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Research paper

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.

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

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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) Size Exclusion Chromatography (SEC) Fourier Transform Infrared (FT-IR) Scattering or Viscometry	Molecular weight & molecular mass distribution [2,3].	These techniques are highly complex, destructive in nature and require high level of expertise [2,3].
Melt Flow Analysis using melt indexing Rheometry analysis Melt Elongation analysis	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].
Thermogravimetric Analysis (TGA) Differential Scanning Calorimetry (DCS) Thermomechanical Analysis (TMA)	Phase transition, thermal degradation [3].	Destructive, laborious and require high level of expertise to operate the test equipment [3].
Mass Spectroscopy (MS) X-Ray Diffraction (XRD) NMR technique Transmission Optical Microscopy (TOM) X-Ray Photoelectron Spectroscopy (XPS)	Morphological characteristics [3].	These techniques are costly, time consuming, require laborious sample preparation and are destructive in nature [3].
Atomic Force Microscopy (AFM) Electron Microscopy (scanning, transmission)	Physical characteristics [2,3].	Laborious, time consuming, destructive and require time consuming sample preparation process [2,3].
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].

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.

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