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Article

An innovative microwave cavity sensor for non-destructive characterisation of polymers

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Abstract

This paper investigates the feasibility of using an innovative microwave sensing technology to characterise and study various properties of polymer material such as difference between various polymer types, particle size and particle size distribution, contamination and pigmentation. A microwave sensor designed previously has been utilised to carry out this initial study to analyse the capability of microwave techniques to carry out the analysis. The curves obtained from material response to microwaves are distinguishable showing the shift to the lower frequency end with the insertion of polymer material. S_{11} measurements have shown distinctive peaks for each size and type of the sample tested. The results are quantifiable in terms of various polymer properties under consideration. In terms of S_{21} measurements, microwave sensor clearly distinguishes between coarse and fine polymer samples in terms of particle size. The effect of air voids in the sample and the particle size distribution has also been studied. The results are promising and justifies a thorough design and development of a dedicated microwave sensor unit for the characterisation of polymers. The sensor will have a significant industrial benefit in terms of costs associated with the industrial analysis, increase in the efficiency of manufacturing and production operation as well as material quality, control and validation.

Keywords: Microwave sensor; non-destructive analysis; microwave analysis; resonant cavity; polymer; polymer characterization.

1. Introduction

1.1. *Scope of the study*

The current research study was carried out to address the problems of an industrial partner, Matrix Polymers, regarding the characterisation of polymer material (polyethylene to be precise). For over four decades, the polymer industry as a whole is heavily relying on the conventional methods of polymer characterisation. Specifically the properties of interest for Matrix Polymer includes but not limited to the detection of various polymer types, their particle size and particle size distribution, contamination, the effect of pigmentation and air voids in the samples and many more. Matrix polymer has been seeking innovative ways to be developed and introduced in their production line to carry out such an analysis online or offline. As part of the collaborative efforts, Low Carbon Innovation Hub at Liverpool John Moores University has attempted to investigate the potential of using an innovative and unconventional microwave based sensing technology to measure and detect some of the above said properties.

Current practice at the premises of Matrix Polymers involved in this collaborative research work follows conventional mechanical and visual testing methods to study the above properties. These methods include sieve analysis and visual inspection of the samples. These methods are time consuming, need a lot of effort to carry out and are often inefficient, inaccurate and inconsistent. To overcome these drawbacks and improve the manufacturing process as well as the operational efficiency of the organisation, there was a need to provide the company with sufficient information on the potential of developing and introducing new and innovative method of measurement, analysis and detection. This paper is a proof of concept and explores the potential of introducing a microwave resonant cavity sensor as an alternative to the above methods. Further research work will follow to design and develop a bespoke sensor to analyse the properties of interest.

1.2. *Polymer characterisation techniques: An overview*

The physical properties of various types of polymeric systems depend on the factors such as their chemical constituents, configuration of their macromolecules, thermal characteristics, mechanical characteristics as well as the relationship among the chains (morphology) of the polymeric systems. There are numerous techniques available to analyse these complex features, and, they vary in their principle of operation [1-3]. The characterisation of

polymers may be divided into five parts, namely; molecular characterisation, melt rheology analysis, solid—state morphological characterisation, physical property determination and electrical property testing. Briefly, all these categories of testing methods analyse complex aspects of polymer characterisation such as [2, 3]:

- Molecular weight determination and compositional characteristics such as compositional distribution of polymers.
- Response of polyethylene to deformation forces, showing the molecular weight and branch distribution of polyethylene.
- Phase transition, thermal degradation, melting point and reaction kinetics of the polymers.
- Morphology of the polyethylene representing the relationship among the chains, the way molecules are arranged, conformation and orientation of them, structural and functional groups, etc.
- Elastic modulus, tensile strength, electrical resistivity and surface characteristics such as roughness which are more of a representative of the end product.

These existing techniques along with the properties they analyse and their drawbacks are summarised in Table 1.

Table 1: Summary of the analysis techniques for polymers, the properties studied and drawbacks

Technique	Properties analysed/studied of polymers	Drawbacks
Gel Permeation Chromatography (GPC)	Molecular weight & molecular mass distribution [2, 3].	These techniques are highly complex, destructive in nature and require high level of expertise [2, 3].
Size Exclusion Chromatography (SEC)		
Fourier Transform Infrared (FT-IR)		
Scattering or Viscometry		
Melt Flow Analysis using melt indexing	Polymer response to deformation forces [2].	Techniques are highly complex, require expense pieces of equipment, complex experimental arrangements, human expertise and are destructive in nature [2].
Rheometry analysis		
Melt Elongation analysis		
Thermogravimetric Analysis (TGA)	Phase transition, thermal degradation [3].	Destructive, laborious and require high level of expertise to operate the test equipment [3].
Differential Scanning Calorimetry (DCS)		

Thermomechanical Analysis (TMA)		
Mass Spectroscopy (MS)	Morphological characteristics [3].	These techniques are costly, time consuming, require laborious sample preparation and are destructive in nature [3].
X-Ray Diffraction (XRD)		
NMR technique		
Transmission Optical Microscopy (TOM)		
X-Ray Photoelectron Spectroscopy (XPS)		
Atomic Force Microscopy (AFM)	Physical characteristics [2, 3].	Laborious, time consuming, destructive and require time consuming sample preparation process [2, 3].
Electron Microscopy (scanning, transmission)		
Vibrational spectroscopy and chemical imaging using Raman spectroscopy, near and mid infrared techniques	Non destructive techniques to study the Molecular characteristics, functional groups, compositional characteristics, etc. [3].	The problem is the existence of overlapping peaks that pose a hurdle in the correct analysis. To overcome this problem, these techniques can be combined with other techniques that makes them very complex, time consuming and costly choice [3].
Malvern mastersizer alongwith the knowledge of refractive index, uses laser diffraction technique	Determine the particle size and particle size distribution [4].	There is an upper size limitation on the equipment, hence need to be combined with dynamic image analysis to yield the results [4]. Requires knowledge of additional parameters, expensive analysis kit, require expertise to operate the equipment [4].

1.3. Existing problem and alternative proposed technique

The current problem lies in the existing techniques being too complex, time consuming, costly and laborious. There is also a need of expertise in the area of testing to achieve accurate results. Despite being a long list, none of these techniques, except the laser diffraction, studies simple characteristics of polymers such as particle size, particle size distribution and the presence of contamination. Measuring these properties of polymers is a key requirement of Matrix Polymers to improve the efficiency of their manufacturing process. The size distribution method is also found to be very complex and costly.

The range of applications of the polymers also require a technique capable of doing rapid, reliable and accurate evaluation of polymers in terms of the above properties. This paper, therefore, propose an innovative, simple and effective microwave analysis technique that can study the particle size, particle size distribution, contamination and air voids in the

samples with least effort, higher efficiently, low cost and least level of expertise required to carry out the analysis. The technique if developed further can provide a reasonable solution to the industrial problems.

2. Microwave sensing theory and methodology

Microwave based sensing is relatively a new and developing technology. Microwave based permittivity measurements of the material and sensing for material characterisation has been much in use in the research for over couple of decades [5, 6]. Microwave frequencies at a single and multiple modes have been utilised by researchers such as Ren et al [7, 8] and Meng et al [9] to characterise the dielectric properties of various materials with and without the influence of high temperature (up to 1000°C). These research studies have used cavity perturbation and extended cavity perturbation techniques to accurately determine the values of dielectric constant and loss tangent. The results found to be accurate determination of complex dielectric properties, a key parameter in the characterisation of materials, and shows agreement with theory. Microwave analysis is a cost effective technique and has a capability to analyse small or large volumes of samples [10-12]. It is an instantaneous, robust and non-invasive method (operating at low power, i.e. 0 dBm or 0.001 watt). It uses Electromagnetic (EM) waves in the microwave band of the spectrum (300 MHz to 300 GHz). It is an efficient technique because it can easily propagate through low-loss substances such as plastics, glass, ceramic, etc. [12-15]. The instrumentation for measurements can be setup in minutes and the measurements taken in seconds providing real-time data [16, 17]. With all the advantages of the technique, there are also certain disadvantages. This includes higher degree of specialisation to analyse and interpret the results as well as the existence of the influence of multiple parameters such as density, moisture contents, temperature, etc. on the measurements results [18].

The technique has been implemented in various industrial applications. These include but not limited to the measurement of the dielectric properties of various materials/substances [6, 19, 20], monitoring of glucose concentration and blood glucose monitoring [21, 22], multiphase flow monitoring [23], characterisation of construction materials [15, 24], food industry, material moisture contents, etc. [25, 26] to name a few.

The fundamental principle of the microwave sensing is its ability to utilise material's dielectric properties to monitor any changes in the material itself. The material's dielectric properties are determined by its molecular structure. Any change in the molecular structure affects its dielectric properties and is reflected in the microwave spectrum obtained when the material interacts with microwaves [14, 17, 26]. The interaction may cause a change in the frequency, attenuation or reflection of the signal measured through the transmitted (S_{21}) and/or reflected (S_{11}) microwave signals also known as the transmission and reflection coefficients respectively. By considering how these parameters change at discrete frequency intervals, the change can be linked to the material type, its composition, concentrations of the constituents, etc. in the sample [26].

2.1. *Microwave sensor and experimental setup*

This experimental investigation utilised a cavity based microwave sensor technology (cavity resonator) to study the properties of (polyethylene) polymers, specifically, differentiating between various samples of polymers, their particle size and particle size distribution, contamination and air void content in the sample. It can also be classed as a black box where the waves are inserted from the input port interacting with the material and leaving through the output port. The interaction of these microwaves with the material under test helps in studying its properties. In the current study a cylindrical cavity resonator made out of aluminium was designed and used as shown in Figure 1. The cavity had a radius b of 6.5 cm and a depth d of 1.1 cm. Loop antennas were designed and used in the cavity, the position of which is shown in Figure 1. The cavity has a sample holder in the middle of it. The whole setup including the microwave source, i.e. Vector Network Analyser (VNA), input and output coupling structures, cables, connectors and cavity resonator is shown in Figure 2 for the understanding of the reader. The VNA was used as a microwave source. The feasibility study carried out was at the laboratory scale, the aim of which was to explore the potential of using the microwave sensing technique to study the properties of interest. The next stage of the study would be to not only develop a bespoke sensor unit but also a low cost microwave source capable of generating the frequency range of interest for the polymer analysis.
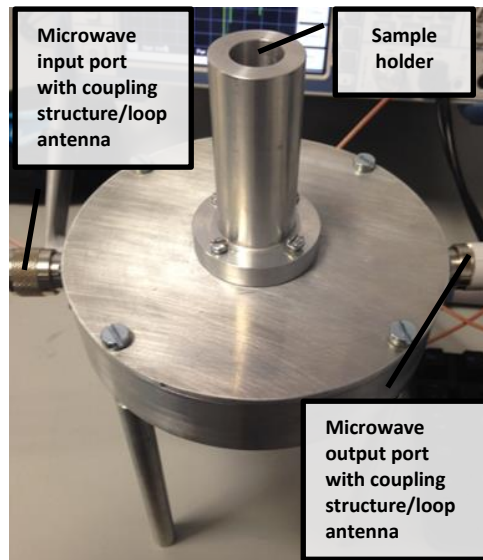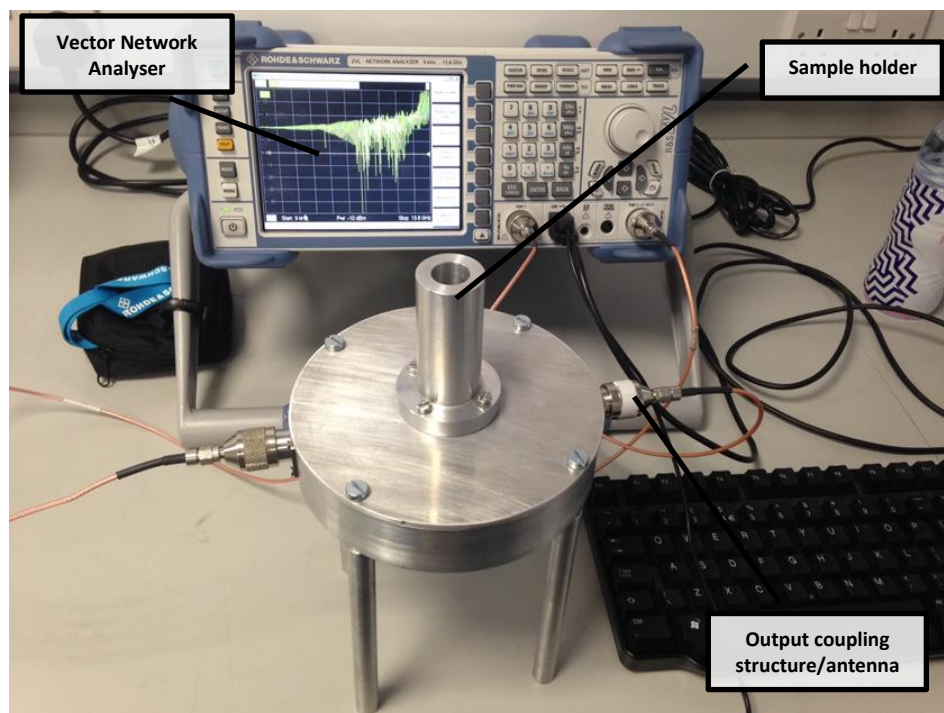
Figure 1: Microwave cavity resonator sensor

Figure 2: Experimental setup utilising the microwave sensor, VNA, cables and connectors

2.2. Cylindrical cavity theory

As mentioned earlier, a cylindrical cavity was utilised as a sensor to study various polymer properties. In a resonant cavity, resonance occurs when both the applied electric and magnetic fields form a standing wave pattern. There exists a single or multiple modes at a time inside the cavity, each of which corresponds to a specific electric and magnetic field

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pattern. Each of these modes has its own resonant frequency associated with it. A quality factor Q is used to represent the quality of the peak generated when microwaves interact with the material under test. The sharper the peak, the higher would be the Q value and the more readily a sample can be analysed. Thus, the Q factor improves the accuracy of the measurement [23].

The three fundamental modes generated in the cylindrical cavity are TM_{010}, TE_{111} and TE_{011}. In the case of TM (Transverse Magnetic) mode, the electric component of the wave is in the direction of propagation of wave. TE (Transverse Electric) mode on the other hand has its magnetic component in the direction of propagation of wave [23]. The theoretical value of the resonant frequency in the cylindrical cavity can be calculated using the equation (1).

$$f_{mnl} = \frac{c}{2\pi\sqrt{\mu_r \varepsilon_r}} \left[\left(\frac{p_{nm}}{b}\right)^2 + \left(\frac{l\pi}{d}\right)^2 \right]^{1/2} \tag{1}$$

In equation (1), ε_r is the relative permittivity of the material, μ_r is the relative permeability of the material, c is the velocity of light, d is the depth of the cavity, b is the radius of the cavity and p_{nm} is the nth root of the Bessel function of the mth order.

2.3. Samples preparation and testing

The polymer samples that were provided by Matrix Polymers were prepared and analysed in 15ml polypropylene test tubes. The samples are shown in Figure 3. The samples consisted of number of various types of polymers in terms of their size, contamination, pigmentation as well as size distribution. Typically, the company classed a sample as contaminated when it had a small amount of varying type of polymer mixed accidently in it. The polymer type tested was Linear Low Density Polyethylene (LLDPE). The description of the size range and types of polymer samples tested is given in Table 2.

The samples were tested both individually as well as in the form of proportional mix of two types of polymers in a test tube. As stated earlier, as a source of contamination a small proportion of yellow Materbatch pallets were mixed with the natural white polymer in the 15 ml sample tube to see the detection capability of the sensor. It was also important from the company's perspective to monitor the changes in the microwave spectrum with changing particle size. The samples analysed are listed in Table 2, whereby, sample 6 represents the contaminated sample.
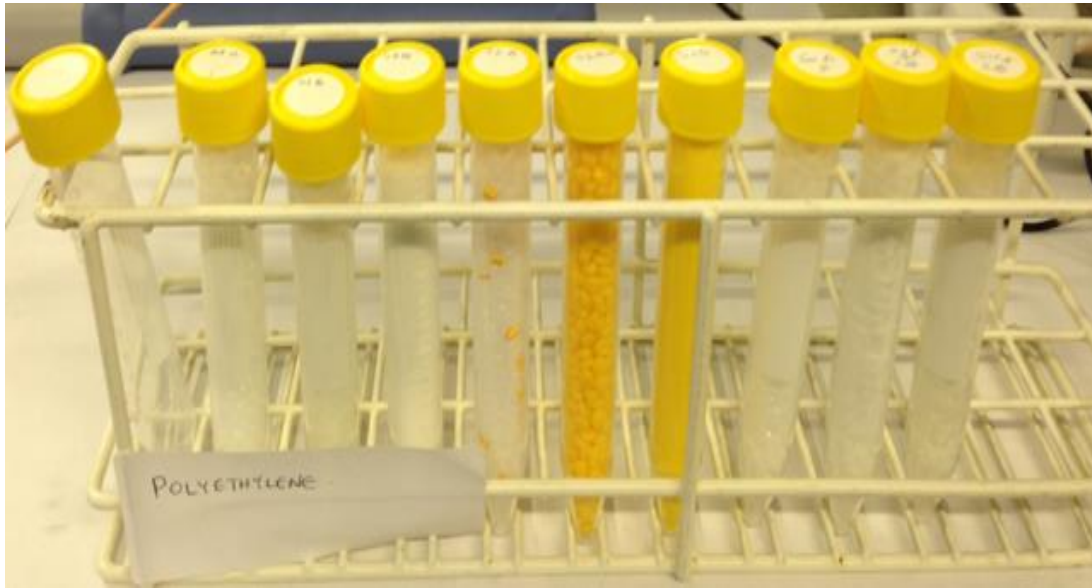
Figure 3: Polymer samples to be tested (Courtesy of Matrix Polymers)

Table 2: Samples analysed and tested using microwave sensing technique

Sample #	Sample Abbreviation	Sample type & description	Sample size (approximately)
1	Empty cavity	Empty cavity without any sample tube	N/A
2	Empty tube	Cavity with an empty tube	N/A
3	S1A	Natural Linear low density polyethylene (LLDPE) granules	2-4 mm
4	S1C	White LLDPE granules (elongated shape)	2 mm
5	S1D	White LLDPE powder	100-600 microns
6	S2A	Natural LLDPE granules with Yellow Materbatch pallets	2-4 mm with 2 mm yellow pallets
7	S2B+C	Yellow LLDPE granules	2 mm
8	S2D	Yellow LLDPE powder	100-600 microns
9	S1A+S1D	Combination of S1A and S1C in equal amounts (7.5 ml each)	Combination of 2-4 mm Natural + 2 mm white granules
10	S1C+S1D	Combination of S1C and S1D in equal amounts (7.5 ml each)	Combination of 2 mm white granules + 100-600 microns white powder

2.4. COMSOL simulations

COMSOL simulations were carried out to understand the wave pattern inside the cavity at the experimental measurement frequencies. Since the cavity was not specifically designed for the current feasibility, results of the simulations gave an insight into the resonant

frequencies and the modes generated. To compare the simulated and experimented results, the resonant frequency for the empty cavity was simulated and the modes identified. The results of an empty cavity (filled with air) are shown in Figure 4 for the resonant frequency of 4.05 and 7.16 GHz identified in the simulation results. These are also the experimental response frequencies identified for the measurements.
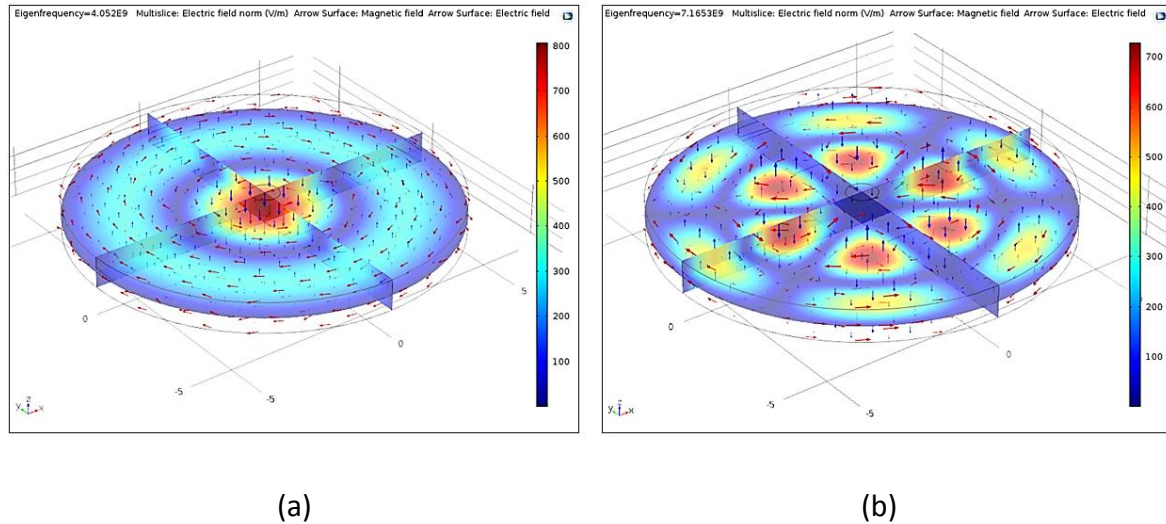

(a) (b)

Figure 4: Simulation results of the resonant frequencies generated inside the cylindrical cavity at (a) 4.05 GHz (b) 7.165 GHz

3. Results and discussion

3.1. Experimental procedure

Measurements of both the S_{11} and S_{21} parameters, in the first instance, were captured over the full spectrum range of the VNA between 9 KHz and 13.6 GHz frequency for all the samples. The results of both S_{11} and S_{21} measurements are shown in Figure 5 & 6 respectively. The spectrums were carefully examined to identify the resonant frequency peaks in the full range. This was to study the response of polymer samples to microwaves. It was found that the change in the frequency response of polymer samples to microwaves was at different frequencies for the S_{11} and S_{21} parameter. This was best identified in the region of 7.05-7.138 GHz for the S_{11} parameter and 3.95-4.10 GHz for the S_{21} parameter, hence the resonant modes varied. From the simulations and experimental measurements, the cavity was found to be operating in TM_{020} mode for the S_{21} measurements and in TM_{320} mode for the S_{11} measurements. Areas of peaks for further analysis are highlighted in Figure 5 & 6. The reason for selecting the microwave response region in the lower frequency range was to avoid the complex signals with higher order modes, complexities in the

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measurements and the cost associated with the development of sensors at high frequencies suitable for industrial use as well as to remove no response zones in the spectrum with higher noise level found below 3 GHz. The selection of the frequency range for further study was verified for consistency by carrying out repeated and multiple measurements of the samples in the cavity.
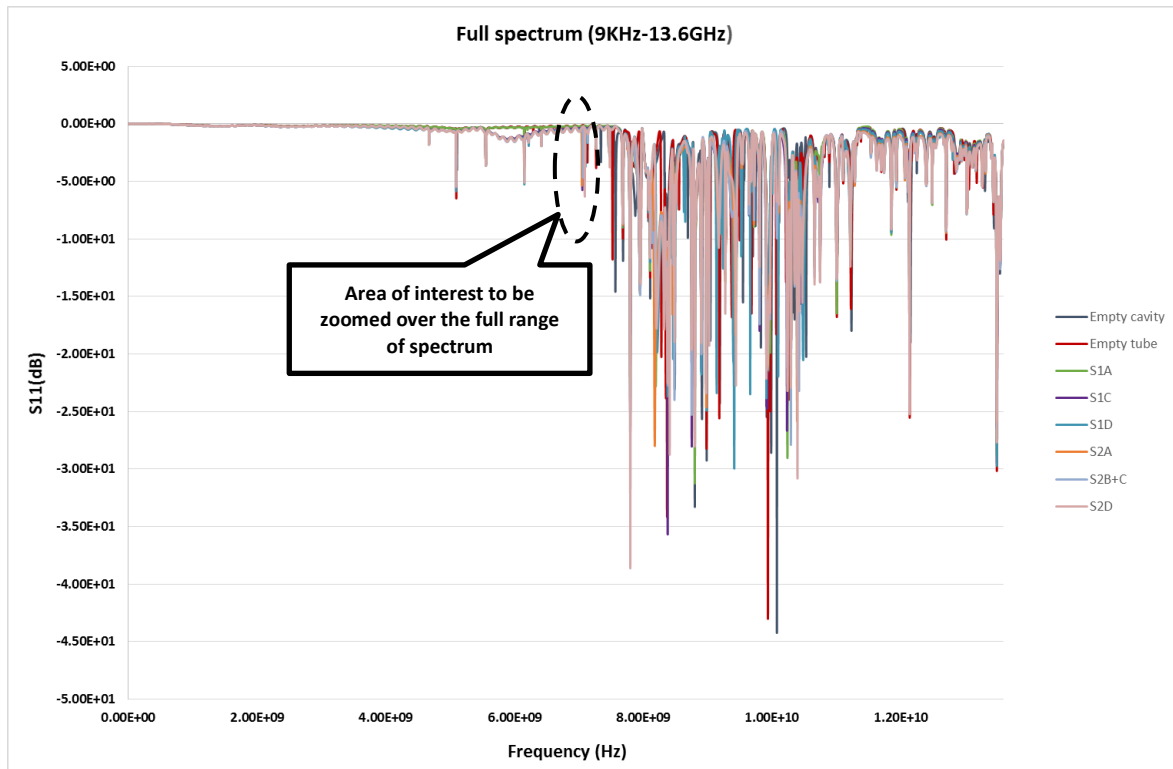

Figure 5: Reflected power S_{11} (dB) measurements using the microwave sensor for the full range of frequencies, 9 kHz to 13.6 GHz

The detailed material response to microwaves, resonant peaks as a result and the ability to differentiate between different types of polymers is shown and discussed in the results and discussion section. It can be noticed in these initial results that the sensor had the potential to identify various properties of the polymers under test, hence providing the proof of concept. It is proposed that a dedicated sensor system should be developed as a prototype. This will address any anomalies in the sensor currently utilised in this study.
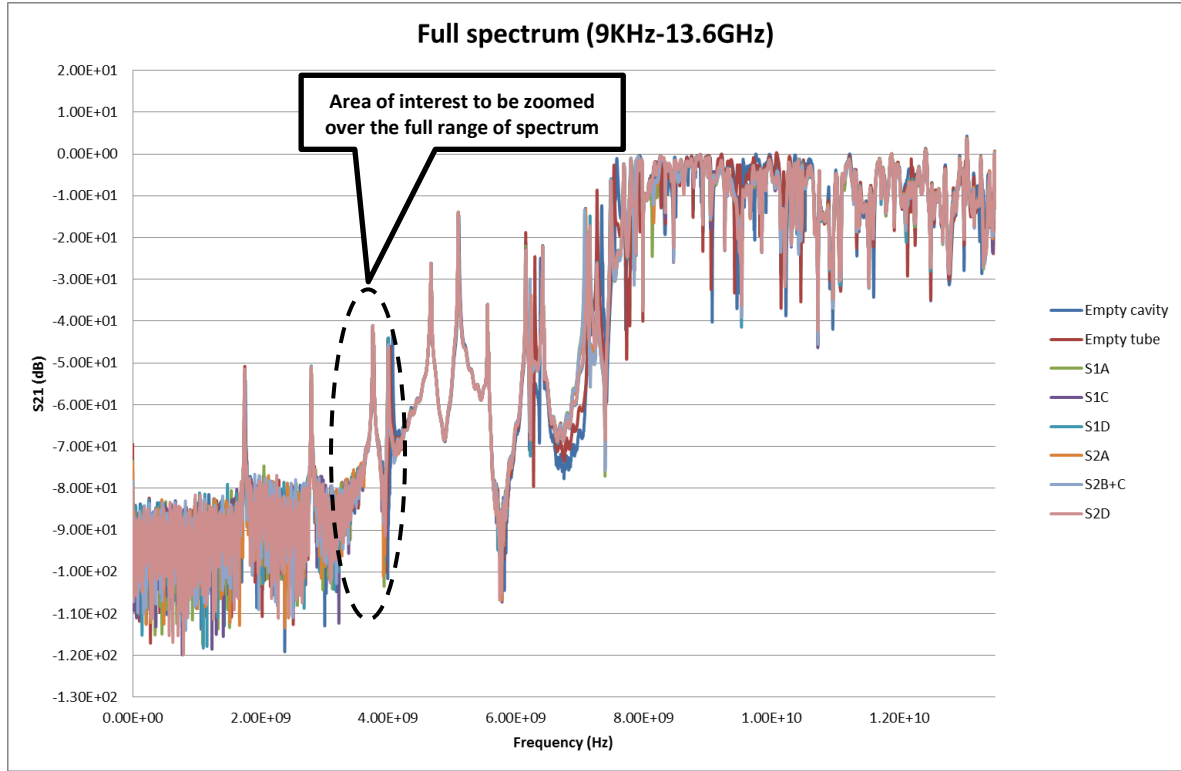
Figure 6: Transmitted power S21 (dB) measurements using the microwave sensor for the full range of frequencies, 9 kHz to 13.6 GHz

3.2. Theoretical resonant frequencies

Equation (1) was used to calculate the theoretical resonant frequencies. The simulated and experimental S_{11} measurement of the resonant frequency for the empty cavity was identified at 7.165 and 7.1334 GHz respectively (Figure 4 & 7). In case of S_{21} measurements, the simulated and experimental resonant peak of the empty cavity was at 4.05 and 4.06 GHz (Figure 4 & 9). The theoretical calculations of the resonant frequencies for the modes identified, i.e. TM_{020} and TM_{320} were also carried out (using equation (1)) to compare the theoretical, simulated and experimental results and are presented in Table 3.

Table 3: Theoretical, simulated and experimental resonant peaks for an empty cavity

Modes	Theoretical resonant frequency, f_{mnl} (GHz), $$f_{mnl} = \frac{c}{2\pi\sqrt{\mu_r \varepsilon_r}}\left[\left(\frac{p_{nm}}{b}\right)^2 + \left(\frac{l\pi}{d}\right)^2\right]^{1/2}$$	Experimental resonant frequency, (GHz)	Simulated resonant frequency (GHz)
TM_{020}	4.054	4.06	4.05
TM_{320}	7.1699	7.1334	7.165

3.3. Microwave spectrum results

As mentioned in section 7, the highlighted areas in Figure 5 & 6 have been investigated further to identify specific peaks and to see if they change with each of the different polymer samples. The highlighted areas in Figure 5 & 6 were zoomed in for the S_{11} and S_{21} parameters, and, the resonant peaks/response curves were identified in the frequency ranges of 7.05-7.138 GHz (Figure 7) and 3.95-4.09 GHz (Figure 9) respectively. The instrument (VNA) was calibrated and the measurements carried out between these frequencies.

3.3.1. S_{11} measurements analysis

The results obtained were promising and the response frequency of around 7.05-7.138 GHz (Figure 7) can identify distinctive peaks for each of the polymer samples compared to the baseline/control samples of the empty tube and empty cavity. By looking at the simulation results in Figure 4 (b) at the resonant frequency of 7.165 GHz, it is clear that the strong electromagnetic field area exists around the location of the sample insertion. It was interesting to see the influence of these fields on the microwave response curves after the sample insertion and is discussed below.
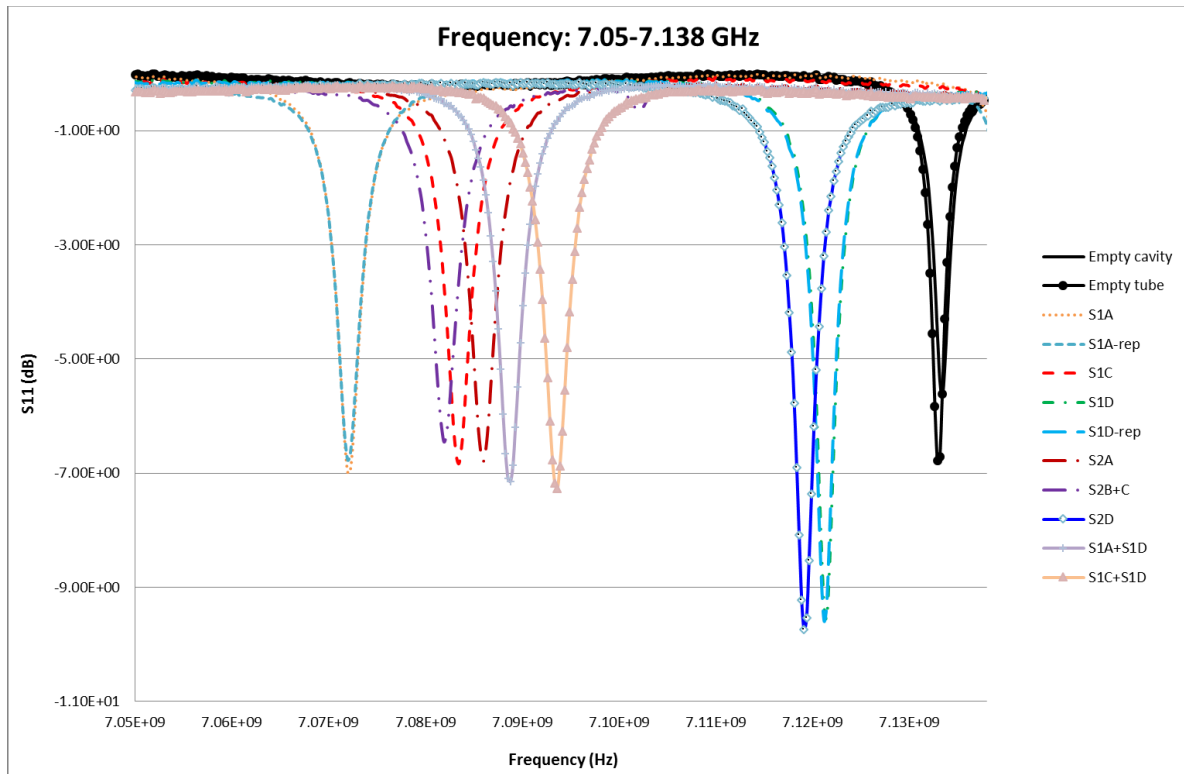

Figure 7: Reflected power S_{11} (dB) measurements for various polymer samples between 7.05-7.138 GHz

The microwave response curves obtained show that the microwave cavity is exhibiting high quality factor in its results. A detailed analysis of the Figure 7 shows that there is a significant shift towards the lower frequency (to the left) as well as change in the amplitude of the polymers samples when tested. This shows changes in the properties of polymer samples compared to the empty tube and empty cavity (right most curves) measurements. It is observed that the quality of resonant peaks are not affected by the insertion of polymer samples (loaded cavity) in comparison to the empty cavity. This could be attributed to the low loss nature of the polymers under analysis. Further analysis of Figure 7 shows that the resonant peak of the empty cavity and empty tube samples are identified at the frequencies of 7.1334 GHz and 7.1329 GHz respectively. Taking the empty tube as a control sample a significant shift has occurred in the frequencies of all the polymer samples to the left compared to the control sample showing a decrease in the frequencies and an increase in the permittivity value. Clarke [27] has listed the permittivity value of Polyethylene as approximately ~2.3 in comparison to the air which is 1. Similarly, a quantifiable frequency shift can also be observed between different polymer samples. The results of the frequency shift for each of the sample is presented in Figure 8 showing the frequency shift trend.
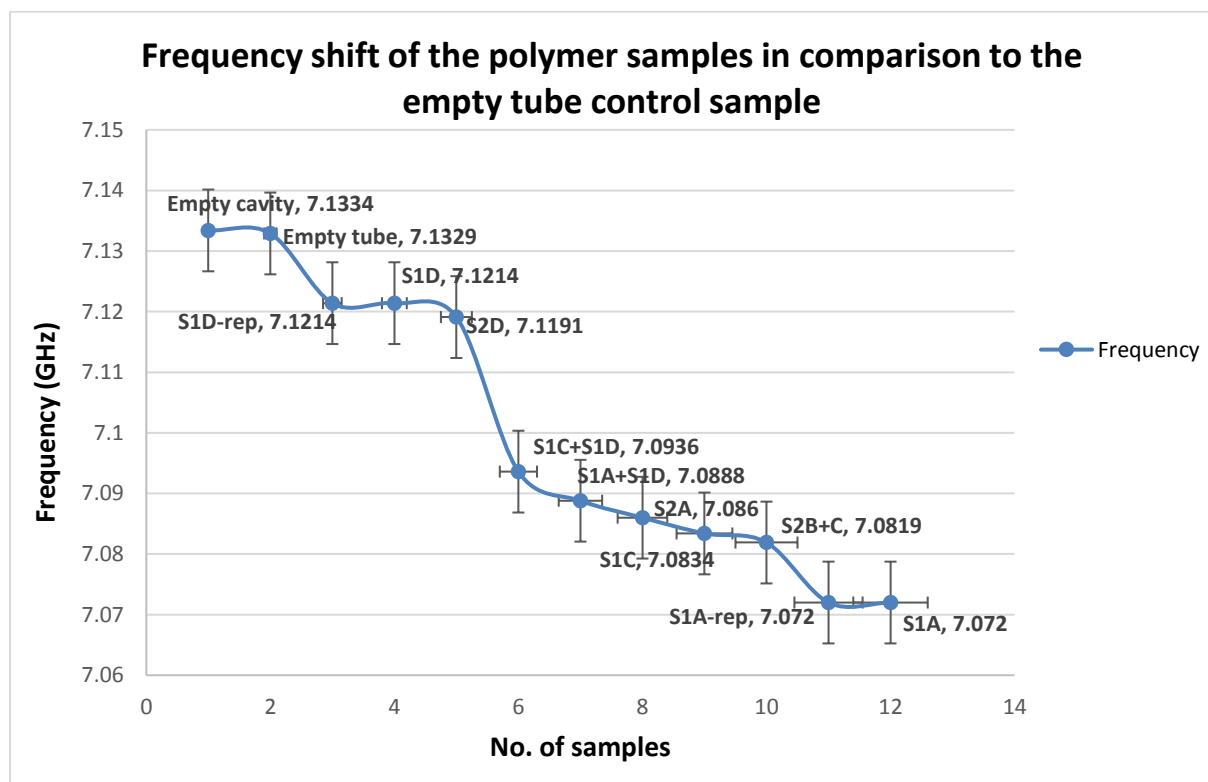

Figure 8: Graph of the frequency shift of polymer samples in comparison to the control sample of an empty tube

The results from Figure 7 and 8 show that:

- Maximum shift has occurred in the curve of sample S1A and S1A-rep with resonant peaks for both of them recorded at the frequency of 7.0720 GHz. This is approximately a 60.9 MHz shift towards the left which is a significant value. Both the S1A and S1A (repeat measurement of S1A) represents a sample of LLDPE granules of largest size as in Table 2. This shows that the shift is maximum in the case of largest size granules.

- The sample S2B+C had a resonant frequency peak at the frequency of 7.0819 GHz which is a shift of approximately 51 MHz. It is again a significant value, but less than the white granules of S1A. The S2B+C are the yellow LLDPE granules of the size 2mm as in Table 2. The larger shift could be accounted for the pigmentation in addition to the size of the sample in comparison to the sample S1C of the same size but white in colour.

- In comparison to larger granules, sample S1C was a different type of polyethylene sample, white in colour with granules of 2mm in size as in Table 2. The shift observed for this sample is approximately 49.5 MHz. This shift was smaller than the sample S2B+C of the similar size which was pigmented.

- The next highest shift is observed for the sample S2A which is a combination of S1A granules with a small amount of yellow pallets in it acting as a contaminant. The shift is around 46.9 MHz to the left from the control sample. This shows that when the sample of 2-4 mm white granules was contaminated with the yellow 2 mm granules, the sensor detected this contamination. The shift was still significant from the control sample, but also a lot less than the pure S1A sample.

- Another interesting aspect was to monitor the impact of mixing a powder with granules on the microwave response curve. This was aimed to show if the change in the size distribution could be detected by the microwave sensor. Both the S1A+S1D and S1C+S1D samples (Table 2) were the mix of granules sizes 2-4mm and 2 mm respectively mixed with equal amount of white powder of the size 100-600 microns. The powder was mixed with granules to monitor what is the impact on microwave response curves. The shifts to the left are again measurable and are around 44.1 and 39.3 MHz respectively. The resonant peaks for the corresponding shifts are 7.0888

and 7.0936 GHz respectively. The results shows that the impact of the addition of the powder in the granules is quite significant in terms of the shift in frequency.

- The curves of the samples S2D (100-600 microns yellow powder) and S1D (100-600 microns white powder) had their resonant peaks at the frequencies of 7.1191 and 7.1214 GHz. It is corresponding to the shift of 13.8 and 11.5 MHz respectively. This shows that the microwave response to the powder samples is the least in terms of the shift occurred. Yet, the values are high in comparison to the control sample keeping in mind the sensitivity of the microwave analysis technique. The repetition of sample S1D-rep also exhibited the peak at 7.1214 GHz showing the promising repetition capability of the sensor.

- Change in the amplitude of the samples were also observed in addition to the frequency shifts and could be used in the optimum design of the bespoke sensor unit.

From the analysis above it can be concluded that the varying response of the microwave sensor could be due to the varying type, varying size of granules, varying size distribution and the pigmentation. All the samples with larger granules of different types had the highest shifts to the left in comparison to the control sample. As the granule size became smaller, the amount of shift reduced yet was reasonable and measurable in comparison to the control sample of empty tube. With further investigation and experimentation (not covered in this experimental study), individual changes can be linked to the microwave response curves to carefully monitor the impact of each parameter.

3.3.2. S_{21} measurements analysis

Figure 9 shows the S_{21} (transmission coefficient) measurements of the highlighted area in Figure 6. The resonant peaks are presented in Figure 9 for various polymer samples. When analysing it can be observed that when polymer samples are introduced into the cavity, there is a significant shift in the microwave response curves to the left (towards lower frequency representing higher permittivity) in comparison to the control sample curve. This could be due to the strong electromagnetic fields concentration at the centre of the cavity at 4.05 GHz (Figure 4(a)), strongly influencing the microwave response when the sample is inserted into the cavity, hence a significant shift. The polymer samples tested are from Table 2 presenting different sizes (4 mm, 2-4mm and 100-600 microns) and types. The shifts are

following similar trends as in Figure 7. The retained sharp resonant peaks could again be attributed to the low loss nature of polymer samples. The difference between various coarse polymer samples represented by the shift in the frequency spectrum is fairly low compared to the reflected signals in Figure 7. The peaks however are observed to be significantly prominent, and, distinctive shifts in the frequencies with change in the samples were observed. Although the exact nature of factors influencing these shifts may not be known at this stage, it could possibly be because of the lower sensitivity exhibited by the sensor in its S_{21} signal response. The powder samples S1D and S2D (100-600 microns, as in Table 2) still shifts significantly to the left from the control samples.
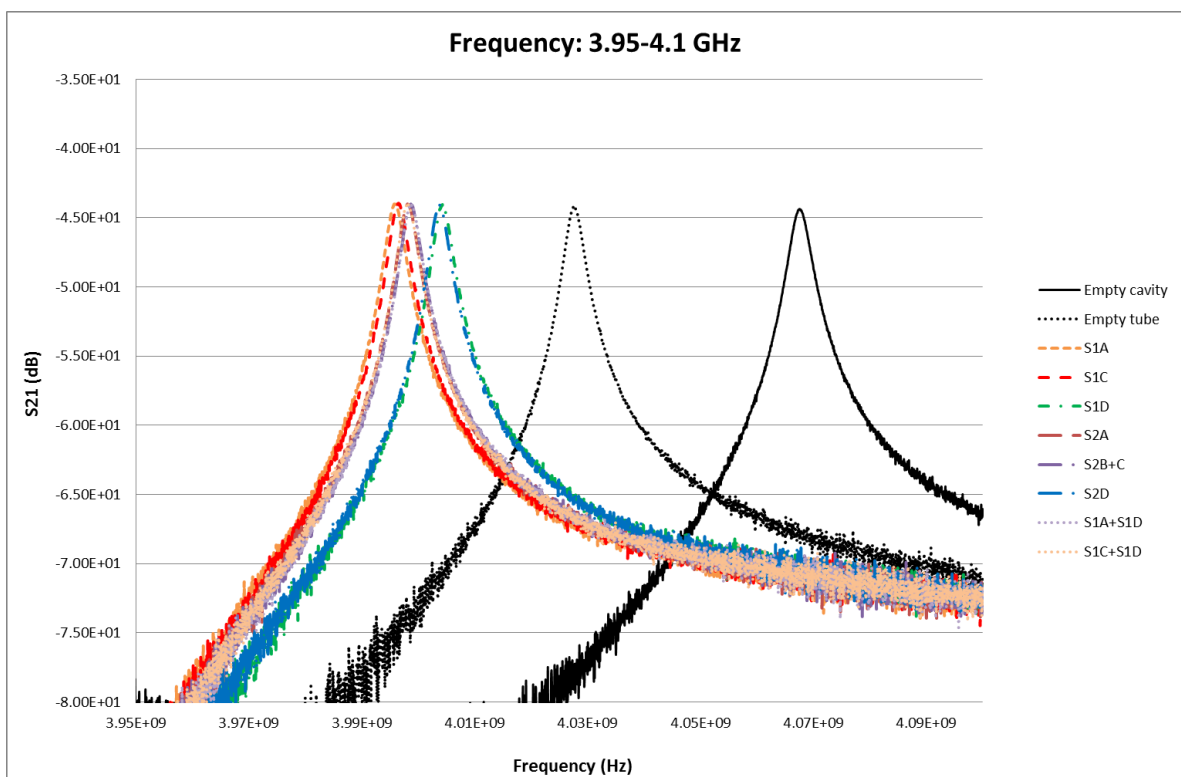

Figure 9: Transmitted power S_{21} (dB) measurements for various polymer samples between 3.95-4.10 GHz

The measurements for them are distinctive in comparison to the samples with granules. The peaks are significantly to the right identified at higher frequencies than the polymers of large granule size, i.e. 2 mm and 2-4 mm. This nature of the shift is similar in trend to the S_{11} measurements in Figure 7. Maximum shift towards the lower frequency is observed in the samples of white granules of sizes 2-4 and 2 mm respectively. The samples S2A (white granules with yellow pallets as a contamination), the yellow granules sample S2B+C and the

two samples, S1A+S1D and S1C+S1D, containing a mix of powder and granules have their resonant curves in between the pure white granules and powder samples.

There is a lesser distinction observed between various granule types and their sizes in Figure 9 in comparison to the S_{11} measurements presented in Figure 7, however, still identifiable. Although, the frequency span over which the measurements were taken was smaller for S_{11} compared to S_{21}, more significant distinction and higher frequency shifts was observed for various polymer samples in the case of 7 GHz frequency range, indicating the high sensitivity. The results from the S_{21} measurements could possibly be tuned to improve the sensitivity of the instrument with a careful design of a bespoke sensor unit that uses transmission coefficient S_{21} as the measurement signal.

3.3.3. Impact of air voids and size distribution on the S_{21} measurements

In addition to differentiating between the polymer sizes, types, pigmentation, contamination and mixtures, an additional test was conducted on identifying the impact of air voids and size distribution on the microwave response curve of the sample. It is presented in Figure 10. This was a preliminary test to investigate the capability of microwave sensor in identifying these two parameters. Sample S1A was analysed first and then rotated around its axis to re-analyse it at four different angles. It can be observed that there is a small shift in the resonant peak frequency of each of the samples. The percent change in the resonant frequency was worked out to quantify it. The percent change is observed to be the following:

- S1A sample's resonant peak is identified at the frequency of 3.9958 GHz. The corresponding frequencies in the order of reduction are 3.9946, 3.9941, 3.9938 and 3.9937 GHz for the samples S1A_L, S1A_U, S1A_D and S1A_R respectively. From the frequency shifts, it was calculated that the approximate percent change in the frequencies of the samples from S1A is 0.03, 0.045, 0.05 and 0.052% respectively. This small percentage change allowed investigation of irregularities, the air voids and changes in the size distribution of the sample S1A. The sensor can be designed to either reduce this change or accommodate it as an error which would depend on the importance of the sensitivity of the results.
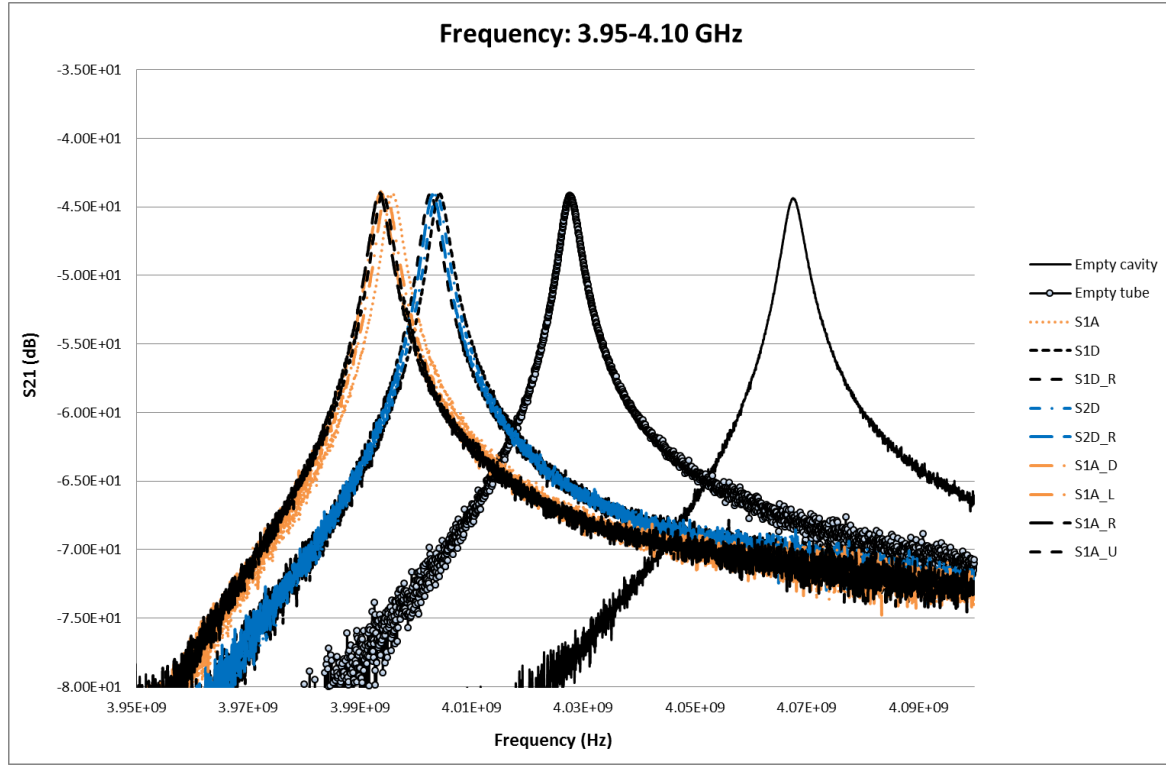
Figure 10: Transmitted power S$_{21}$ (dB) measurements for various polymer samples between 3.95-4.10 GHz, with S1A, S1D and S2D analysed by rotating around its axis

- S2D was also rotated once to observe changes in the size distribution and air voids. The peak frequency of the sample is observed at 4.0038 GHz whereas the rotated sample S2D_R is observed at 4.0032 GHz. This is approximately a shift of around 0.6 MHz towards the lower frequency. This equals to approximately 0.014% change in the frequency. Again the amount of change is very small and can be accommodated as a design parameter.

- Both the S1A and S2D samples were repeated twice showing the same results on repetition. This highlights the consistency of the results from the microwave sensor technique but shows a need of further detailed investigation.

- A similar trend in the measurement of S1D and S1D_R sample was observed with a minor shift in the frequency on rotation of the sample tube.

3.3.4. Repeatability of measurements & statistical analysis

To establish any measurement and analysis method, accurate repetition of the results is important. Repeatability of the method helps to prove that the analysis method is reliable and can be used with confidence. For the microwave analysis method presented in this

paper, it was important to show how repeatable the measurements are. To serve this purpose both the S_{11} and S_{21} measurements for each sample was repeated 5 times. To provide the evidence of repeatability and consistency of the measurement technique, repetitions of S_{21} measurements are presented in Table 4. The repeat measurements of the resonant peaks recorded for each sample was averaged to obtain the average resonant peak. Standard deviation as then calculated for each sample. Also, the percentage deviation from the averaged resonant peak was calculated. The results are presented in Table 4. It can be seen from the results that the variation in the standard deviation of the averaged resonant frequency is very small with a maximum value recorded at 0.00019%.

Table 4: Averaged resonant frequency and the standard deviation of the test results to measure the repeatability of the measurements and percentage deviation from the averaged values

Sample type	Measurement type	Frequency range (GHz)	Averaged resonant frequency (GHz)	Standard deviation $\sigma = \sqrt{\frac{\Sigma(x-\mu)^2}{N}}$ (MHz)	Deviation in % from the avergaged resonant frequency (Approx.)
Empty cavity			4.0722	0.0748	<0.00001%
Empty tube			4.0311	0.250	<0.00006%
S1A			3.9993	0.190	<0.00005%
S1C	S_{21}	3.95-4.1	4.0004	0.332	<0.00009%
S1D			4.0062	0.261	<0.00007%
S2A			3.9973	0.734	<0.00019%
S2B+C			4.0013	0.256	<0.00007%
S2D			4.0044	0.354	<0.00009%
S1A+S1D			3.9968	0.169	<0.00005%
S1C+S1D			3.9985	0.126	<0.00004%

4. Conclusions and Recommendations

The initial feasibility study carried out in assessing various properties of polymers using microwave based sensing technique has shown good potential to carry out real time measurements for industrial based applications. The results demonstrate that the technique is capable of detecting various properties of polymers, hence, can be further developed as an alternative to existing conventional time consuming physical and chemical testing methods. Individual peaks or sections of the spectrum were analysed to find areas of interest and to investigate the capability of microwave sensors to differentiate between samples. These samples consisted of different polymer sizes, types, size distribution. The analysis helped in studying the properties of interest, specifically the sample type, polymers

particle shapes and size, contamination in the sample, pigmentation, sample size distribution and air voids. The following are the conclusions and recommendations from the investigation.

- The study on the feasibility of using a microwave sensing technique has successfully shown differences in individual samples of polymers and they have been distinctively identified. These differences are noticeable between the samples in terms of various polymer properties discussed above. These results however, although promising and showing the potential of microwave sensors to perform the polymer analysis, need further investigation through the design of a dedicated sensor that can improve the performance of the microwave sensor used in this study. Errors such as the change in the microwave response curves with air voids and particle size distribution could be addressed and proposed investigation will also help to relate the changes to specific properties of polymers.

- The shift to the lower frequencies (left) demonstrated the increase in the permittivity value when the polymer sample was inserted in the cavity. As we know that permittivity of air is 1, the shift to the left shows the increase in the permittivity value. Hence, the cavity was able to differentiate distinctively between the air and polymer samples.

- The irregularities in the sample, size distribution and/or air pockets are possibly the cause of slight change when the same sample tube was rotated around its axis. This has been studied through a specific test conducted, shown in Figure 10. As discussed above, the errors were quantified and the percentage of error was calculated. These results could help in the design of a dedicated sensor unit to address the anomalies or to accommodate the percentage of error that could possibly exist in the sensor. However, this design compromise should not affect the industry standards on the quality control of the polymer products.

- The small change monitored in Figure 10 presented the size distribution and change in it when the sample was rotated. This could, on the other hand, be used in the design of a new sensor to identify the size range of the sample.

- This was a preliminary feasibility focusing on exploring the potential of using the microwave analysis technique and microwave cavity sensor to study polymer

properties. The results indicate that the study can be extended further and a detailed analysis of the quality factor Q can be added to the next stage. A dedicated sensor is proposed to be developed and the individual properties of polymers linked to the analysis through advance statistical analysis. The analysis can be strengthened further by adding artificial intelligence and neural network analysis techniques. This will be part of the next phase of the study.

- The theoretical calculations were close to the simulated and experimental results. This demonstrate the accuracy of the results. This also shows that the experimental measurements carried out are reproducible as demonstrated in Figure 10.

- The repetition of measurements as shown in Table 4 showed high accuracy in the measurements and the results were reliable.

- The study was innovative because the literature study had shown that there exists no simple monitoring system that can analyse quickly and cost effectively with minimal effort the properties such as change in the size, contamination in the sample, as well as the pigmentation. Microwave based sensing is proposed to be a simple solution which is robust and the measurements can be carried out in real-time in seconds.

- The study has shown that Electromagnetic wave sensors operating in a microwave frequency region are capable of detecting the difference in various polymer samples. A dedicated sensor needs designing and developing to target the anomalies and to function more accurately. This will also include building up a database of all the signatures captured for the future reference and analysis.

In the light of the above results and discussions it is recommended that there is a significant potential in identifying the particle size, particle size distribution, contamination in the samples and pigmentation as demonstrated. The results suggest designing and developing a prototype with the aim to target only the polymer material. This could also have a significant impact on the polymer industry in terms of the capability of analysing and testing batch of samples instantaneously under operational time constraints. This will also be helpful in the quality control and validation of the raw materials with lower costs and greater accuracy.

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