying MPs are often limited by factors such as complexity, particle size range, and the inability to monitor in real-time. In this paper, we propose a novel microwave microfluidic platform for the flow-through quantification of MPs, focusing on single-particle monitoring. This method simplifies the detection process and provides a cost-effective solution for detecting MPs in the size range of 20 μm to 300 μm . The platform begins with prefiltration of environmental samples using a sieve stack method to sort particles into defined size ranges. Tailored disposable microfluidic chips are then integrated with a planar microwave reader to perform the detection. The raw sensor data is processed using a K-nearest neighbors (KNN) algorithm to classify the particles based on their resonance profiles, while the prefiltration step determines particle size. Additionally, the platform’s single-particle detection capability enables accurate quantification of particle concentration. This approach offers a practical solution for real-time monitoring and precise detection of MP contamination, with potential applications in environmental monitoring and public health research.

6.1 Introduction

Microplastics (MPs), defined as polymer-based particles smaller than 5 mm, are increasingly recognized as a significant environmental concern, found in diverse ecosystems such as oceans, freshwater sources, soils, and even air [278], [244], [279], [280], [245]. Their proliferation is linked to the staggering global plastic production of approximately 320 million tons annually [22], and a low recycling rate of just 9%-14% [23]. MPs pose serious threats to water quality and human health [15], with documented presence in drinking water [16], [17] and human blood [18], highlighting the need for further research into their toxicological effects and exposure pathways. While primary MPs are intentionally manufactured for products such as cleaning agents, and cosmetics, secondary MPs arise from the breakdown of larger plastic debris, presenting regulatory challenges [16], [281]. Despite the widespread acknowledgment of MPs, assessing their ecological risks remains complex due to difficulties in measurement and a lack of robust data, particularly in aquatic environments [24], [25], [26]. Enhanced monitoring techniques are urgently needed to effectively quantify MPs and understand their transport dynamics, crucial for mitigating this pressing environmental issue.

There are multiple established methods for analyzing environmental MPs in the 1 μm to 5 mm size range [242], [30]. Common techniques, such as visual inspection [27], [242], are prone to misidentification errors [24], [282], even when using Nile Red [58], [59], due

to interference from biological particles, which can lead to detection inaccuracies [60], [61]. Other techniques are Fourier-transform infrared (FTIR) spectroscopy [35], [43], Raman spectroscopy [35], [48], and pyrolysis gas-chromatography mass spectrometry [38], [283]. While these methods are reliable for quantifying MPs, especially at sub-ppm concentrations, they often require complex and costly laboratory setups, limiting their application in the field settings [25], [27]. Alternative approaches, such as portable pyrolysis-mass spectrometry, have been proposed to address these limitations; however, they also encounter challenges, including interference from biological materials [284]. Current sampling protocols predominantly rely on time-intensive laboratory processes, often involving chemical pretreatment and extensive sample preparation, which can delay results and compromise representativeness [39], [44]. This highlights the urgent need for the development of field-deployable sensors that facilitate real-time monitoring of MPs, allowing for improved spatial coverage and sampling frequency without the extensive preparation traditionally required.

Electrical sensing techniques could be a promising candidate for on-field detection of MPs, thanks to their compact detection systems and scalability. The electrical sensing techniques currently found in the literature can be classified into several categories, including resonance microwave spectroscopy (RMS), dielectric spectroscopy (DS), high-frequency impedance spectroscopy (HFIS), and electrical impedance spectroscopy (EIS). These techniques vary in how they interact with the electrical characteristics of materials, each offering distinct advantages for the MP detection [62], [63], [64], [65].

While most electrical sensing techniques operate on similar principles, RMS has emerged as a particularly promising technique for on-field MP detection. Its broader frequency range, typically spanning from 0.1 to 100 GHz, allows for more precise and versatile detection of different types of particles. By differentiating materials based on their permittivity, microwave sensing is highly effective in detecting MPs, which typically exhibit permittivity values in the range of 2 to 3.5, distinct from surrounding materials like water (~ 80), wet sediments (~ 10 -30), and blood (~ 50 -60) [66], [67]. Additionally, microwave sensors can be designed as compact, lightweight, and cost-efficient systems, offering durability and practicality for long-term real-world measurements [264], [265], [266], [267]. These innovations present a feasible solution for continuous monitoring across various applications [268], [269].

Electrical sensing methods for MP detection can generally be divided into two categories: bulk monitoring and single-particle monitoring. Bulk monitoring methods primarily focus on measuring the concentration of MPs, but they cannot provide information on particle size or type. A few techniques, such as spiral resonators and multi-complementary split-ring resonators (CSRR), can estimate particle size within a specific range, but they complicate the system by requiring the integration of physical filters [81], [63]. In contrast,

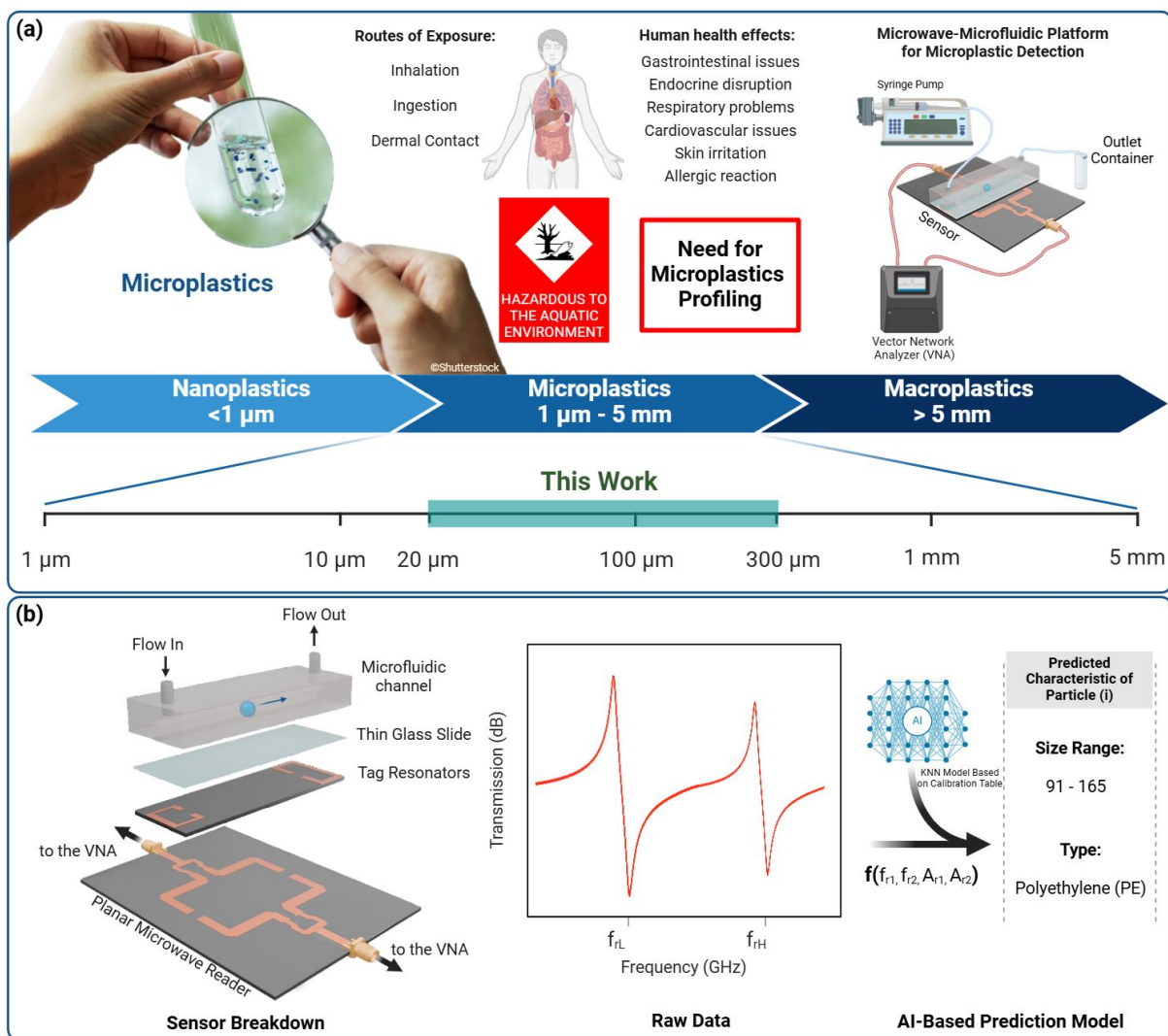


Figure 6.1: Overview of the Microwave-Microfluidic Platform for Microplastic Detection: (a) Microplastics (ranging in size from $1 \mu\text{m}$ to 5mm) are a growing environmental issue that poses risks to both human health and aquatic ecosystems. This underscores the importance of developing a platform for their characterization, alongside an overview of the developed microwave microfluidic sensing platform for microplastic detection in this work. (b) Sensor breakdown illustrating the components of the microwave microfluidic sensor, including the planar microwave reader, microfluidic channel, and tag resonators. The figure also presents the raw data transmission curve and an AI-based prediction model for determining the size and type of detected microplastic particles.

single-particle monitoring methods, like high-frequency impedance spectroscopy (HFIS) and electrical impedance spectroscopy (EIS) [62], [64], are capable of determining particle size, concentration, and type. However, they typically demand more complex systems to measure both low- and high-frequency responses, or they have limitations regarding the particle size range.

In this study, we propose a microwave microfluidic platform for single-particle monitoring, designed for the flow-through quantification of MPs. This method offers a simplified measurement setup while covering a broad particle size range from 20 μm to 300 μm . The sensing process begins with the prefiltration of the environmental sample using a sieve stack method, which categorizes particles into specific size ranges. Disposable chips are then designed for each size range, allowing integration with a microwave reader that is compatible with all the chips. The raw data from the sensing platform is processed using a KNN model to predict the type of each single particle, while the size range is determined through the prefiltration step. As this method involves single-particle detection, the concentration of particles can also be quantified. An overview of the developed microwave-microfluidic sensing platform is illustrated in Figure 6.1.

6.2 Materials and Methods

6.2.1 Materials.

In this study, various particles were selected to explore their properties and behavior under experimental conditions. Polyethylene microspheres were purchased from Cospheric LLC in four distinct size ranges: 250-300 μm , 106-125 μm , 63-75 μm , and 20-27 μm . Additionally, polystyrene particles (175-210 μm) and soda lime glass particles (250-300 μm) were obtained from Cospheric LLC. Brine shrimp eggs (180-300 μm) sourced from San Francisco Bay Brand were also included in the particle set. These particles were chosen for their diverse sizes and compositions, providing a comprehensive selection of materials to assess their behavior in the experiments. The polyethylene microspheres span a broad size range from 20 μm to 300 μm , demonstrating that our platform can detect and analyze a wide spectrum of particle sizes. Specifically, in the 165-300 μm range, four different types of particles were used to showcase the platform’s ability to differentiate between various particle types, further highlighting its versatility and precision in single-particle monitoring.

6.2.2 Fabrication of Microwave Microfluidic Sensing Platform.

The microwave microfluidic sensing platform consists of three main components: the microwave reader, tag resonators, and microfluidic channel. Each of these elements was fabricated using distinct processes to ensure precision and performance in detecting MPs.

The tag resonators and microwave reader were fabricated on RT/duroid 5880 laminate using a wet chemical etching process. The design for the resonators was first printed onto glossy photo paper and laminated onto a copper-coated polymer slide using a thermal laminator. This step transferred the design onto the copper surface. The patterned slide was then subjected to copper etching, followed by acetone washing to remove any remaining photoresist, completing the fabrication of the resonators. For tag resonators smaller than $100\text{ }\mu\text{m}$, a Nova machine from Voltera was used to print directly onto the RT/duroid 5880 laminate without copper coating, ensuring fine resolution and precision for these smaller components.

The microfluidic channels were fabricated using two different approaches, depending on the required channel dimensions. Channels larger than $50\text{ }\mu\text{m}$ were fabricated using a 3D printing technique, while those smaller than $50\text{ }\mu\text{m}$ were produced using a soft-lithography method. Both fabrication methods utilized PDMS (polydimethylsiloxane) for its biocompatibility, and optical transparency, making it an ideal material for microfluidic applications. For channels larger than $50\text{ }\mu\text{m}$, 3D-printed molds were used. These molds were designed using CAD software and fabricated with a Formlabs Form 3 resin-based 3D printer using Clear Resin (Formlabs). After printing, the molds were cleaned and post-cured to ensure structural stability and accuracy. PDMS was mixed with a curing agent in a 10:1 ratio, degassed for 30 minutes, and poured over the 3D-printed molds. The molds were then placed on a hot plate and cured overnight at 85°C to achieve complete polymerization. Once cured, the PDMS structures were carefully peeled off, and fluidic access holes were created using a biopsy punch. This approach offered scalability and flexibility for fabricating larger microchannels. For channels smaller than $50\text{ }\mu\text{m}$, a soft-lithography technique was employed. PDMS was mixed with a curing agent in a 10:1 ratio and degassed. The mixture was then molded against SU-8/silicon masters, which were cured at 95°C for two hours. After curing, the molds were cut out, and fluidic access holes were made using a biopsy punch. The PDMS chip and a thin glass slide were treated with oxygen plasma at 29.7 W and 500 mTorr for 30 seconds to create a strong, leak-proof bond between the PDMS and the glass. Once the tag resonators, microwave reader, and microfluidic channels were fabricated, they were assembled into a complete sensing platform. An SMA connector was soldered to the microwave reader, enabling seamless connection to a vector network analyzer (VNA) for real-time monitoring and

data collection. This assembly allowed for the integration of the microfluidic chip with the microwave sensing system, providing a compact and efficient platform for single-particle detection of MPs.

6.2.3 Sample Preparation.

The sample solutions used in this study consisted of a 0.1% Tween 20 solution with suspended particles. The preparation process began by creating the 0.1% Tween 20 solution, which was made by dissolving Tween 20 in boiling deionized water. To ensure thorough dispersion of the surfactant, a magnetic stir bar was employed. Once the Tween 20 solution was prepared, it was added to a vial containing the particles. The vial was then centrifuged for 5 minutes to ensure a homogeneous distribution of the particles within the suspension.

6.2.4 Experimental Procedure.

After centrifugation, the solution containing 0.1% Tween 20 and the suspended particles was transferred into a syringe, which was subsequently connected to a syringe pump. The pump was used to inject the sample solution into the microfluidic channel for analysis. For measurement, the resonance profile of the sensor was monitored using a VNA connected to the microwave reader. Data collection was automated through a Python-based code, with the resonance profile being recorded every 500 milliseconds. In parallel, the flow of particles through the microfluidic channel was visually inspected using a microscope. This visual inspection allowed us to track the number of particles flowing through the channel, which was then compared with the sensor's resonance data to assess the accuracy and performance of the sensor in detecting particles.

6.2.5 Particle Classification Using k-Nearest Neighbors.

For the 165 μm - 300 μm range, particle detections were classified as polyethylene, polystyrene, soda lime glass, or brine shrimp egg using the k-nearest neighbors (k-NN) classification algorithm. A 4-dimensional (4D) k-NN model was trained using the changes in two resonance frequencies within the resonance profile, along with the amplitude of S_{21} at each of these frequencies. The k-NN model was trained using datasets of known particles, implemented in Python, where the classification was performed based on four extracted features (Δf_{r1} , Δf_{r2} , ΔA_1 , ΔA_2). The model utilized three nearest neighbors ($k = 3$), with the default neighbor search method (auto-selected from ball-tree, kd-tree, or brute force), and a

Euclidean distance metric for similarity measurement. Additionally, data standardization was applied to normalize feature distributions, ensuring optimal performance. The trained k-NN model was then used to classify all detected particles based on their closest match in the feature space.

6.2.6 Particle Sizing and Pre-Filtration Method.

In this study, a vibratory sieve shaker from Cole-Parmer, model SS-3CP, was employed as a pre-filtration method to investigate the efficiency of this technique for size sorting of the particles. The sieve shaker uses a throwing motion with angular momentum to separate particles based on their size. With a measuring range of 165-300 μm , the shaker effectively sorted the particles into defined size categories, providing precise control over the particle distribution before introducing them into the microfluidic channel for analysis. This pre-filtration method was crucial for preparing the sample solution and ensuring the particles were within the desired size range for our experimentation.

6.3 Results and Discussions

6.3.1 Sensor Design and Simulations.

As shown in Figure 6.1 (b), the proposed sensing platform is a coupled reader-tag-based structure. It features two symmetric Wilkinson power dividers on the reader, which energize two distinct Split Ring Resonators (SRRs) integrated with a PDMS-based microfluidic channel through electromagnetic coupling. The channel width is specifically designed to align with the gap size of the SRRs, ensuring optimal interaction between the liquid under test and the electromagnetic field in the gap area. This design is highly effective due to the significant contrast in dielectric properties at the designed frequency range, such as the permittivity of polyethylene particles (~ 2.4) compared to water (~ 80). The microfluidic channel is positioned above the SRR gaps, so particles passing through the channel are directly exposed to the sensing hotspots, areas with high electric field intensity. The two independent resonances at different frequencies, resulting from the simultaneous excitation of the ports, provide valuable insights into the particles passing through. The approach proposed in this work not only improves selectivity by differentiating particles based on their electromagnetic responses but also enhances sensitivity by confining the resonators to the channel size, ensuring maximum exposure to the electric field. By integrating the

SRRs with the microfluidic channel, the system creates a disposable microwave-microfluidic sensing chip while keeping the measurement circuitry on the reader's side.

The simulation results, conducted using HFSS to analyze the performance of the proposed sensing platform, shown in Figure 2(a) demonstrate the presence of two distinct resonances in the frequency response, attributed to the two SRRs. The resonance frequency is given by $f_r \propto \frac{1}{\sqrt{LC}}$, where the smaller SRR corresponds to the higher frequency resonance ($f_{rH} = 2.617$ GHz), and the larger SRR corresponds to the lower frequency resonance ($f_{rL} = 1.227$ GHz). The power divider design on the reader ensures isolation between the output ports by precisely matching the impedance of the input and output branches while maintaining a physical separation of half a wavelength between the outputs. This configuration allows for the simultaneous monitoring of both sensing regions without interference, which is crucial for characterizing MPs and selecting the appropriate frequency range for optimal sensitivity. Figure 2(b) illustrates the electric field distribution on the two resonators at their respective resonance frequencies, further confirming the isolation between the two resonators and showing that each resonator is functioning at its designated resonance frequency.

6.3.2 Analysis of Resonance Profiles of Selected Materials.

Here, the resonance profiles of the sensor with double resonances were investigated by monitoring changes in both the resonance frequency and the amplitude of S_{21} at those resonance frequencies for four different materials: Polyethylene, Polystyrene, Soda Lime Glass, and Brine Shrimp Egg. As shown in Figure 6.3(a), the frequency shifts (Δf_r) at the two resonance frequencies, 1.2 GHz and 2 GHz, were recorded for each material. The data points, totaling 50 for each material, reflect the variation in frequency shifts as the materials pass through the microfluidic channel. Figure 6.3(b) illustrates the corresponding changes in the amplitude of S_{21} at these resonance frequencies.

The results reveal distinct patterns in the behavior of each material. Polyethylene and Polystyrene exhibit more substantial frequency shifts at both resonance frequencies, while Brine Shrimp Egg shows a smaller frequency shift. Soda Lime Glass falls in between, showing moderate frequency shifts. The changes in the S_{21} amplitude, as shown in Figure 6.3(b), follow a similar trend, with Polyethylene and Polystyrene exhibiting higher amplitude variations compared to Brine Shrimp Egg. These variations in resonance frequency and S_{21} amplitude provide insights into the electromagnetic properties of these materials and their interactions with the sensor, enabling the sensor to differentiate between particle types.

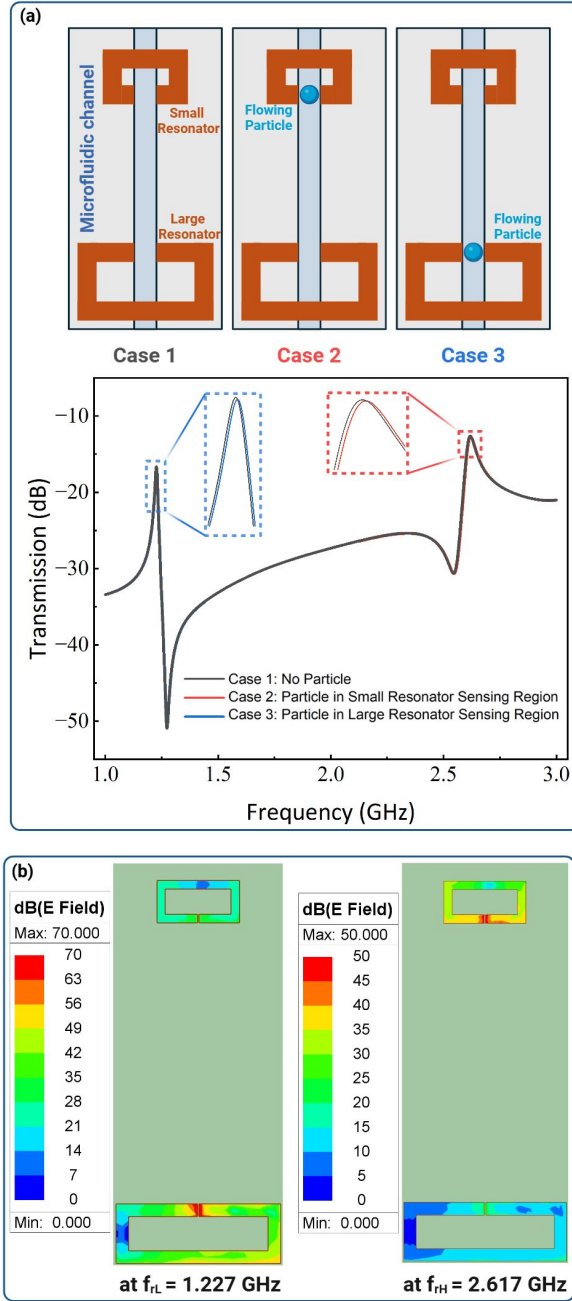


Figure 6.2: Simulation Results: (a) Transmission coefficient (S_{21}) for three cases: Case 1: No particle, Case 2: Particle in the small resonator sensing region, and Case 3: Particle in the large resonator sensing region. (b) Electric field intensity on the two resonators at their respective resonance frequencies.

The observed resonance behavior of each material suggests that the microwave microfluidic platform is capable of distinguishing various materials based on their unique resonance profiles. This ability to differentiate particles based on frequency shifts and amplitude changes is crucial for the development of field-deployable sensors for MP detection and environmental monitoring.

6.3.3 Accuracy of k-NN Model in Predicting Particle Type.

We also evaluate the performance of the 4D k-NN model, which was trained with the dataset discussed in the previous subsection. The model, as described in the Materials and Methods section, was trained using data from four particle types: Polyethylene, Polystyrene, Soda Lime Glass, and Brine Shrimp Egg, with 50 data points for each type.

To assess the accuracy of the model, a test set consisting of 10 data points for each particle type was used. The results show that the k-NN model demonstrated high accuracy in predicting the particle types. Specifically, for Polyethylene and Brine Shrimp Egg, all 10 test samples were correctly classified, indicating perfect prediction performance for these two materials. For Soda Lime Glass, the model correctly predicted 9 out of 10 test samples, achieving a prediction accuracy of 90%. However, for Polystyrene, the accuracy was slightly lower, with 8 out of 10 samples being correctly classified, resulting in an 80% accuracy rate.

Figure 6.4(a) illustrates the number of correct predictions (out of 10) for each particle type. These results highlight the effectiveness of the k-NN model in distinguishing between different particles based on their resonance profiles, with high accuracy achieved for most materials. This suggests that the developed microwave microfluidic sensing platform, in combination with the k-NN model, holds significant potential for accurate detection and classification of MPs.

6.3.4 Performance of the Microwave Microfluidic Sensing Platform Across Different Particle Size Ranges

In addition, we evaluate the performance of the microwave microfluidic sensing platform for detecting MP particles across different size ranges. Polyethylene was selected as the particle type, and measurements were conducted for four different size ranges: 165 μm - 300 μm , 91 μm - 165 μm , 51 μm - 91 μm , and 16 μm - 29 μm . The resonance profile was measured every 500 milliseconds, with a flow rate of 50 $\mu\text{L}/\text{min}$. At this flow rate, particles in the 250–300 μm size range required approximately 20 seconds to travel from the inlet

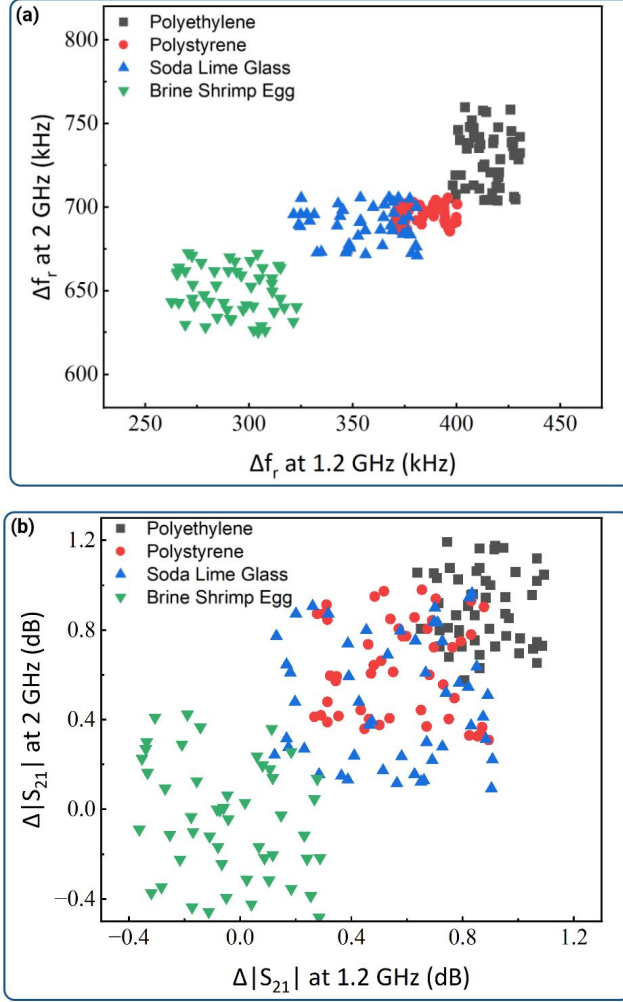


Figure 6.3: Changes in resonance frequency and amplitude of S_{21} for four different materials. (a) Frequency shifts (Δf_r) at 1.2 GHz and 2 GHz for Polyethylene, Polystyrene, Soda Lime Glass, and Brine Shrimp Egg, with 50 data points for each material. (b) Corresponding changes in the amplitude of S_{21} at the two resonance frequencies for the same materials.

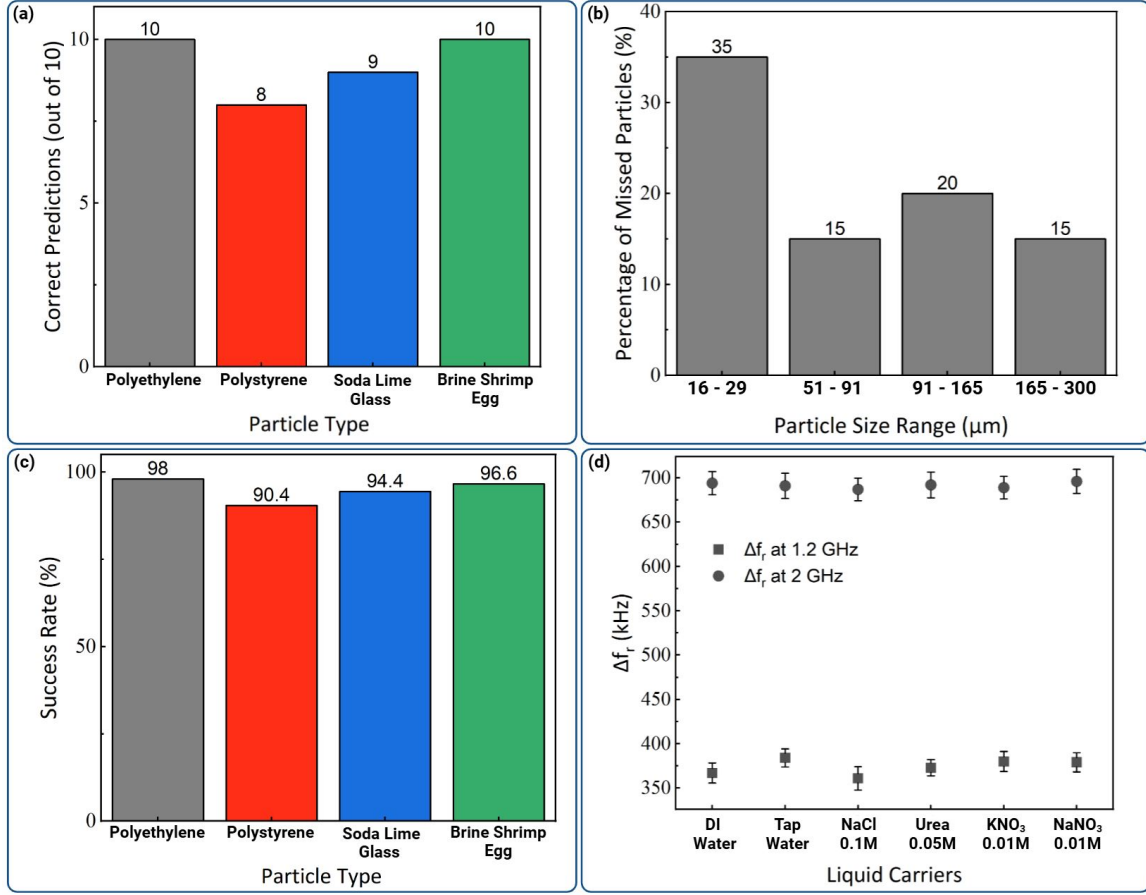


Figure 6.4: Performance evaluation of the microwave microfluidic sensing platform. (a) Number of correct predictions (out of 10) for each particle type predicted by the 4D k-NN model. The model achieved perfect classification for Polyethylene and Brine Shrimp Egg (10 out of 10 correct predictions), 9 out of 10 correct predictions for Soda Lime Glass, and 8 out of 10 correct predictions for Polystyrene. (b) Percentage of missed particles for each size range, based on a total of 20 reads (two sets of 10 particles per size range). The plot shows that smaller particles exhibit a higher percentage of missed detections. (c) Success rate (%) for each particle type, demonstrating the efficiency of the sieve shaker machine in sorting different materials. (d) Frequency shifts (Δf) at 1.2 GHz and 2 GHz for different liquid carriers. The plot shows the average resonance frequency shifts for each liquid carrier, illustrating the impact of the liquid carrier's dielectric properties on the sensor's performance.

to the outlet of the microchannel. Despite this relatively short measurement interval, the speed of the particles could still exceed the measurement time, leading to potential particle misses as they pass through the sensing region of each resonator.

Two sets of 10 particles were tested for each size range. For the 165 μm - 300 μm size range, in the first set, one particle was missed, while in the second set, two particles were missed. For the 91 μm - 165 μm range, two particles were missed in both sets. For the 51 μm - 91 μm range, two particles were missed in the first set, and one particle was missed in the second. In the smallest size range, 16 μm - 29 μm , we observed that three particles were missed in the first set, and four particles were missed in the second set.

Figure 6.4(b) illustrates the percentage of missed particles for each size range. These results suggest that while the sensing platform performs well across various size ranges, the accuracy of particle detection can be affected by the speed at which the particles flow through the sensing region. This limitation may result in missed detections, especially for smaller particles. Despite this, the platform still demonstrates significant potential for detecting and classifying MPs of different sizes in real-time.

6.3.5 Efficiency of Sieve Shaker for Particle Size Sorting Across Different Materials

In this experiment, we evaluate the efficiency of the sieve shaker machine in sorting MP particles within the size range of 165 μm to 300 μm . We used a total of 500 particles from each of the four selected materials: Polyethylene, Polystyrene, Soda Lime Glass, and Brine Shrimp Egg. The sieve shaker was set to its maximum power and run for one minute to sort the particles.

After the sorting process, the number of particles remaining on the top sieve (300 μm) was recorded. For Polyethylene, 10 particles remained on the 300 μm sieve, indicating an efficient sorting process. Soda Lime Glass had 28 particles left on the top sieve, while Polystyrene had 48, and Brine Shrimp Egg had 17 particles remaining. These results show the varying efficiency of the sieve shaker in handling different materials, with Polyethylene exhibiting the best sorting efficiency. The success rate for each particle type is shown in Figure 6.4(c).

6.3.6 Evaluation of Microwave Microfluidic Sensing Platform Performance Across Different Liquid Carriers

In this work, we primarily focused on prefiltration of particles followed by their addition to deionized (DI) water. However, to assess the sensor’s efficiency under varying conditions, we also tested the performance of the sensor with different liquid carriers. The selected carriers included DI water, tap water, and solutions of 0.1 M sodium chloride (NaCl), 0.05 M urea, 0.01 M potassium nitrate (KNO₃), and 0.01 M sodium nitrate (NaNO₃) dissolved in DI water. A 0.1% Tween 20 solution was used as a surfactant in all cases, as described in the sample preparation. This Tween 20 solution was employed to suspend the MP particles, specifically blue polyethylene microspheres in the 250–300 μm size range. To ensure uniform dispersion and minimize aggregation, the MP particles were added to the Tween 20 solution in each liquid carrier and centrifuged for 5 minutes. This step allowed for consistent sensor measurements across all tests.

The relative permittivity (ϵ_r) of the liquid carriers was measured over a broad frequency range, as shown in Figure 6.7. The results indicated that salts and urea significantly affect the liquid permittivity, even at low concentrations. These findings underscore the importance of considering the impact of liquid carriers in sensor design and performance analysis. In real environmental conditions, the presence of mixed solutes and impurities will likely lead to even greater variability in permittivity. Therefore, resonance frequency shifts should be calculated relative to the baseline resonance frequency of the same liquid carrier without particles, ensuring accurate detection and analysis in diverse liquid environments.

From the measured data presented in Figure 6.4(d), the frequency shifts of the two resonators were analyzed for all six liquid carriers. For each carrier, data was collected for 20 particles, and both the average frequency shift and standard deviation were plotted to ensure statistical reliability. The small resonator showed an average resonance frequency shift of approximately 374 kHz, while the large resonator exhibited a shift of 691 kHz. The slight variations observed across the liquid carriers are attributed to differences in their dielectric properties, which affect the baseline resonance frequencies of the sensor. Overall, these results validate the ability of the microwave microfluidic sensing platform to detect particles with high sensitivity and consistency, even across diverse liquid environments.

6.4 Environmental Implications

The detection and quantification of MPs in the environment are crucial for understanding their distribution and potential impacts on ecosystems. This study has demonstrated

the potential of a microwave microfluidic sensing platform for the real-time detection and classification of MP particles across various sizes and materials. This platform offers a compact, scalable, and efficient method for identifying MPs, with the ability to differentiate between different polymer types. This capability is vital, given the growing concerns about the widespread presence of MPs in aquatic systems and their impact on both environmental health and human well-being. One of the key strengths of this platform is its ability to detect MPs with high sensitivity and accuracy, even in complex liquid environments. The performance across multiple particle types, including polyethylene, polystyrene, soda lime glass, and brine shrimp egg, highlights the robustness of the platform in classifying particles based on their resonance profiles. Furthermore, the system's ability to operate in real-time without the need for extensive pre-treatment or complex setups makes it highly suitable for field deployment, which is a critical need for monitoring MPs in dynamic environmental conditions.

Despite these promising advancements, there are several limitations that must be addressed to fully optimize this system for widespread environmental monitoring. First, the prefiltration method using the sieve stack may not be the most efficient solution. Over time, the sieve stack can become clogged, which could lead to inaccuracies in particle size sorting. Exploring alternative microfluidic techniques for initial particle sorting could provide a more efficient and reliable prefiltering step, especially in real-world applications where sample volumes and particle types vary widely. Second, the current design of the sensing platform does not allow for full control over the particles' movement through the system. The accuracy of the detection could be improved if particles could be forced to remain in the sensing region for a longer period, ensuring that the sensor can capture a more accurate resonance signal. Modifying the system to provide greater control over particle flow, perhaps through adjustments to the microfluidic channel or incorporating flow regulation techniques, could enhance the overall sensitivity of the platform. Lastly, the use of a syringe pump to drive the flow of sample solutions through the microfluidic channel introduces another potential issue. Some particles tend to deposit at the bottom of the syringe, especially when using viscous or heterogeneous solutions, making it challenging to transport all particles into the channel for analysis. Exploring alternative sample introduction methods, such as funnel-based sample entry or a vertical pump system, could alleviate this problem by ensuring a more consistent and reliable flow of particles into the detection region.

Another important consideration for real-world application is the variability in the conductivity and composition of environmental water samples, which can significantly affect the resonance amplitude of the sensor. Since the detection mechanism relies in part on amplitude changes to classify particle types, inconsistencies in the electrical properties of

the carrier fluid could compromise the reliability of the results. For instance, if the ionic strength or conductivity of the sample changes over time, due to salinity, organic content, or other dissolved substances, it becomes difficult to isolate the effect of the particles themselves. To address this, future iterations of the platform should incorporate conductivity monitoring or control mechanisms, or employ a standardized carrier fluid for consistent performance.

While the proposed sensing platform shows great promise for MP detection in the environment, addressing these technical challenges will further improve its accuracy, efficiency, and applicability. Future research should focus on optimizing the prefiltration techniques, improving particle flow control, and refining sample introduction methods to create a more robust system for widespread deployment in environmental monitoring and research.

6.5 Uncertainty Analysis

To better understand the limitations of the proposed sensing system and to assess its measurement accuracy and repeatability, we include a detailed analysis of potential sources of error. These sources are broadly categorized into systematic and operational uncertainties, both of which affect the platform’s performance in detecting and classifying microplastic particles.

Systematic errors are inherent to the measurement system and sensor design. The Vector Network Analyzer (VNA), despite its high sensitivity, has a frequency resolution limit that restricts the ability to distinguish extremely small shifts, particularly at the lower end of the particle size range. Furthermore, even slight fluctuations in ambient temperature or electromagnetic interference can introduce variability in the sensor’s resonance profile, potentially affecting detection sensitivity. The microfluidic channel, fabricated from PDMS and bonded to glass, is susceptible to geometric variability from the molding and bonding processes, which may alter the local electromagnetic field distribution. Additionally, the soldering quality of SMA connectors, alignment between the microwave reader and microfluidic chip, and cable shielding all influence signal integrity and can introduce small, but consistent, deviations in the measured transmission response.

Operational errors arise from sample preparation, handling, and fluidic control. In this study, a syringe pump is used to drive the particle-laden sample through the microfluidic channel. However, settling of particles within the syringe, especially in heterogeneous or viscous suspensions, can lead to uneven distribution and partial delivery of the sample, resulting in inconsistent particle detection. Manual sample preparation steps, such

as pipetting, dilution, centrifugation, and vortexing, may also introduce concentration inaccuracies or incomplete dispersion. Even with the use of a surfactant (Tween 20), full homogenization is not guaranteed, and particle aggregation or charge-based clustering may occur. Human error in handling the chip, aligning the sensor, or initiating data collection further contributes to experimental variability. Finally, the prefiltration process using a sieve stack, while useful for isolating specific size ranges, may not achieve perfect separation and may become clogged over time, especially when dealing with environmental samples containing mixed debris.

By identifying these sources of error, this section provides a foundation for improving system performance. Future enhancements may include integrating real-time temperature and flow-rate compensation, automating sample handling, and exploring alternative fluid propulsion methods to improve particle delivery and retention within the sensing region. These steps will contribute to a more robust, field-deployable platform for reliable, real-time microplastic detection.

6.6 Summary

This paper presents a novel microwave microfluidic platform for the real-time detection and quantification of MPs in environmental samples. The platform is designed to simplify the detection process and provide an efficient, cost-effective solution for monitoring MPs in the size range of 20 μm to 300 μm . The system integrates prefiltration of particles using a sieve stack method, disposable microfluidic chips, and a planar microwave reader to perform the detection. The raw data from the sensor is processed using a k-nearest neighbors (KNN) algorithm to classify the particles based on their resonance profiles, while particle size is determined by the prefiltration step. The platform is capable of single-particle detection, enabling accurate quantification of particle concentration. The study demonstrates the platform’s ability to differentiate between various materials, including polyethylene, polystyrene, soda lime glass, and brine shrimp eggs, based on changes in resonance frequency and amplitude. The proposed system offers a promising solution for environmental monitoring, providing high sensitivity, real-time detection, and the ability to classify particles in complex liquid environments without the need for extensive sample preparation. However, the paper also highlights areas for improvement, such as optimizing the prefiltration method and enhancing particle flow control to improve detection accuracy and efficiency in field applications.

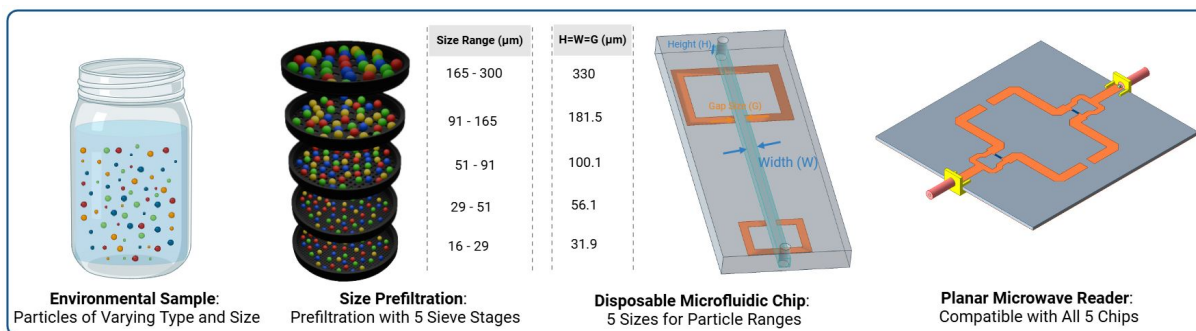


Figure 6.5: Experimental setup for particle size analysis: Environmental sample with particles of varying sizes is prefiltered using a five-stage sieve system, followed by microfluidic chip-based particle detection. The planar microwave reader is compatible with all five chips for particle size classification.

6.7 Supplementary Information

In addition to the main findings presented in this chapter, supplementary information is included to further clarify and elaborate on the methodologies, data analysis, and results discussed.

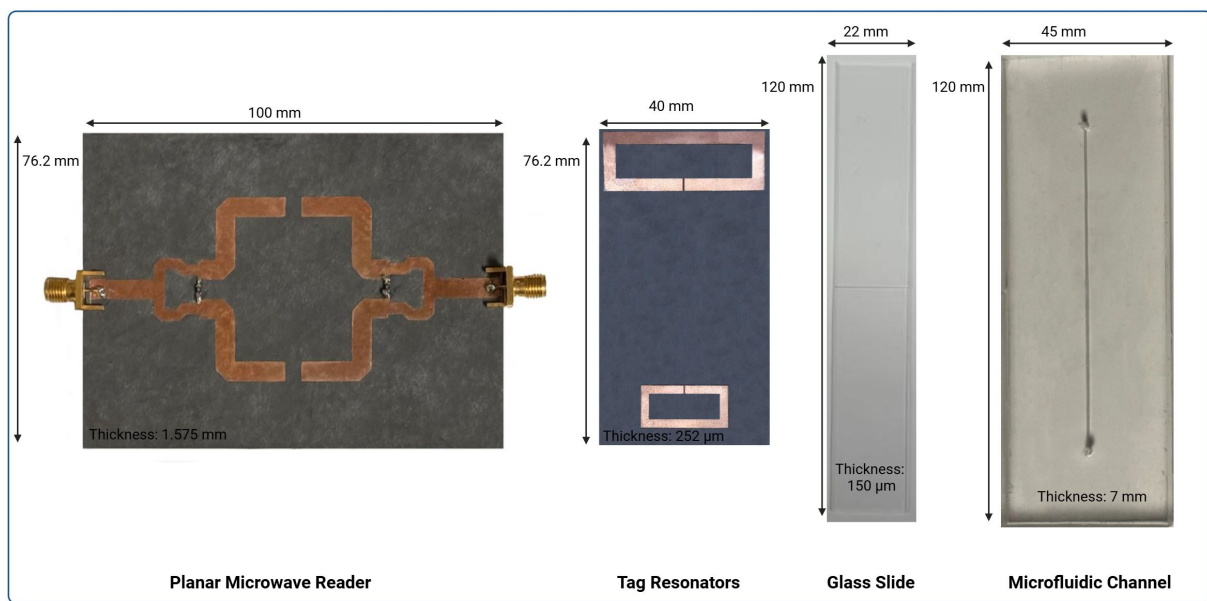


Figure 6.6: Schematic representation of the microwave microfluidic sensin platform, with detailed dimensions of the planar microwave reader, tag resonators, glass slide, and microfluidic channels used for particle characterization.

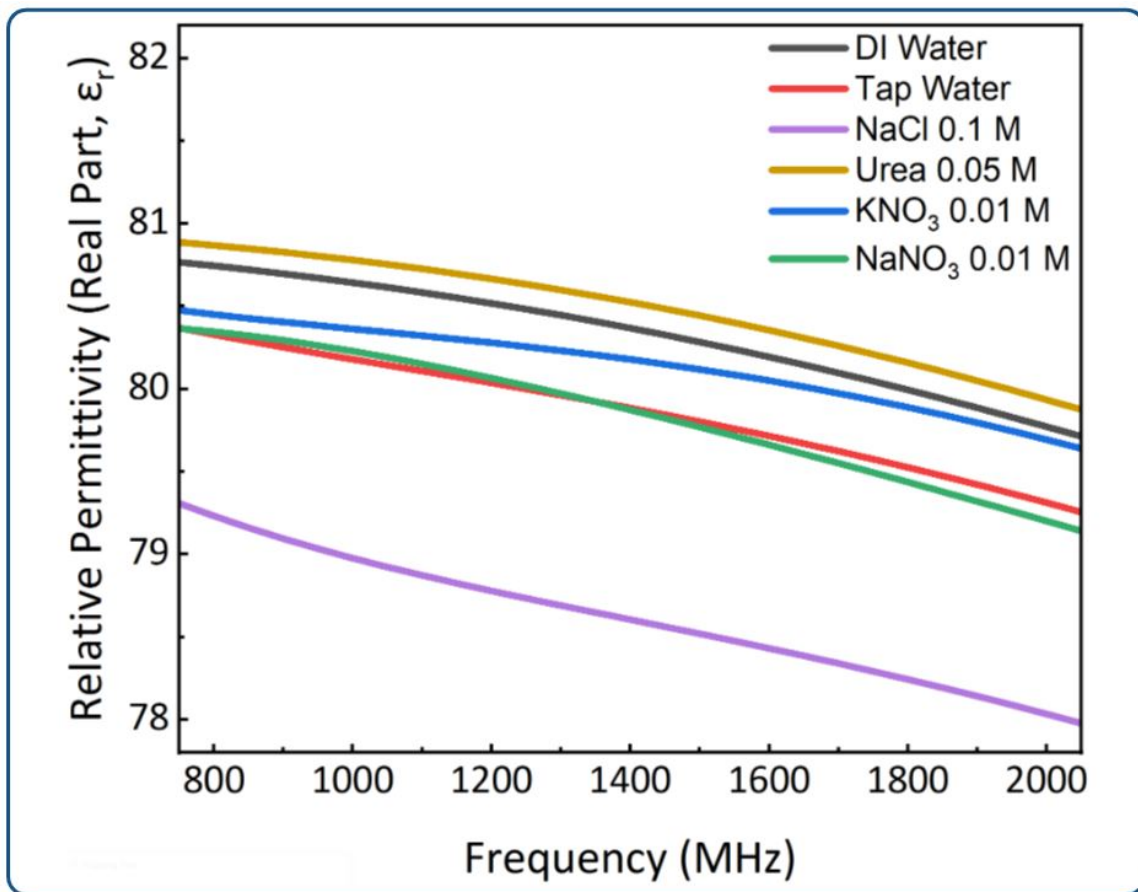


Figure 6.7: Measured relative permittivity (real part, ϵ_r) of selected liquid carriers using the SPEAG DAK-TL2 probe, across a frequency range of 800 MHz to 2000 MHz.

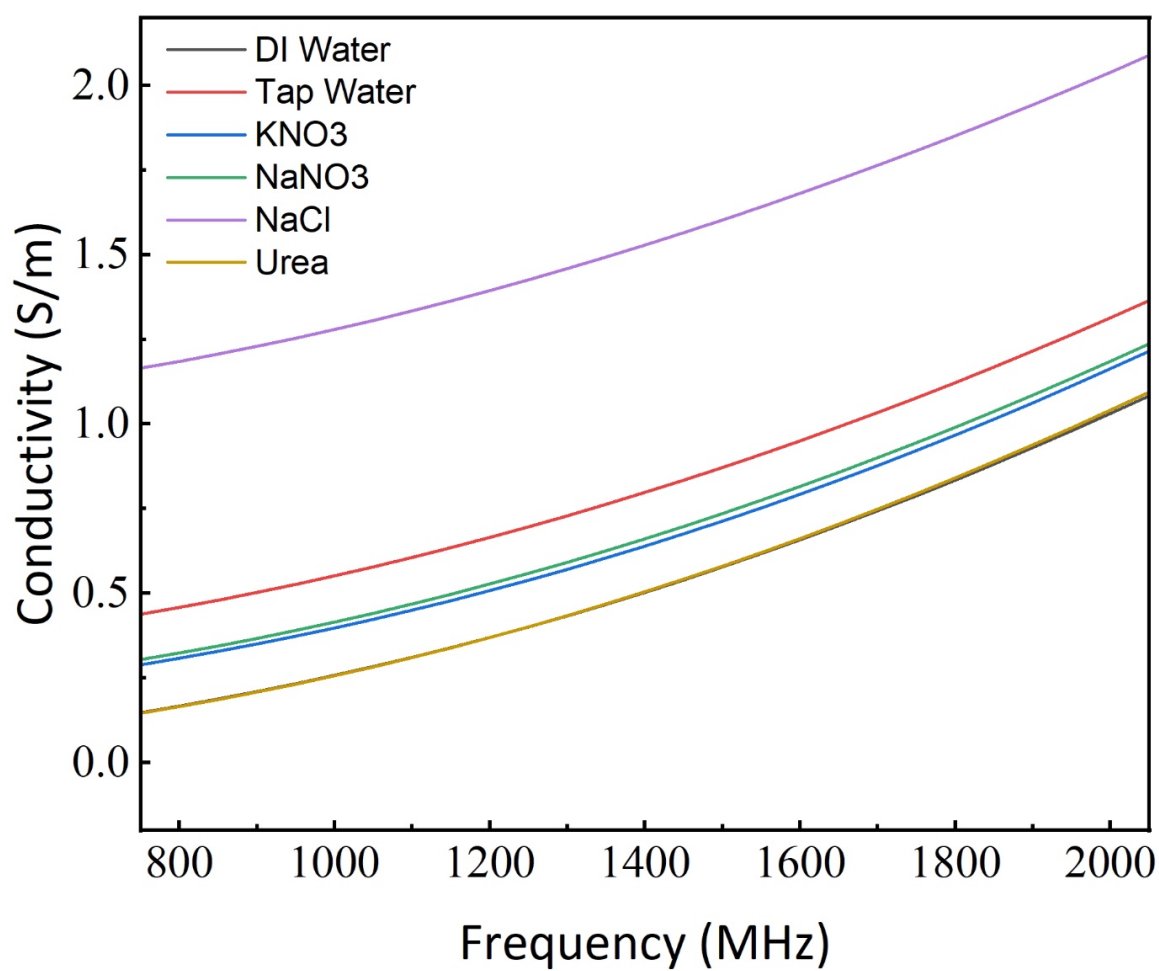


Figure 6.8: Measured conductivity of selected liquid carriers using the SPEAG DAK-TL2 probe, across a frequency range of 800 MHz to 2000 MHz.

Chapter 7

Expanding Applications of Microwave-Microfluidic Sensors: From Environmental to Biological Samples

7.1 Overview

While the primary focus of this thesis is the development of microwave-microfluidic sensors for detecting plastic particles in environmental samples, the versatility of these sensors offers opportunities for application in other fields. One such area is biomedical diagnostics, where particle and biomolecule detection play a critical role in patient monitoring and treatment. This chapter explores the application of the developed microwave-microfluidic sensor platform in the detection of biomolecules in biological samples, specifically targeting the monitoring of amylase levels in postoperative peritoneal drainage fluid as an indicator of anastomotic leakage. This study demonstrates the adaptability of the sensor system and highlights its potential for cross-disciplinary applications, paving the way for broader impacts in both environmental and biomedical sciences.

This chapter presents a collaborative project between the Waterloo Microfluidics Lab and FluidAI. Maziar ShafieiDarabi, Alice Mao, and Weijia Cui are co-first authors of the resulting publication, which has been submitted with the information provided below. Dr. Cui, a former postdoctoral researcher in the Waterloo Microfluidics Lab, initiated the project before departing the lab. Following his departure, Maziar and Alice took

the lead—Alice focusing on the chemical coating aspects, while Maziar conducted the electromagnetic simulations, performed the experiments, processed the data, wrote the manuscript, and created the original figures.

Maziar ShafieiDarabi, Alice Mao, Weijia Cui, Matthew Courtney, Robert Miranda, Lauren LeSergent, Ricky Tjandra, Luke Pyung-Se Lee, and Carolyn L. Ren, "Development of a Microwave-Based Biosensor for Monitoring Anastomotic Leakage Through the Detection of Amylase in Drainage Fluid," *Elsevier Journal of Biosensors & Bioelectronics*, 2025.

7.2 Introduction

Colorectal cancer (CRC) is the third most commonly diagnosed cancer worldwide and remained the second leading cause of cancer-related deaths in 2022 [285, 286]. Despite significant advancements in surgical techniques, complications such as anastomotic leakage (AL) continue to pose considerable challenges, leading to higher mortality rates, longer hospital stays, and negative long-term outcomes [287]. Although there is ongoing debate regarding the definition of anastomotic leakage, the International Study Group of Rectal Cancer defines AL as a defect in the intestinal wall at the anastomotic site, resulting in communication between the intraluminal and extraluminal compartments [288].

One of the challenges related to AL is delayed diagnosis [289]. As illustrated in Figure 7.1, the timeline of anastomotic leakage emphasizes the stages from surgery to diagnosis and treatment, highlighting the significance of early detection for better patient outcomes. While some leaks are apparent and occur early, others appear later in the postoperative period, often showing more subtle characteristics [289]. The average time to detect an AL is 8.8 days post-operation [289]. In some cases, these leaks may not be recognized until several months after they occur [290]. Early detection is crucial, as prompt intervention has been demonstrated to lower morbidity and mortality, resulting in improved patient outcomes. Consequently, considerable efforts have been made in recent years to find reliable biomarkers for the early diagnosis of AL [291]. Currently, no clinically reliable test is available for detecting AL, as traditional clinical indicators such as temperature, white blood cell count, blood pressure, and respiratory rate are not consistently indicative of leak occurrence [292]. Systemic inflammatory markers, including C-reactive protein (CRP) and leukocyte count, have also been found unreliable as positive predictors for AL [293].

Recent studies have highlighted specific biomarkers in peritoneal fluid, such as lactate, pH, and elevated cytokine levels, as more reliable indicators of AL [294]. Peritoneal fluid

biomarkers have garnered significant attention since samples are obtained from the site of the leak. This localization allows for quicker identification than systemic biomarkers and provides access to markers that may not be significant in systemic circulation [293]. One promising biomarker is amylase, which is sensitive, responsive, and easily measurable from drain fluid samples [291, 295]. A study by Clark et al. found that the median drain fluid amylase level in patients with an anastomotic leak following ileal pouch surgery without a diverting ileostomy was 1373.5 U/L, which is significantly higher than the reference range of 130 U/L [291]. In some cases, amylase levels reached as high as 21,897 U/L [295]. Amylase tests are typically colorimetric and require specialized personnel and an equipped clinical laboratory for accurate results [296]. Therefore, developing a rapid and dependable measurement system that can identify significant increases in amylase concentration is crucial for the early detection of AL.

During elective colorectal surgery, the surgeon has the option to place a drain next to the anastomosis [297]. The reason for using a drain is to help evacuate peri-anastomotic fluid collections, thus preventing the buildup of potentially harmful fluid [297]. This fluid contains various biomarkers, with amylase being a significant target of investigation [291, 295]. The effectiveness of prophylactic drains following anastomosis surgery remains a topic of debate, as some studies have shown no significant benefits associated with their use [298]. However, their utility may be reconsidered when integrated with advanced monitoring systems. Such systems, capable of providing real-time analysis of biomarkers like amylase, might improve the detection of complications such as AL, potentially justifying the routine use of drains in postoperative care.

Many sensors have been developed to detect amylase; however, these devices primarily focus on measuring amylase levels in saliva or serum [299, 300, 301, 302, 303], many being disposable point-of-care devices [304, 305, 306, 307, 308]. None of these devices or measurement methods have been demonstrated for monitoring AL. So far, two sensors have been created to monitor amylase in drain effluent [309, 310]. The first sensor is an optical amylase biosensor that uses surface plasmon resonance in a plastic optical fiber, which is then metalized with a 60 nm layer of gold and interrogated with white light [309]. This sensor was made specifically with anti-amylase antibodies to detect amylase in drainage fluid following pancreatectomy [309]. During preliminary tests, the sensor was able to detect amylase concentrations within a linear range of 0.8 to 25.8 U/L, with a limit of detection (LOD) of 0.5 U/L [309]. While relevant for detecting pancreatic fistula in pancreatic drainage fluid [309], this operating range is far below the range required for the high spikes observed in drainage fluid following colectomy. This limitation may arise from the optical sensor's inability to maintain linearity at high concentrations. The second sensor employs portable droplet microfluidics to encapsulate drain liquid in nano-liter-sized

droplets of starch reagent [310]. These droplets undergo a reaction with alpha-amylase, and the resulting fluorescence is measured upon release [310]. An issue associated with droplet microfluidic devices is their ability to deliver reliable measurements under varying environmental conditions and amid significant patient movement, which may occur during bedside use [311]. Thus, there remains considerable potential for future work to improve amylase sensors to detect AL through the analysis of drainage fluid.

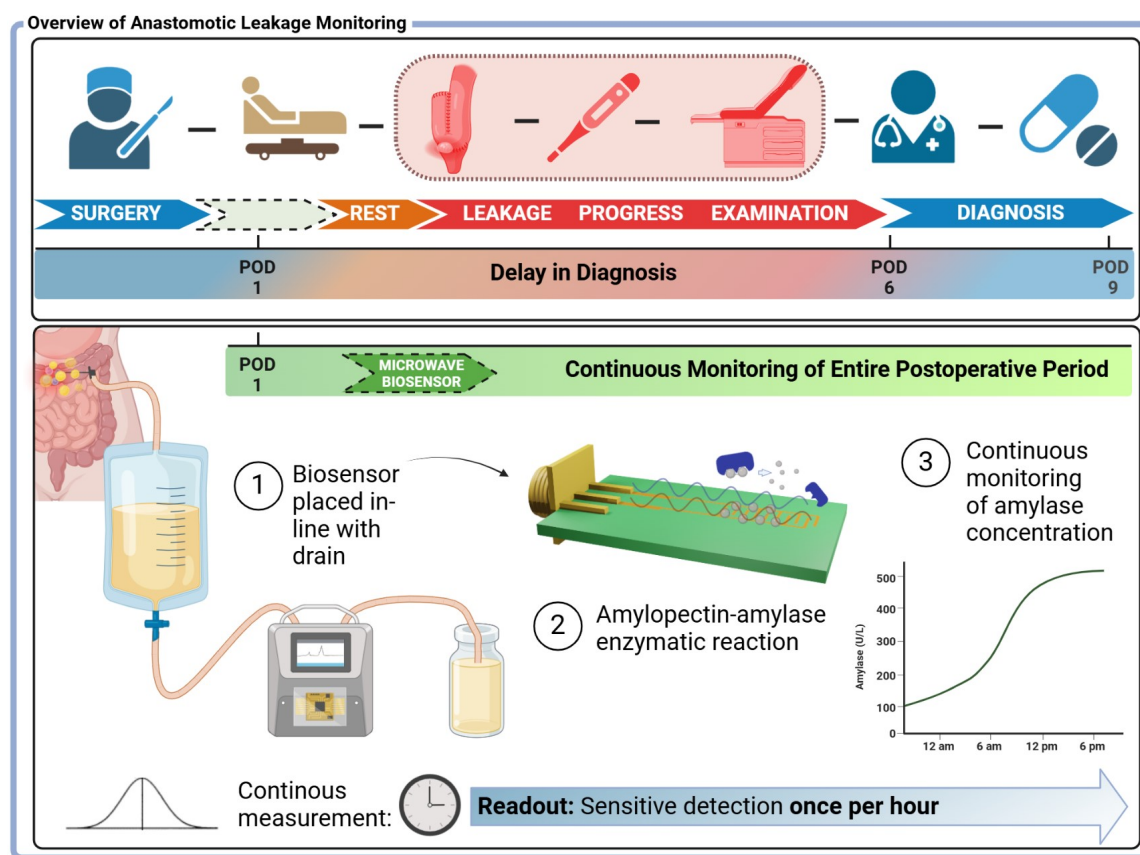


Figure 7.1: Overview of Anastomotic Leakage Monitoring. The top section illustrates the general diagnostic process for detecting anastomotic leakage (AL), showing the typical delay in diagnosis between postoperative day (POD) 6 and POD 9. The bottom section depicts our microwave biosensor, placed in-line with the drainage system from POD 1. The biosensor operates via an amylopectin-amylase enzymatic reaction, enabling continuous monitoring of amylase concentration in the drainage fluid. The readout offers sensitive detection once per hour, facilitating early and ongoing detection of AL throughout the entire postoperative period.

This paper presents the development of a microwave-based biosensor for monitoring AL by detecting amylase in drainage fluid. To accomplish this, a printed circuit board (PCB) sensor was designed and simulated using commercially available full-wave simulation software to optimize its geometry and performance. The sensor was then fabricated and chemically functionalized with an amylopectin coating to enhance its specificity for amylase detection. This platform aims to enable quantitative measurement of amylase concentrations in complex media such as simulated peritoneal fluid (SPF), particularly targeting the detection of high surges associated with leakage events. Additionally, the sensor is designed to stay robust under varying environmental conditions, providing the potential for bedside integration to allow real-time, continuous monitoring of amylase levels in drainage fluid.

7.3 Experimental Section

7.3.1 Sensing Principle

Microwave sensors differentiate materials based on their electrical properties, such as permittivity. In this study, the sensing mechanism relies on an enzymatic reaction between amylopectin coated on the sensor surface and amylase present in the sample fluid. This reaction partially removes the amylopectin coating, leading to changes in the local permittivity around the sensor surface. These changes manifest as shifts in the sensor's resonance frequency. The resonance frequency, defined as the frequency at which the reflection coefficient (S_{11}) reaches its minimum, is measured using a Vector Network Analyzer (VNA), as illustrated in Figure 7.2. This frequency shift provides a quantitative measure of amylase concentration, enabling the detection of elevated levels associated with anastomotic leakage.

Principle of Working

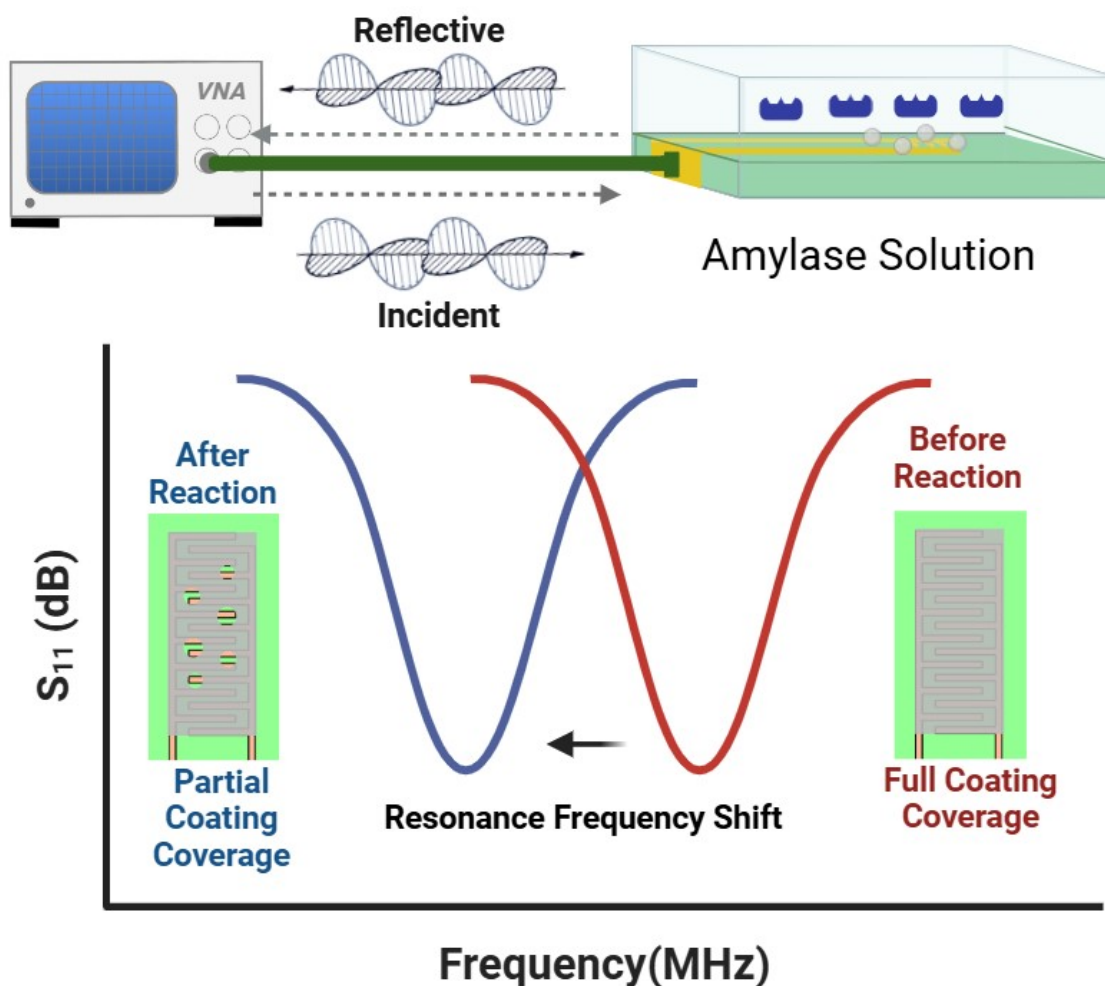


Figure 7.2: Principle of Working. Illustration of the experimental setup, with a focus on the reflection coefficient (S_{11}) as the primary measured parameter. An inset displays the resonance profile changes resulting from the amylopectin-amylose interaction.

7.3.2 Sensor Fabrication

The microwave-based biosensor was initially designed and simulated using High-Frequency Structure Simulator (HFSS) [312] software to optimize its geometry and performance parameters. The simulations offered insights into electric field distribution, resonance frequency, and the expected sensitivity to variations in amylase concentrations. Based on the simulation results, the sensor was fabricated using a standard PCB manufacturing process. The chosen substrate material was FR4, a commonly used dielectric material with copper metal traces [313]. Two different passivation layers, tin, and gold, were examined to protect the copper traces from corrosion, oxidation, and degradation.

7.3.3 Sensor Coating Procedure

The coating of the sensor surface involved several preparation and application steps to ensure the formation of a functionalized layer designed explicitly for amylase detection. The uncoated sensor can detect amylase in deionized (DI) water, serving as a straightforward host medium; however, this detection is characterized by minimal frequency shifts. In more complex media, such as drain fluid or SPF, these small frequency shifts may also result from other substances in the sample. This lack of selectivity makes it challenging to determine whether the observed frequency shift is caused by amylase or other components. The coating addresses this issue by providing a surface specifically sensitive to amylase, enhancing the sensor's selectivity. It also amplifies the frequency shift in response to amylase, enabling more reliable detection than the uncoated sensor. An amylopectin coating was applied to the sensor surface to achieve this functionality. Its preparation process was inspired by the work of Peregrino et al. [314], customized to suit our specific application, and is described below. The coating process involved several steps, starting with surface functionalization using 3-mercapto-1-propanesulfonate (MPS). MPS was selected for its ability to create a thiol-functionalized surface, which provides strong adhesion and stability for the subsequent amylopectin coating. This functionalization ensures effective immobilization of the amylopectin layer, thus enhancing the sensor's selectivity and sensitivity for amylase detection. The MPS solution was prepared by dissolving sodium 3-mercapto-1-propanesulfonate (Sigma-Aldrich) in DI water to achieve a final concentration of 1 mM. The sensors were cleaned with isopropyl alcohol, followed by plasma cleaning to ensure surface cleanliness and activation. Initially, oxygen plasma cleaning was used; however, it was noted that the success rate of the coating was inconsistent, often resulting in sensors that were coated non-uniformly. To address this issue, hydrogen plasma cleaning was implemented to address this issue, which significantly enhanced coating uniformity and re-

producibility. Oxygen plasma cleaning primarily targets biological contaminants by using ultraviolet energy to break down organic bonds and reacting with contaminants to produce low molecular weight hydrocarbons, which are subsequently removed via vacuum [315]. In contrast, hydrogen plasma cleaning eliminates oxide layers from metal surfaces through vapor formation [316]. The removal of this oxide layer improved the thiol-functionalization of MPS, resulting in a higher sensor yield after coating. Immediately following cleaning, the sensors were soaked in a 1mM MPS solution for two nights to functionalize the surface. A 10 mg/mL amylopectin solution was prepared by dissolving 250 mg of amylopectin sourced from maize (Sigma-Aldrich) in 25 mL of deionized water. To improve the stability of the coating, 1% Span 85 surfactant (Sigma-Aldrich) was added by volume, amounting to 0.25 mL. After being removed from the MPS solution, the sensors were dried in a heating chamber at 35 °C for 15 minutes and then positioned in a custom-designed coating device. The coating device, designed and 3D-printed in-house, ensured that the sensor surface remained flat and that the amylopectin solution was applied only to the sensing region of the sensor. A 100 μ L drop of the prepared amylopectin solution was carefully pipetted onto the sensor surface and left undisturbed for 5 minutes to guarantee uniform distribution of the coating. With the sensors secured inside, the coating device was then transferred to a heating chamber set at 35 °C and incubated for two days to allow the coating to cure and stabilize.

7.3.4 Measurement Setup

The coated sensors were then integrated into a custom-designed 3D-printed measurement jig equipped with an SMA connector. The jig provided a stable connection between the sensor and external measurement equipment, which was a Vector Network Analyzer (VNA, Copper Mountain M5090).

7.3.5 Sample Preparation and Testing Protocol

The study began with simple DI water as the host medium to evaluate the sensor’s ability to detect amylase concentrations and its relevance to anastomotic leakage. Specific amylase concentrations were added to DI water to determine the sensor’s limit of detection (LOD) and the frequency shift corresponding to each amylase concentration. This step established the baseline performance of the sensor. After initial validation of sensor abilities, DI water was replaced with SPF, a medium closer to clinical drainage fluid. To isolate the effect of amylase on the sensor, the SPF was custom-prepared by Biochemazone (BZ289)

without amylase. Specific concentrations of amylase (Amylase from Porcine pancreas, Sigma-Aldrich) were then added to the SPF, and the sensor's response was measured like the DI water experiments. This approach allowed for a comparative study of the sensor's performance in idealized and clinically relevant conditions. Throughout the testing process, the measurement jig was securely fixed to the lab table to prevent movement, and a constant volume of sample (8 mL) was poured over the sensor surface for each measurement. The sensor's response was recorded over time using the VNA to capture the reflection coefficient (S11). Measurements were repeated at least three times for each concentration to ensure repeatability. The error bars in the plots were derived from the average and standard deviation of these repeated measurements.

7.3.6 Comparison with Standard Amylase Assay Kit

In addition to testing the microwave-based biosensor, the EnzChek™ Ultra Amylase Assay Kit (Thermo Fisher Scientific) was utilized to validate the results obtained from the sensor. This standard fluorescence-based assay served as a benchmark for comparing the sensor's performance in measuring amylase concentrations under various experimental conditions. The assay was performed according to the manufacturer's protocol, and fluorescence was measured using a microplate reader set for excitation at 485 ± 12 nm and emission detection at 520 ± 12 nm. The fluorescence of the blank sample (without enzyme) was subtracted from each measured value [317]. The results from the assay kit were then compared to the frequency shifts observed with the microwave biosensor to ensure both accuracy and reliability.

7.4 Results and Discussion

The microwave-based biosensor was developed as a proof-of-concept device for detecting different amylase concentrations. It serves as a potential biomarker for anastomotic leakage in postoperative peritoneal drainage fluid. To evaluate the performance of the sensor, a series of experiments and simulations were conducted to assess its design, specificity, response to environmental factors, and its ability to perform quasi-continuous monitoring. The results indicate that the sensor is not only sensitive to amylase concentrations but also capable of distinguishing between different concentrations with high specificity.

7.4.1 Sensor Design

As part of the sensor design, full-wave numerical simulations illustrated the sensor's electric field distribution, highlighting significant field intensity over the metal traces of the sensor, as shown in Figure 7.3(A). This indicates that the coated sensor part can effectively function as the sensing region. As shown in the inset of Figure 7.3(A), the electric field intensity, viewed from the X-Z plane at the midsection of the sensor, is significantly stronger near the surface than at greater heights. This observation underscores the importance of the coating, which reacts explicitly with amylase. By confining the reaction to the sensitive surface region, the coating enhances the sensor's response, resulting in more pronounced effects on the resonance profile and more significant frequency shifts. This makes the sensor more effective in distinguishing amylase from other substances in complex mediums. Additionally, Figure 7.3(B) presents the changes in the reflection coefficient before and after the amylopectin-amylase reaction. The reflection coefficient shift (Δf) indicates how the resonance profile is altered, demonstrating that the sensor's response is highly influenced by the interaction between amylase and the amylopectin coating. The frequency shift in the reflection coefficient is evident, with a noticeable change between the sensor before and after the reaction, further validating the coating's role in enhancing the sensor's specificity to amylase. Additionally, Figure 7.3(C) highlights the effect of coating thickness on sensor performance. The simulations demonstrated that the sensor is more sensitive with a coating thickness of 20 μm compared to 5 μm . This conclusion was drawn by comparing their performance at different coating coverage levels and analyzing the corresponding frequency shifts. The thicker coating (20 μm) resulted in more pronounced frequency shifts across varying levels of coverage, underscoring the importance of optimizing the coating thickness to achieve enhanced detection performance.

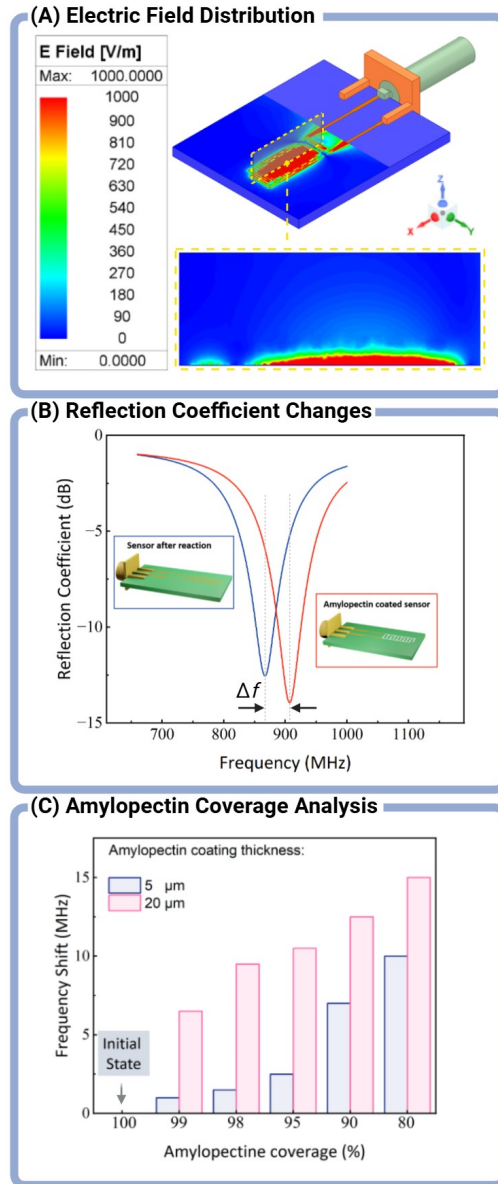


Figure 7.3: Full-wave simulation results: (A) Electric field distribution on the surface of the microwave-based biosensor, with an inset showing the electric field intensity in the X-Z plane at the sensor's midsection. (B) Numerical simulation results showing changes in the reflection coefficient before and after the Amylase-Amylopectin reaction. (C) Numerical simulation depicting the relationship between sensor surface amylopectin coverage and frequency shift, with 100% coverage defined as the initial state (sensor before reaction), for two different coating thicknesses of 5 μm and 20 μm .

7.4.2 Quasi-Continuous Monitoring of Amylase

As demonstrated in Figure 2, the microwave biosensor's ability to perform quasi-continuous monitoring of amylase concentrations over time is shown. Frequency shift refers to the change in the resonance frequency of the sensor, which occurs due to the interaction between the sensor surface and the amylase present in the sample. The reference data for frequency shift is the resonance frequency measured at the initial time, with the frequency shift calculated as the difference between the initial resonance frequency and the resonance frequency at subsequent time intervals. In Figure 7.4(A), the resonance profile of the biosensor is shown for an amylase concentration of 5000 U/L, with measurements taken at multiple time intervals (15, 30, 45, and 60 minutes). As the time increases, a clear frequency shift is observed, which correlates with the interaction between amylase and the amylopectin coating on the sensor surface. Figure 7.4(B) shows the frequency shift over time for five different amylase concentrations, ranging from 1000 to 5000 U/L, measured at regular intervals from 0 to 60 minutes. The results indicate that the biosensor exhibits an increasing trend in frequency shift with increasing amylase concentration. These findings highlight the potential of the microwave biosensor for quasi-continuous monitoring of amylase, making it suitable for both early detection and long-term monitoring in clinical applications, such as detecting anastomotic leakage. The ability to track amylase levels over time, with measurements taken at intervals, provides valuable information for continuous patient assessment.

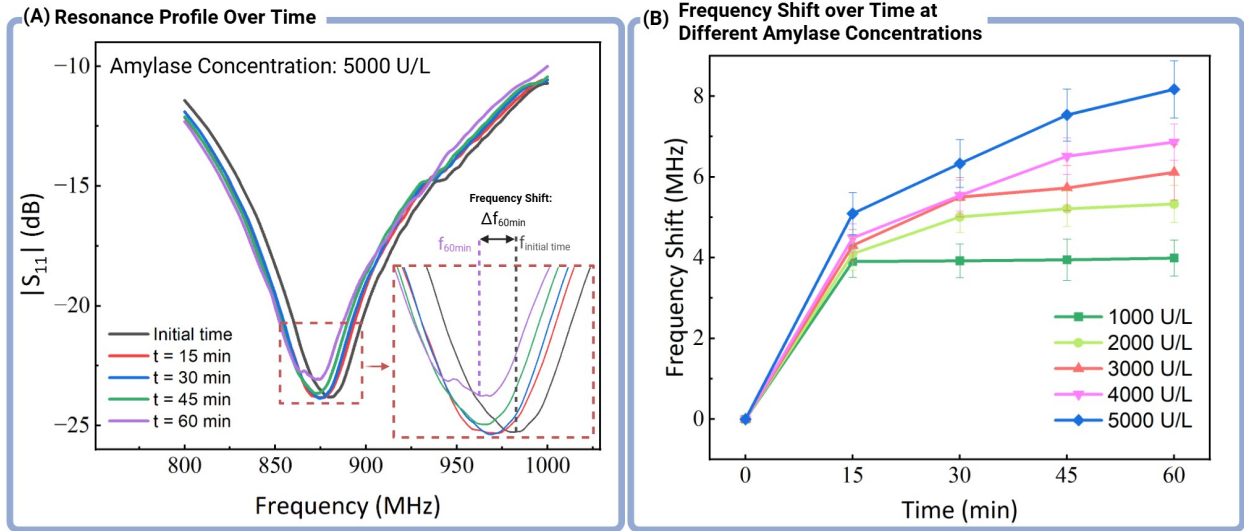


Figure 7.4: Quasi-Continuous Monitoring of Amylase. (A) Resonance profile of the microwave biosensor at an amylase concentration of 5000 U/L, measured at multiple time intervals (0, 15, 30, 45, and 60 minutes). The inset shows a zoomed-in view, highlighting the frequency shift more clearly. (B) Frequency shift over time for five different amylase concentrations (1000 to 5000 U/L), measured at regular intervals from 0 to 60 minutes.

7.4.3 Specificity and Environmental Effects

The performance of the sensor was evaluated in terms of specificity, optimal coating volume, temperature effects, and material selection for the coating. Figure 7.5(A) illustrates how the amylopectin-amylase enzymatic reaction enhances the sensor's specificity to amylase. The interaction between amylase and the amylopectin coating leads to enzymatic activity, which directly influences the resonance frequency of the sensor, allowing for selective detection of amylase. The coating process was executed precisely using the fabrication jig, ensuring a uniform application of amylopectin onto the sensor's surface, as illustrated in Figure 7.6(A). Microscopic and visual characterizations, presented in Figure 7.6(B), confirmed the uniformity and consistency of the amylopectin layer. They also demonstrated how the reaction between amylase and the amylopectin coating could partially remove the coating from the sensor surface, consequently resulting in a frequency shift. Quantitative analysis in Figure 7.6(C) showed that increasing amylase concentration led to the removal of a larger area of the amylopectin coating from the sensor. This observation indicates

that higher concentrations of amylase remove a larger area of the coating from the sensor surface after the reaction, resulting in reduced coating coverage and correspondingly higher frequency shifts in the resonance profile, thereby validating the efficiency of the coating under varied conditions. Additionally, Figure 7.5(B) presents the control tests, comparing the sensor's response in bare and coated conditions when immersed in DI water and an amylase solution with a concentration of 5000 U/L. For these tests, a coating aliquot of 100 μ L was used. The frequency shifts were defined as the difference between the resonance frequency at the initial time and after one hour at room temperature. Bare sensors exhibited minimal frequency shifts between water and amylase solutions after one hour (0.242 MHz), indicating low specificity. In contrast, coated sensors demonstrated significant frequency shifts in the presence of amylase compared to DI water (6.316 MHz), highlighting the critical role of the amylopectin coating in enhancing the sensor's specificity and enabling selective detection of amylase, even in complex media such as SPF or clinical drainage fluid. Figure 7.5(C) illustrates the effect of temperature on sensor performance, with tests carried out at various temperatures. The frequency shift, defined as the change in resonance frequency from the initial measurement to after one hour, was recorded during these tests. An aliquot of 100 μ L was used for coating, and frequency shifts were documented for four different concentrations of amylase (1000, 2000, 3000, and 4000 U/L) at four distinct temperatures (22°C, 27°C, 32°C, and 37°C). The results indicate that the sensor operates best at 37°C, a temperature consistent with physiological conditions. At this temperature, the frequency shift for a specific amylase concentration (e.g., 4000 U/L) is 39.349 MHz, which is significantly greater than the shifts observed at other tested temperatures (27.869 MHz at 32°C, 17.268 MHz at 27°C, and 6.864 MHz at 22°C). These findings emphasize the sensor's heightened sensitivity under physiologically relevant conditions.

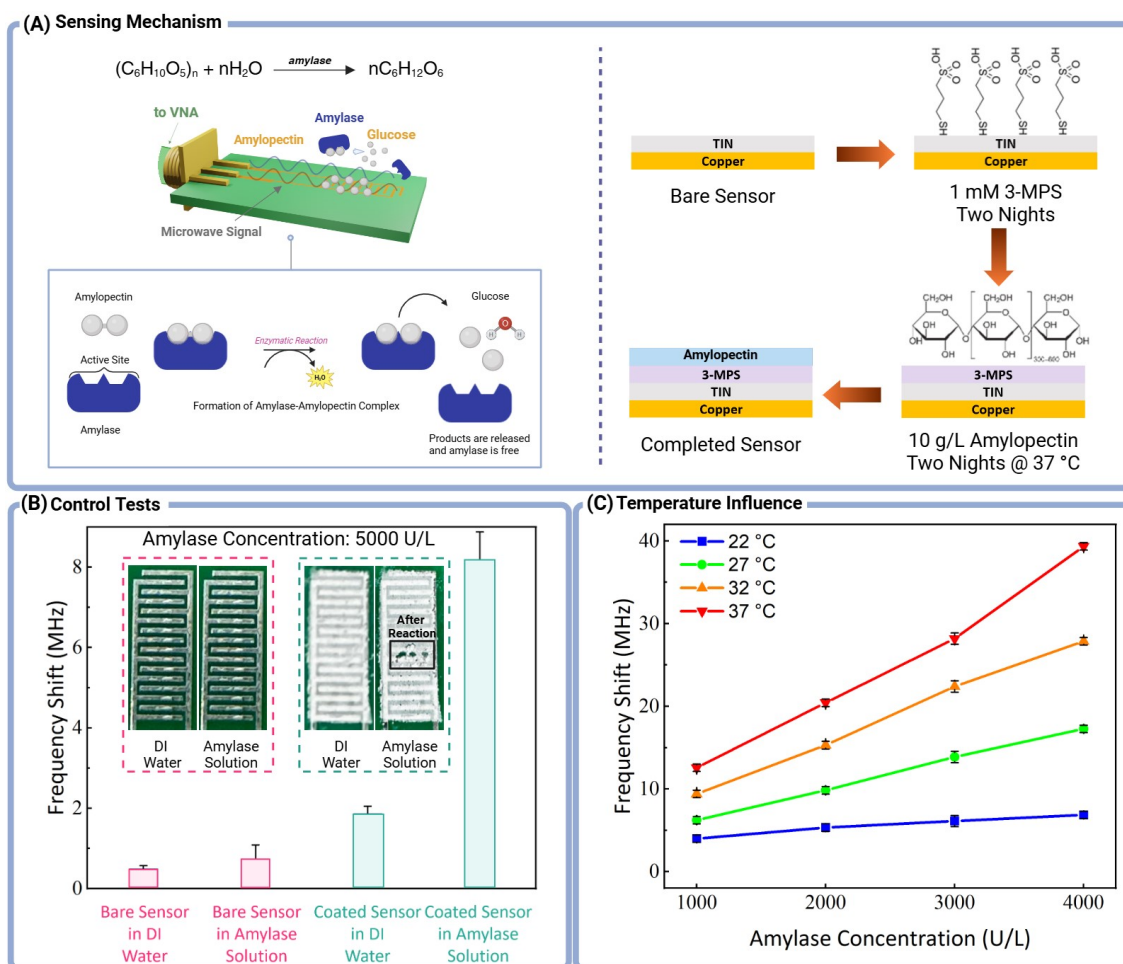


Figure 7.5: Sensor Sensing Mechanism, Control Tests, and Temperature Influence. (A) On the left, a schematic of the microwave-based biosensor featuring an amylopectin coating. The sensor functions through an enzymatic reaction between amylase and amylopectin, which leads to the release of glucose, as shown in the chemical reaction formula. The inset illustrates the reaction mechanism between amylopectin and amylase. On the right, the stepwise functionalization process is depicted, beginning with bare sensor, followed by treatment with 3-MPS, and the final amylopectin coating at 37°C to create the completed sensor. (B) Control tests with bare and coated sensors in DI water and amylase solution (5000 U/L). The inset shows the sensor's response before and after the reaction for both bare and coated sensors, demonstrating the enhanced sensitivity of the coated sensor. (C) Influence of temperature on the sensor's performance, showing the frequency shift for different amylase concentrations (1000–4000 U/L) measured at various temperatures ranging from 22°C to 37°C.

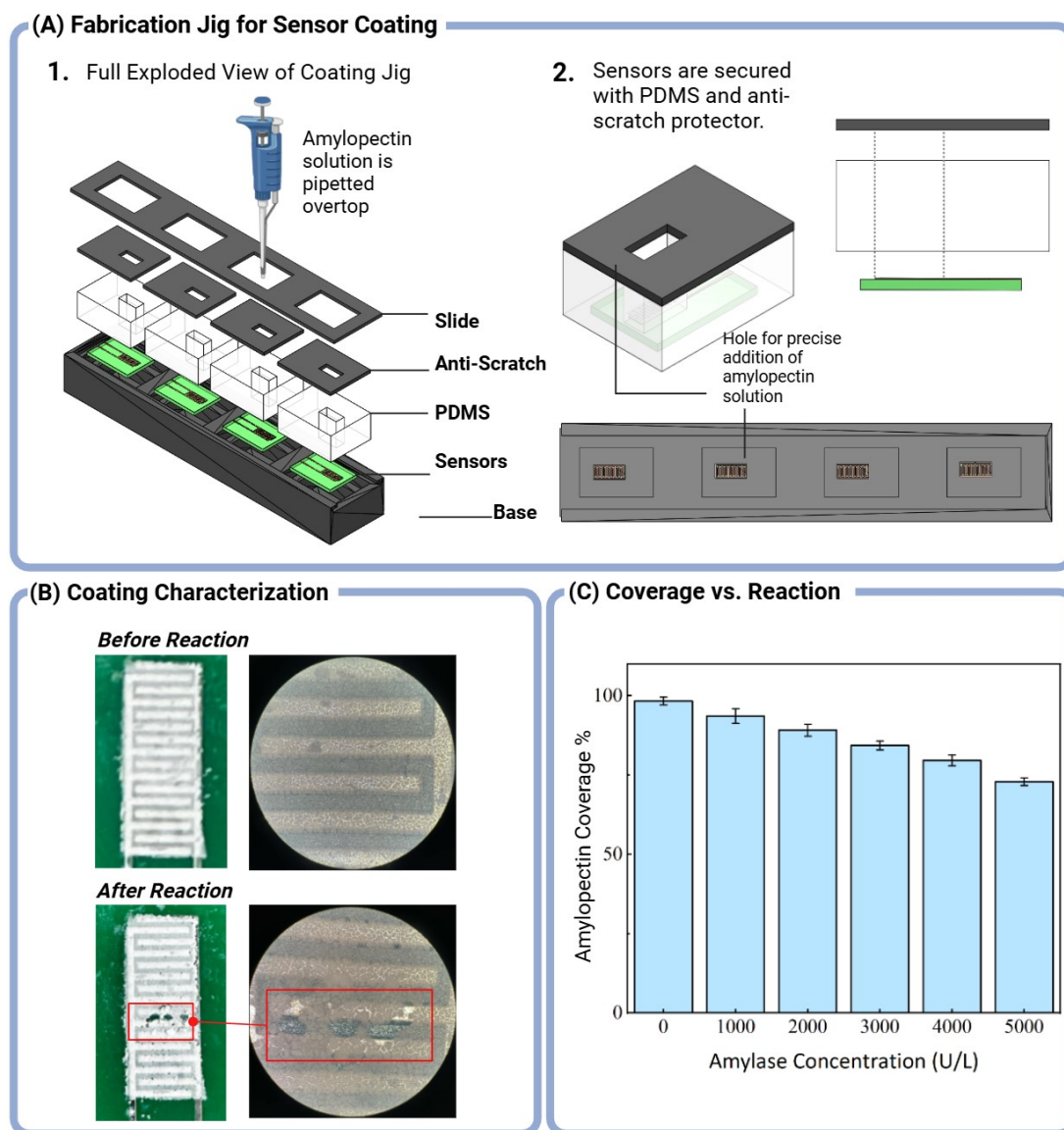


Figure 7.6: Coating Device, and Characterization. (A) Fabrication Jig for Sensor Coating: Overview of the components used for assembling the coating jig, including holders, sensors, PDMS layers, anti-scratch protectors, and slides. The setup enables precise addition of amylopectin solution for uniform coating. (B) Coating Characterization: Visual and microscopic analysis of the sensor before and after reaction, highlighting the amylopectin layer's formation and uniformity. (C) Coverage vs. Reaction: Quantitative analysis of amylopectin coverage percentage across varying amylase concentrations (0–5000 U/L), demonstrating coating efficiency under different conditions.

Coating quality and consistency play a key role in the detection of amylase. However, the coating is prone to mechanical disruption. For example, pipetting samples into the testing jig can minimally disrupt the coating, causing additional undesirable frequency shifts. Therefore, careful handling is required to avoid physical disruption of the coating during testing. To ensure that the coating is optimized, two additional sets of analyses were conducted. Figure 7.7(A) illustrates how the volume of amylopectin coating affects amylase detection. Sensors were coated with varying volumes of amylopectin, from 75 μL to 200 μL , and tested by placing them in the testing jig and exposing them to DI water. The frequency shift was defined as the difference between the initial resonance frequency and the resonance frequency after one hour at room temperature. Because the sample contained only DI water without any amylase, a negligible frequency shift was anticipated. The results demonstrate that variations in coating volume significantly influence sensor performance. An optimal volume of 100 μL provides the smallest frequency shift (1.85 MHz) and the smallest error bar (± 0.2 MHz). This finding underscores the importance of precisely controlling the coating volume to ensure optimal sensor performance and maximize detection efficiency. Figure 7.7(B) compares the performance of tin and gold coatings as passivation layers for the sensor. Two sensors, each with a 100 μL amylopectin coating, were tested with an amylase solution at a concentration of 5000 U/L. The frequency shifts were measured by taking the difference between the resonance frequency at the initial time and after one hour at room temperature. The frequency shift for the sensor with the gold passivation layer was 8.372 MHz, while the sensor with the tin passivation layer exhibited a frequency shift of 8.169 MHz. Although the gold layer showed slightly better sensitivity, the difference was minimal. Given the significantly lower cost of tin and the trade-off between cost and performance, tin was chosen for subsequent experiments as a more practical and cost-effective option. Collectively, these findings demonstrate the sensor's robustness and practicality for detecting amylase. The optimal conditions for specificity, coating volume, temperature, and material selection for the passivation layer were identified.

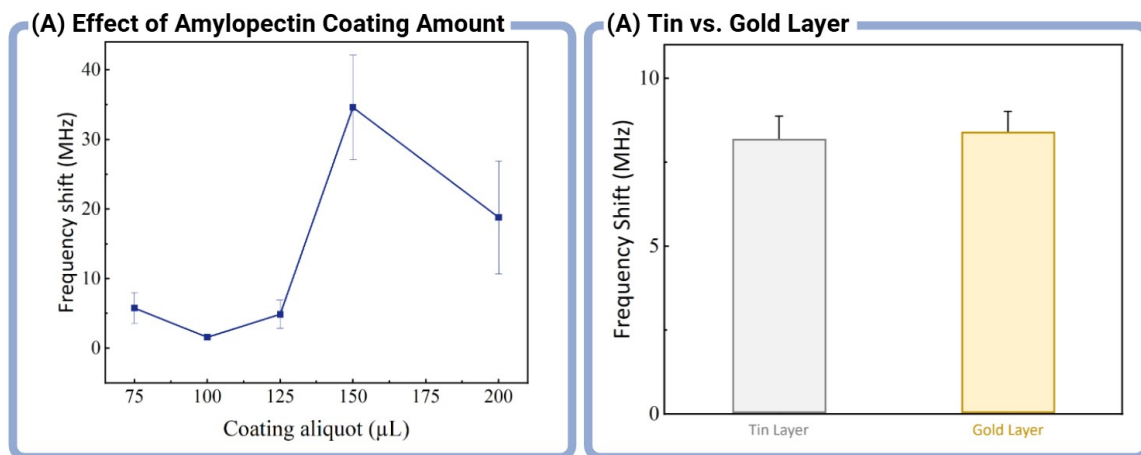


Figure 7.7: Characterization of sensor performance with varying conditions: (A) Effect of amylopectin coating amount on amylase sensing results. (B) Comparison of tin and gold sensor surfaces with a 100 μL amylopectin coating at 37°C .

7.4.4 Comparison with Optical Methods and Clinical Relevance

To validate the results obtained from the microwave-based biosensor, we used the EnzChek™ Ultra Amylase Assay Kit (Thermo Fisher Scientific) as a standard. This kit employs a fluorescence-based sensing principle, where the enzymatic activity of amylase hydrolyzes a labeled substrate to produce a fluorescent product, and the intensity of the fluorescence is proportional to the amylase concentration. Although the kit's standard curve in its documentation is designed for concentrations ranging from 0 to 20 U/L, it is possible to measure higher enzyme activities by diluting samples into this range. For instance, in our experiments with amylase concentrations of up to 2000 U/L, we appropriately diluted the samples to bring their activity within the kit's linear detection range. The original enzyme activity was then calculated by multiplying the measured values by the dilution factor. The performance of the microwave-based biosensor was benchmarked against the optical amylase assay. In deionized water and SPF, the biosensor demonstrated an almost linear response to amylase concentrations ranging from 1000 to 5000 U/L, with consistent shifts in resonance frequency, as shown in Figure ref. The reason for starting at 1000 U/L is that for concentrations lower than this, the biosensor did not yield repeatable trends when amylase was added to either deionized water or SPF without amylase, establishing 1000 U/L as the minimum concentration at which reliable frequency shifts could be observed. In contrast, the optical test provided fluorescence intensity readings for

the concentration range of 0 to 2000 U/L, as depicted in Figure 7.8. This limitation in concentration range for the optical assay arises from the necessity for samples to be diluted into the kit's linear range, as explained above.

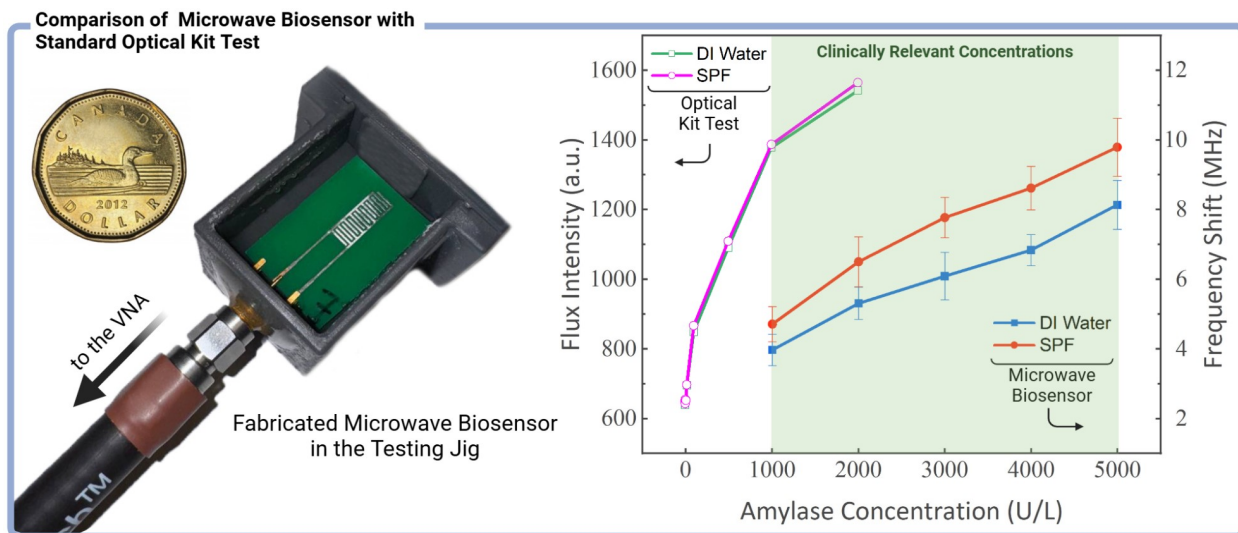


Figure 7.8: Comparison of the proposed sensor with the optical amylase kit test. The left side of the figure shows the fabricated microwave biosensor placed in the testing jig, with a Canadian dollar coin for scale. The right side displays a comparison between the resonance frequency shift of the microwave biosensor (red line) and the flux intensity of the optical amylase kit test (blue line), both for different concentrations of amylase. The microwave biosensor operates within the clinically relevant concentration range of 1000 to 5000 U/L in deionized (DI) water and simulated peritoneal fluid (SPF) with 100 μ L of amylopectin coating at room temperature. In contrast, the optical amylase kit test is shown for amylase concentrations ranging from 0 to 2000 U/L in both DI water and SPF at room temperature.

The microwave-based biosensor demonstrated comparable sensitivity and accuracy to the optical kit while offering several distinct advantages. Unlike the optical assay, which is costly (approximately 500 CAD per kit) and involves multiple preparation steps, the biosensor allows for real-time and continuous monitoring of amylase levels. Moreover, it has the potential to be integrated into bedside setups, enabling direct, continuous observation of a patient's drain fluid to track amylase levels in real time. While the microwave biosensor is less effective at detecting very low concentrations compared to the optical kit,

it excels at measuring high amylase concentrations, with a range that extends up to 5000 U/L. This capability is particularly important in clinical situations like severe anastomotic leakage, where amylase levels in drainage fluid often become significantly elevated. In these circumstances, the optical kit requires dilution to bring the sample within its measurable range, adding complexity and time to the analysis. In contrast, the biosensor can measure these elevated levels directly, making it a more practical, efficient, and cost-effective solution for clinical applications involving high amylase concentrations. The results emphasize the potential of the sensor to detect amylase as a biomarker for anastomotic leakage. The study simulated realistic clinical scenarios by starting with DI water and advancing to SPF. The frequency shifts observed across different amylase concentrations confirmed the sensor's ability to deliver reliable and specific measurements. These findings lay a foundation for future clinical evaluations and potential integration into postoperative monitoring systems.

7.5 Uncertainty Analysis

Despite the demonstrated sensitivity and specificity of the proposed microwave biosensor, several sources of error and uncertainty must be acknowledged. These uncertainties can be broadly divided into systematic and operational categories, both of which influence the precision, repeatability, and clinical applicability of the sensor.

Systematic errors are primarily associated with the measurement instrumentation, material properties, and sensor architecture. The frequency measurements were conducted using a VNA, which has a resolution limit below which frequency shifts cannot be confidently distinguished from noise. While the sensor displayed detectable shifts in the MHz range, especially at higher amylase concentrations, this resolution limit becomes a constraint when operating at lower concentrations or over short time intervals. In addition, environmental temperature variations, despite being monitored and controlled during experiments, can impact both the enzymatic activity of amylase and the dielectric properties of the surrounding medium, introducing fluctuations in sensor response. The fabrication of the sensor on an FR4 substrate and the use of thin amylopectin coatings also introduce variability; inconsistencies in coating thickness or uniformity, particularly at the edges, can affect the effective sensing area and shift magnitude. Moreover, connector-related effects, such as SMA connector alignment, soldering imperfections, and cable shielding, may alter signal transmission and add parasitic reflections, subtly affecting the VNA readout.

Operational errors, on the other hand, arise from the manual procedures involved in sensor coating, sample handling, and data collection. The amylopectin coating, though op-

timized for volume and stability, is sensitive to mechanical disturbance. Small disruptions, such as from pipetting or surface agitation during sample loading, can lead to non-specific shifts in resonance frequency. Human error in coating application, incubation time, or reagent preparation can contribute to variability between sensors or test cycles. Furthermore, despite efforts to maintain consistency, the volume and placement of the test fluid in the jig can slightly alter the local dielectric environment, affecting the reproducibility of measurements. Finally, while the sensor was validated using a standard amylase assay kit, the optical kit itself has limitations in concentration range and requires dilution steps, which can introduce additional uncertainty and complicate direct one-to-one comparisons.

This analysis emphasizes that while the current platform offers significant promise for point-of-care detection of amylase in postoperative drainage fluid, further refinements, such as automated fluid delivery, improved coating stability, and embedded environmental compensation, could enhance its clinical robustness. Future iterations may also benefit from integrated thermal and fluidic control systems to minimize both systematic and operational variability during extended bedside monitoring.

7.6 Summary

This paper presents the development of a microwave-based biosensor for detecting amylase in peritoneal drainage fluid, targeting the elevation in amylase associated with anastomotic leakage events following colorectal surgery. The sensor was designed using a printed circuit board fabricated on an FR4 substrate with interdigitated copper electrodes passivated with tin or gold coatings. It was then chemically functionalized with an amylopectin-based coating to enhance specificity and selectivity for amylase detection. The sensor's geometry and performance were first optimized through full-wave simulations, and the coating process was refined to maximize sensitivity and stability. Subsequently, tests were conducted to determine the optimal coating volume and operating temperature. The best performance was achieved with a 100 μL amylopectin coating at 37°C. The sensor's performance was then evaluated in deionized (DI) water and simulated peritoneal fluid (SPF) to mimic realistic conditions. Significant frequency shifts were observed when exposed to high-concentration amylase solutions, demonstrating sensitivity within a near-linear range of 1000–5000 U/L. To further validate the sensor's performance, its results were benchmarked against the EnzChek™ Ultra Amylase Assay Kit, a standard optical assay. The biosensor exhibited comparable sensitivity and accuracy to the optical kit while offering several distinct advantages, including lower costs and the potential for quasi-continuous monitoring. Unlike the optical kit, which requires sample dilution for high-concentration measurements, the

microwave-based biosensor directly detects elevated amylase levels, simplifying the workflow for clinical applications. These findings underscore the potential of this miniaturized, portable microwave biosensor as a highly specific platform for quasi-continuous, long-term detection of elevated amylase concentrations linked to anastomotic leakage. Its ability to provide early and continuous monitoring over extended periods, combined with its high specificity, makes it a promising solution for integration into point-of-care (POC) systems. When benchmarked against the clinically relevant gold standard (optical kit), the biosensor confirms its suitability for both early detection and long-term monitoring, offering significant improvements in post-operative care for colorectal surgery patients.

Chapter 8

Conclusion and Recommendations

8.1 Contributions of this Thesis

This thesis contributes to the development of microwave-microfluidic sensors for the detection of microplastics (MPs) in environmental samples, offering a scalable and cost-effective approach for real-time monitoring of MP contamination. The integration of microwave sensing technology with microfluidics overcomes several limitations in existing MP detection methods, such as sensitivity issues, sample handling challenges, and analytical difficulties. Additionally, this work extends the application of these sensors to the biomedical field, providing a potential platform for monitoring biomarkers in clinical environments.

The first contribution, presented in Chapter 4, introduced the use of a microwave-microfluidic sensor for detecting MPs in water. This exploratory study revealed that the resonance frequency of the sensor could be shifted depending on particle size, concentration, and temperature, showcasing the potential of this technology. Despite the sensor's detection limits being higher than typical MP concentrations in freshwater environments, this study laid the foundation for future improvements to enhance sensitivity and applicability.

In Chapter 5, the development of a sensitivity-enhanced microwave sensing platform marked a significant advancement. The incorporation of coupled planar microwave resonators allowed for real-time characterization of MP size and concentration. The use of a disposable sample holder for multiplex testing addressed cross-contamination concerns, making the system more suitable for field applications. While the sensor demonstrated accurate monitoring capabilities, challenges such as deposition rates and resonance frequency shifts at higher concentrations were identified, highlighting areas for further optimization.

Chapter 6 introduced an AI-powered microwave-microfluidic platform that expanded the sensor’s functionality from simple detection to a comprehensive analysis tool. By integrating a K-nearest neighbors (KNN) algorithm, the platform enabled accurate prediction of MP types based on raw sensor data and allowed for single-particle detection. This enhanced the platform’s potential for precise MP quantification and improved its efficiency in environmental monitoring. However, additional improvements in scalability and error reduction were suggested to increase the system’s performance, particularly for detecting smaller MPs.

Finally, Chapter 7 demonstrated the versatility of the microwave-microfluidic sensor by adapting it for biomedical applications, specifically for detecting biomarkers in postoperative peritoneal drainage fluid to monitor anastomotic leakage. This project highlighted the platform’s potential for real-time clinical diagnostics in addition to environmental monitoring. While promising, further refinement of the system is required to ensure its accuracy and effectiveness in complex biological environments.

Overall, the contributions of this thesis represent significant advancements in both environmental monitoring and biomedical diagnostics, offering a novel approach to MP detection and the potential for widespread application in diverse fields.

8.2 Recommendations for Extension of Work

This thesis has presented the development of microwave-microfluidic sensors for MP detection, spanning from bulk monitoring methods to single-particle detection and biomedical applications. While significant advancements have been made, further research is needed to refine and extend the capabilities of these systems. The following recommendations are divided into three main categories:

Bulk Monitoring Methods

In Chapters 4 and 5, the focus was on bulk monitoring methods for MP detection. The study demonstrated that microwave-microfluidic sensors could effectively detect MP particles, but the system’s limit of detection (LOD) remains too high for practical applications in environmental monitoring. Future work should focus on reducing the LOD by addressing key limitations identified during the study. Enhancements to the microwave structure, microfluidic platform, and sensor material are crucial to increasing sensitivity and improving the detection of smaller particles (less than 20 μm). Additionally, exploring pre-concentration methods, such as on-chip particle capture or external filtering, could help amplify particle concentrations, making detection more efficient.

Field deployment capabilities could be further refined by designing portable and multi-sensor systems. Combining multiple sensors tailored to detect specific types of microplastics or targeting different size categories could improve the system’s versatility and allow for scalable environmental monitoring.

Single-Particle Monitoring

In Chapter 6, single-particle monitoring using AI-powered microwave-microfluidic sensors was explored. This platform demonstrated high sensitivity and real-time classification of MPs across various materials and sizes, offering great potential for detailed MP characterization. However, several areas for improvement remain. First, the prefiltration technique using a sieve stack can become clogged, leading to inaccuracies in particle sorting. Future work should investigate more efficient and reliable microfluidic techniques for pre-sorting particles to enhance the system’s accuracy.

Flow control is another limitation that could be addressed to improve detection sensitivity. Ensuring that particles remain in the sensing region for longer periods would allow for more accurate resonance signals. Modifying the microfluidic channel design to incorporate flow regulation techniques could significantly enhance the platform’s overall sensitivity and measurement precision.

Furthermore, alternative sample introduction methods could be explored to prevent particle deposition issues seen with the syringe pump. Investigating funnel-based sample entry or a vertical pump system may offer more consistent and reliable flow, ensuring better transport of particles into the detection region.

Biomedical Applications

Chapter 7 extended the application of microwave-microfluidic sensors to biomedical settings, specifically for detecting biomarkers in postoperative peritoneal drainage fluid. Although the platform showed promising results, there are several key areas for further development to make it suitable for real-time clinical diagnostics. One major limitation is the lack of multiplexed biomarker detection. Future research should focus on developing biosensors capable of detecting multiple biomarkers simultaneously, providing a more comprehensive understanding of the physiological changes in patients.

Additionally, the system must evolve to support continuous monitoring of biomarkers, rather than relying on sporadic measurements. Incorporating real-time tracking of biomarkers directly into drainage systems would enable clinicians to monitor trends and identify variances more effectively. This continuous monitoring could significantly improve early diagnosis and intervention for complications such as anastomotic leakage (AL).

The integration of advanced materials, microfluidics, and AI algorithms will play a crucial role in the development of these next-generation biosensors. For example, utilizing nanomaterials like graphene or gold nanoparticles could significantly enhance the sensitivity and precision of multiplexed biosensors. AI-based data processing could also enable real-time analysis, providing actionable insights for clinicians.

The research presented in this thesis has laid the groundwork for the development of microwave-microfluidic sensors for both environmental and biomedical applications. However, to realize the full potential of these systems, further advancements are necessary. By addressing these challenges, future work will help optimize the performance of these platforms, enabling their broader application in both environmental monitoring and clinical diagnostics.

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