A Novel Approach of Measuring the Dielectric Properties of PET Preforms for Stretch Blow Moulding

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ABSTRACT: Polyethylene terephthalate (PET) is the most common plastic material used in injection stretch blow molding (ISBM) process for the manufacturing of bottles. The injection-molded PET preform has to be preheated uniformly to a pliable state before the blowing stage of the process. Microwave technology offers a potentially more economical means for preheating the preform in place of the conventional infrared oven heating. For such applications, determination of dielectric properties of PET preform of a given geometry is essential. This article describes a novel approach of measuring the dielectric properties of PET preforms at microwave frequencies instead of the common experimental procedures used for such measurements. The dielectric properties are determined by using both CST Microwave Studio electromagnetic simulation software and the automatic network analyzer together. No complex mathematical solutions are required. Validation of this new approach has been made by comparing the measured dielectric properties of some polymers with those available in the published literature. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 3196–3203, 2008

Key words: polyethylene terephthalate (PET) preform; stretch blow molding; dielectric properties; electromagnetic simulation; waveguide

INTRODUCTION

The injection stretch blow molding process (ISBM) is a commonly used manufacturing process for the mass production of PET bottles in beverage industry. It is a two-stage blow molding process. In the first stage the preforms are injection-molded and stored, and then they are preheated before blowing in the second stage. An infrared oven is used for preheating a preform. The main limitations of this infrared heating are that it provides low energy efficiency for the polymer heating and the heating time is also high (of the order of 25 s in a typical process).

Microwave heating technology is seen as potentially more effective in such an application in terms of power consumption and process time. In microwave heating, dielectric characterization of PET preforms is an important first step to understand the material behavior subjected to microwave power. Microwave heating is different from conventional heating in the fact that the material dielectric properties dictate how much microwave energy is to be absorbed. Hence an understanding of the dielectric characterization expressed as the dielectric properties that consist of the dielectric constant and the loss factor, is crucial for any microwave processing. The dielectric constant and loss factor give core information about the heating rate, the penetration depth and the microwave absorption. The microwave heating technology can also assist in many other industrial processes involving a wide range of ceramic, polymeric, and composite materials.

The early research on dielectric properties measurements are very basic as illustrated by hand calculations performed by Von Hippel[1] or by the developed batch mode program described by Nelson et al.[2,3] In both instances, to obtain the dielectric properties, the solutions of a complex transcendental equation must be found. Von Hippel[1,4] measured the voltage standing wave ratio (VSWR) as the microwave physical response from the sample. The VSWR was then transformed into the reflection coefficient for the calculation of the dielectric properties through solving a complex variable transcendental equation. The method of dielectric constant measurement through a propagation constant also involves experimental data.[4]

In modern measurement methods, the network analyzer is used to measure the reflection coefficient directly and expresses it in terms of one of the four scattering parameters, S_{11}, which are commonly referred to as *S*-parameters. For a transmission measurement, the scattering parameter is S_{12}. The measurement methods involve mathematical calculations using *S*-parameters to determine the dielectric properties of a material.[5] Similarly, high frequency electromagnetic simulation softwares can provide the

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reflection parameter S_{11} or transmission parameter S_{12} of a material. Traveling waves are scattered or reflected when *n*-ports network is inserted into a transmission line. *S*-parameters offer great understanding for measurements and design problems.

This article presents a new technique of measuring dielectric properties of PET preform material involving electromagnetic simulation software and an automatic network analyzer. In this technique unlike the methods used by Von Hippel or Nelson et al., the PET preform is not required to be in a preferred geometry to determine the dielectric properties. Estimated initial values of PET preform dielectric properties are used to obtain a convergence to achieve the final accurate values. To validate results of our technique, we applied the same measuring technique to other known polymers and materials in rod and tube geometry as they are used in the industry, without having to manufacture them to a preferred geometry. This new method proposes an easy-to-use, effortless, and effective methodology for dielectric property measurement of the PET preforms applicable to blow molding industry.

OVERVIEW OF CURRENT TECHNIQUES

The measurement techniques for dielectric properties are numerous and chosen depending on the frequency of interest. At low frequency, a capacitor (parallel plates sample holder) or a cavity can be used. At high frequency, a section of a transmission line such as a coaxial cable or a rectangular waveguide can be used.[1–3,6] For low loss dielectric properties measurements, cavity resonators are used in the full mode or perturbation mode. All these techniques require that a sample must be manufactured from its virgin state to a preferred geometry. Accuracy in dielectric characterization is an important factor. Altschuler[6] has described this in full details and included a comprehensive review of the measuring principles. The classification of measurement techniques can be based on reflection or transmission measurements using resonant or nonresonant systems, with open or closed structures for the dielectric material to be measured.[7]

Many methods which involve network analyzers (NA) have been developed for measuring the dielectric constant and loss factor of a material. A network analyzer is an instrument used to analyze the properties of electrical networks especially those properties associated with the reflection and transmission of electrical signals known as scattering parameters (*S*-parameters). Network analyzers are operating in frequencies ranging from a few kHz to hundreds of GHz. Laverghetta[8] provides comprehensive information about application of network analyzers.

There has been several works done describing some of the automated dielectric measurement techniques with network analyzers.[9–11] Some of these are faster methods compared with the slotted line method.[1] In another development, the open ended coaxial probe method has been used with NA.[9] This method has been useful for fast broadband dielectric measurements for frequency ranging from 100 MHz to 20 GHz with an accuracy of 5% or less especially for liquid samples. Some of the advantages include examination of time, temperature, and frequency resonance effects. The disadvantages of the method are that it gives accurate result only for high loss materials and the sample must be homogeneous and optically flat, with a surface variation less than 25 μm. In this method, air gaps between the probe and the dielectric material can influence the accuracy of the measurement. However, the open shielded coaxial probe method is suitable for low loss materials. Bussey[10] has reported that outer dimension of the sample should be well over that of the inner conductor to achieve better results. Moreover, the sample is required to be homogeneous and prepared to a preferred geometry. Low loss and high loss material, however, can be measured with good accuracy for dielectric properties with the transmission/reflection method, that uses both ports of the network analyzer.[11,12] Sample to be measured has to be in certain geometry shape to use above technique.

METHODOLOGY OF THE PROPOSED MEASUREMENT TECHNIQUE

Dielectric properties are characterized by the relative complex permittivity of the material, ε, which is defined as the absolute permittivity divided by the permittivity of free Space $\varepsilon_0 = \left(36\pi.10^9\right)^{-1}$F/m.[13] The relative complex permittivity of the material can also be expressed as:

$$\varepsilon = \varepsilon' - j\varepsilon'' \tag{1}$$

where, the dielectric constant ε' indicates the ability to store electrical energy, and ε'' is called the loss factor and indicates the ability of the material to absorb electrical energy.

Another descriptive dielectric parameter is referred to as the loss tangent, tan δ, which indicates the material ability to convert electric energy into heat, and is shown by eq. (2). In this equation, δ is the loss angle.[13]

$$\tan\delta = \varepsilon''/\varepsilon' \tag{2}$$

The proposed technique is designed to perform accurate dielectric measurement of PET material using the high frequency electromagnetic simulator

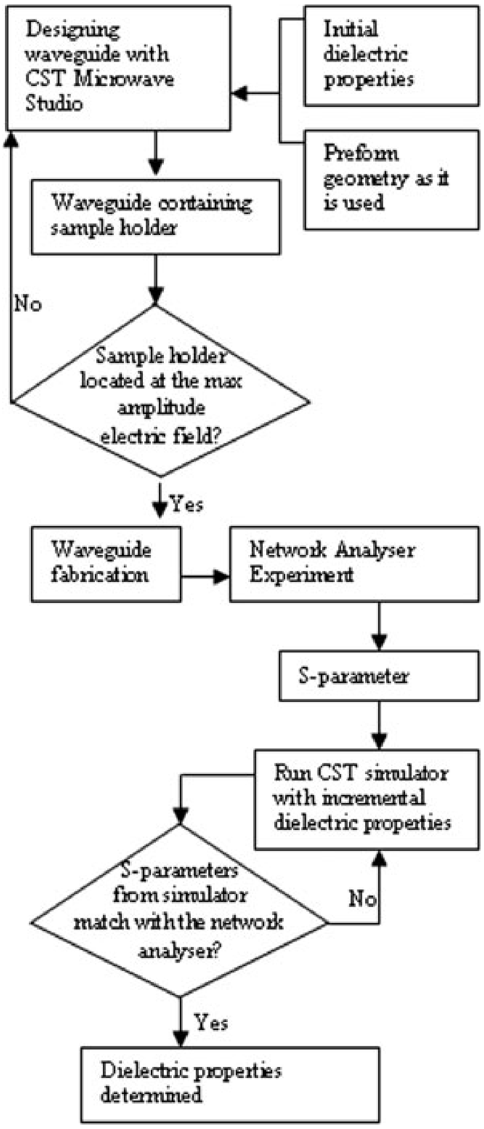

Figure 1 Dielectric measurement methodology.

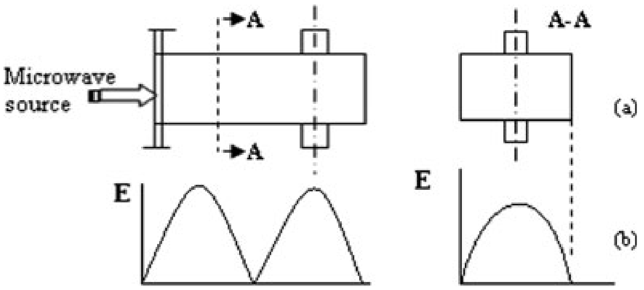

Figure 2 (a) TE waveguide cross section (b) E-field distributions.

and a network analyzer. The unique feature of this technique is that the PET preform with its existing geometry is directly used as the sample in the specially designed waveguide known as microwave applicator. This waveguide is designed to be used with a network analyzer. It can also be used with a high power microwave generator for microwave heating experiments as well. CST microwave studio simulation program is used for designing the waveguide. Figure 1 shows the basic steps of the methodology. In this technique, the geometric model of the preform and the initial estimated approximate values of dielectric property parameters are required as input for the simulation to determine the optimal location of the sample holder of preform in the waveguide. The designed waveguide with optimized preform location is then fabricated and used with the network analyzer to determine the *S*-parameter (S_{11}) experimentally at the given microwave frequency. The CST simulator is then run again using the final waveguide geometry and by varying the dielectric property values in incremental steps to get the required *S*-parameters. The *S*-parameter is observed from the simulator until it matches with the *S*-parameter obtained from the network analyzer. The main elements of the methodology are presented in detail in the following sections.

High frequency EM simulation software

There are several electromagnetic simulation softwares on the market for microwave power design. The CST Microwave Studio (MWS) is one of the high frequency 3D electromagnetic (EM) simulation

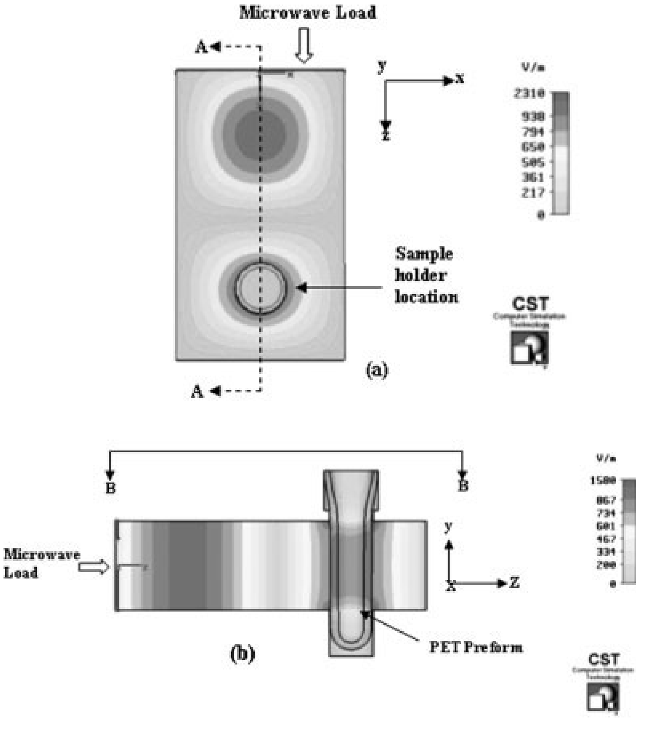

Figure 3 2D scalar propagation fields generated by EM simulator for the sample holder location (a) top view of waveguide, (b) side view cross section of waveguide.

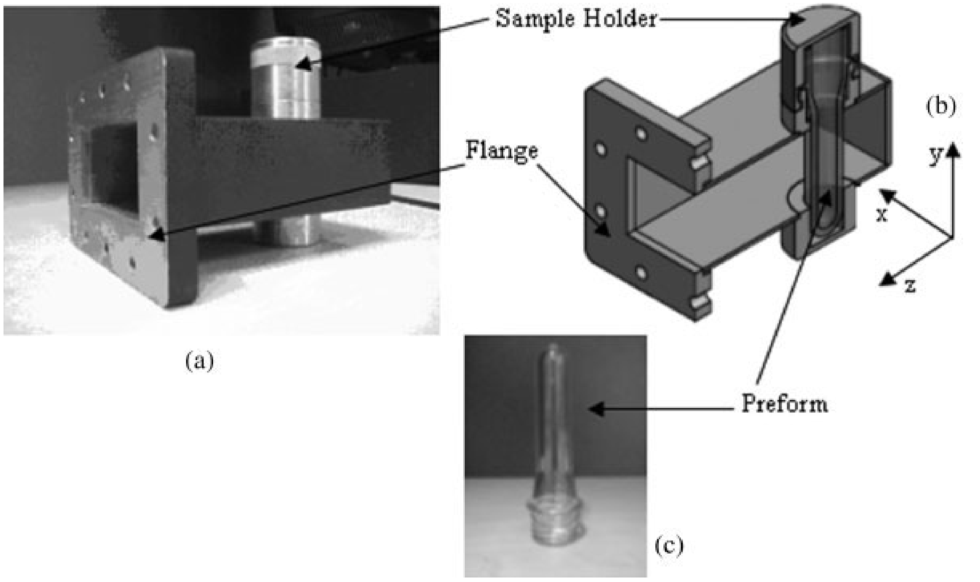

Figure 4 (a) Actual 150-mm long WR340 waveguide with a sample holder (b) CAD assembly of preform and WR340 waveguide cross section (c) 26.5 gr PET preform.

software using the finite-different time-domain (FDTD) technique. The Transient Solver module of the CST software was used for this work. This module is a general purpose module of the 3D EM simulator and is used for obtaining the broadband frequency domain results like *S*-parameters.[14,15] The applicator in the form of a rectangular waveguide was designed with an appropriate location of the sample holder for holding specific geometry of the preform. Designing the waveguide with the simulation software offers the benefits of savings of time and price for experiments.

PET preform model was first generated using the Pro/Engineer CAD program with actual dimensions and shape as used in the ISBM process. It was then converted into .stl extension file format and imported into the CST software.

In this procedure, the initial dielectric properties are required to design the waveguide containing the appropriate sample holder for the preform geometry. Transferred energy known as electromagnetic wave, establishes electric field within the waveguide. Standing wave pattern occurs by combination of the forward and reflected electromagnetic wave through short-circuit ended waveguide.

In designing the waveguide, appropriate location of the sample holder is crucial. It must be located at the point of maximum electrical field. Figure 2 shows a single mode waveguide and the electric field distribution. One end of the waveguide is connected to flange and on the other side is short-circuited. As shown in the Figure 2, sinusoidal electric field distribution occurs by energy transverse to the direction of propagation.

Knowledge of intensity and the gradient of electric field location are crucial to place the dielectric material (preform) at the location of the maximum electric field. The reason for that is because the loss factor, ε'', of PET is very small and the material will not absorb much power. Electric fields penetrate the dielectric material so that the power can be dissipated. Optimum electromagnetic energy transfer is needed to calculate reflection parameter as described above.

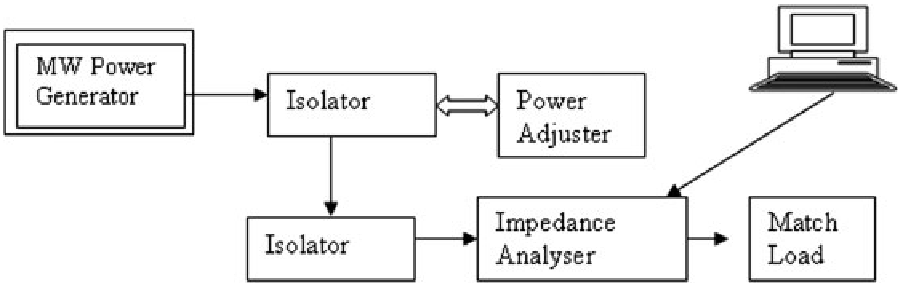

Figure 5 Match load calibration set-up with electric high power impedance analyser.

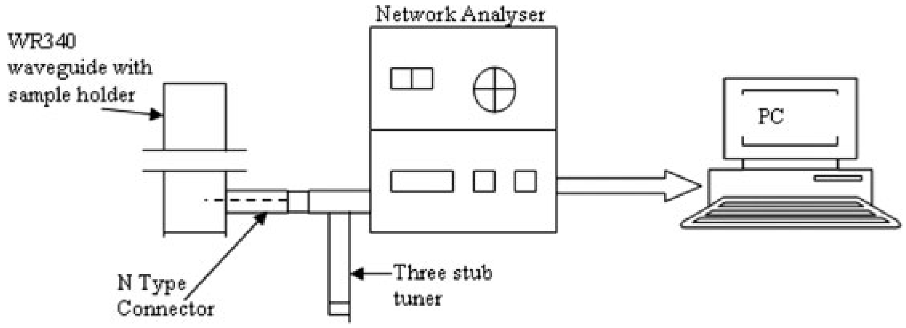

Figure 6 Experimental set up for network analyser measurement.

Figure 3 shows the electromagnetic simulation result with the sample holder location. Sample holder location has been determined according to electromagnetic field distribution result. Maximum electric filed location within the waveguide containing the preform was thus simulated without any mathematical modeling.

Figure 4(a) shows the actual fabricated waveguide with sample holder which are made of brass and Figure 4(b) shows the CAD model assembly of the waveguide. Figure 4(c) shows the PET preform used in the experiment.

Calibration of network analyzer

Calibration is necessary for a NA to get more accurate results. Unknown reflections in the coaxial line system cause systematic errors. These unknown reflection parameters can be solved by three known standards, such as short circuit, open circuit, and a match load as discussed by Somlo and Hunter.[16]

There are three standards required for the calibration to remove the system errors, which is represented by a four-element scattering matrix. These three standards are required when the systematic error matrix is assumed to be linear i.e., symmetrical. These are:

a. Short Circuit (S/C): Variable waveguide S/C is to be placed at (−1, 0) on the polar display.
b. Open Circuit: Variable waveguide S/C is shifted by 180° to give (1, 0) on the polar display.
c. Match load: Match load is located at the centre (0, 0) on the polar display. The accuracy is determined by how well a match load is made.

A match load is assumed to be (0, 0) so it must be as perfect as possible. Its response determines the error of the measurement. Therefore a commercially available match load must be tuned to have as good response as possible. Tuning was carried out using a stable microwave generator. Tuning set-up, shown in Figure 5, involves an isolator to prevent interaction between the microwave generator (2.45 GHz) and the match load, and a high power impedance analyzer. Screws on the match load were adjusted for tuning according to impedance analyzer output. The best result aimed for a match load achievable is 0.01.

Experimental set up for network analyzer measurement

Figure 6 shows the experimental set up used in the proposed method. The designed WR340 waveguide is connected to an HP8140 network analyzer. Special care is taken to keep the inside wall clean and connected surface of the connectors to achieve optimum result.[17] Coaxial connector was used to connect the WR340 waveguide adapter to HP 8410 network analyzer. Waveguide to coaxial cable adaptor is matched to the HP 8410 NA by three stub tuner.

The waveguide sample holder was connected to the network analyzer via a waveguide to coaxial adaptor. PET preform sample was placed in the waveguide sample holder and the experiment was carried out by running the NA at the set microwave frequency (2.45 GHz). This frequency is the most common microwave frequency used in industrial applications. The resulting scattering parameter (*S*-parameter) representing the reflection coefficient S_{11} was observed and noted at the PC both in terms of its magnitude and phase angle. These will later be compared with the *S*-parameter from the simulator

TABLE I
Reflection Parameter (S_{11}) Results from Network Analyzer at 2.45-GHz Frequency

Sample materials	S_{11} magnitude	S_{11} phase
Polyethylene terephthalate (PET) preform	0.9858	−138.80
Polypropylene (PP) rod	0.9881	−140.55
Polyvinylchloride (PVC) rod	0.9786	−161.48
Acetal rod	0.9281	−170.77
Polyamide Nylon 6 (PA-6) rod	0.9643	−173.53
Polyethylene terephthalate Polyester (PETP) rod	0.980	−171.62
Teflon rod	0.9989	−110.92

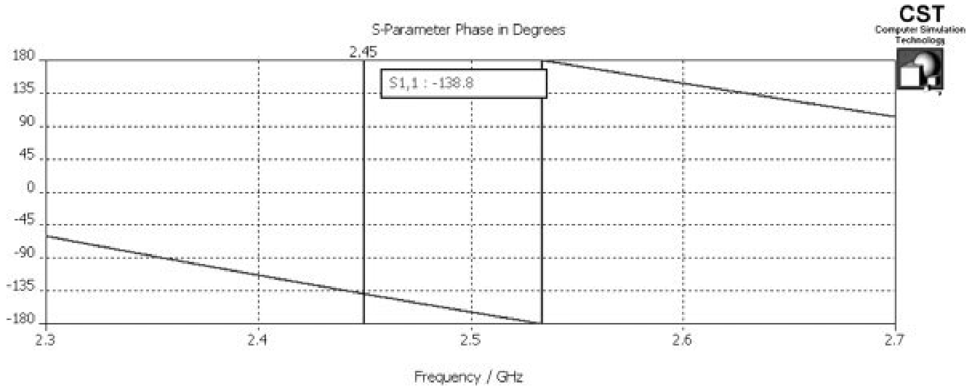

Figure 7 *S*-parameter phase at 2.45-GHz frequency for PET perform.

both in magnitude and the phase. Apart from PET preform, samples of six other polymer materials in rod form were obtained and the experiment was repeated to obtain the *S*-parameter values for these polymers at the same microwave frequency (2.45 GHz) to justify the validity of our approach as explained later. Table I shows the values of S_{11} parameter, both in magnitude and phase, which are obtained from these experimental measurements from the network analyzer at the frequency of 2.45 GHz for PET preform and othersix polymers.

RESULTS AND DISCUSSION

The measured *S*-parameter amplitude and phase from the network analyzer for the PET preform were used as a goal for optimizing and matching the simulated solutions from the CST Microwave Studio simulation software. Simulation software was run, each time entering different dielectric properties values (dielectric constant and loss tangent) incrementally as an input to provide *S*-parameter values in magnitude and phase until the *S*-parameter values obtained from network analyzer and the simulator are matched. Once this matching is achieved, then the last entered dielectric properties values give the final desired accurate values of the given preform material. Figures 7 and 8 show the results of *S*-parameter phase in degrees and *S*-parameter magnitude respectively, obtained from the final CST simulator run. It is noted that, for the PET preform used, these values are exactly the same as *S*-parameter from network analyzer (i.e., the value 0.9858 for magnitude and the value −138.8 for phase as shown in Table I for PET). Hence the actual dielectric properties for the PET preform obtained were 3.05 for the dielectric constant and 0.009 for the loss tangent. During simulation it was observed that the incremental variation in dielectric constant is more sensitive to *S*-parameter in phase and variation in loss tangent values is more sensitive to *S*-parameter in magnitude.

CST simulation process was repeated for six other polymers used during network analyser experiments and the final dielectric constant and loss tangent values were obtained for these polymers after the respective simulations run. Table II shows the results of the dielectric properties (dielectric constant and loss tangent) for the PET preform and six other polymers obtained from simulator using the *S*-parameter matching technique. Densities of the materials used are taken from literature. Table II provides a unique piece of information on dielectric properties of different polymers at common microwave frequency of 2.45 GHz for industrial applications. This technique

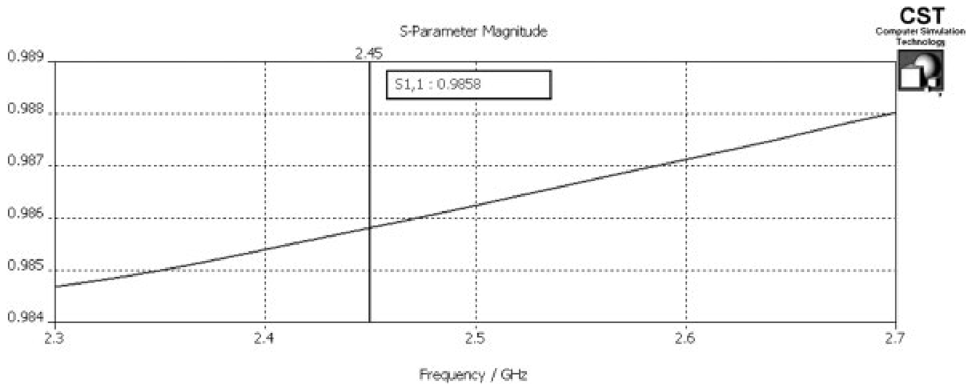

Figure 8 Magnitude of *S*-parameter at 2.45 GHz for PET preform.

TABLE II
Dielectric Properties Results with EM Simulator Using *S*-Parameter from NA at 2.45 GHz

Materials	Density (g/cm^3)	Dielectric constant	Loss tangent
Polyethylene terephthalate (PET) perform	1.3–1.4	3.05	0.009
Polypropylene (PP) rod	0.9	2.43	0.066
Polyvinylchloride (PVC) rod	1.4	2.88	0.012
Acetal rod	1.42	3.14	0.042
Polyamide Nylon 6 (PA-6) rod	1.13	3.23	0.02
Polyethylene terephthalate Polyester (PETP) rod	1.3–1.4	2.98	0.01
Teflon rod	2.26	2.2	0.0003

of using simulation software eliminates the necessity of all the approximate mathematics and it provides a means of measuring the dielectric properties of a material in the shape as they are used ('as is') in industrial applications.

To verify the accuracy of our results, comparisons were made for the dielectric properties values obtained by our method with the dielectric properties obtained by conventional techniques for certain selected polymers available in the published literature. Table III shows the values of the dielectric properties of three selected polymeric materials (PVC, Nylon 66, Teflon) at the same or nearly same frequency levels obtained by conventional techniques, which are available in published literature.[1,13,18]

Figures 9 and 10 shows the graphical comparison of dielectric constant and loss tangent values respectively, for various polymers obtained from the new approach with the values of the three polymers for which data are available. Figure 9 shows that dielectric constants of the three polymers are found to match closely with those obtained from the new approach, thus indicating the validation and soundness of our approach.

However Figure 10 shows that lost tangent values are not as close as those of published data for PVC and Nylon 66. This may be due to very small reflection coefficient obtained for those polymers. This problem can be overcome by using resonant cavities instead of nonresonant waveguide, which makes such materials absorb optimum electromagnetic power. Therefore, the accuracy of loss tangent measurement would increase.

TABLE III
Dielectric Properties of Same Polymers from Literature for Comparison[1,13,18]

Materials	Frequency (GHz)	Dielectric constant	Loss tangent
Polyvinylchloride (PVC)	2.5	2.85	0.0056
Nylon 66	3.0	3.02	0.0135
Teflon rod	3.0	2.04–2.08	0.00014

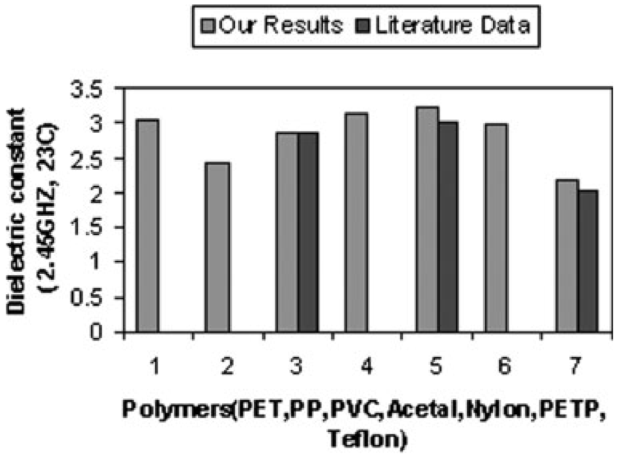

Figure 9 Comparison of dielectric constant values of polymers.

Further study can be carried out for this new dielectric measurement approach using different samples of complex shapes and with various cross section shapes. One of the main advantages of this technique is that the samples with various features and cross section shapes can be used in the waveguide. However careful investigation is necessary for sharp corner featured shapes for accurate results. No other method is normally able to measure the dielectric properties of material with such complex shapes. Research work is continuing at Swinburne to apply the new technique and use the same waveguide for use in microwave heating of preform for blow molding applications and the experimental results are expected in a future publication.

CONCLUSIONS

A new methodology of dielectric characterization of polymeric materials for microwave heating technology has been proposed. The technique has been

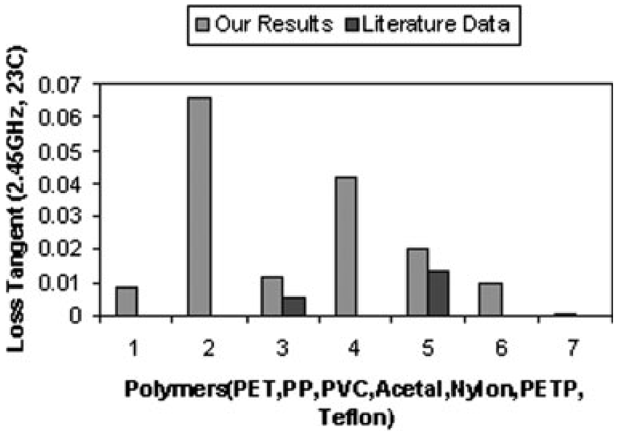

Figure 10 Comparison of lost tangent values of the polymers.

developed for determining the dielectric properties for PET preforms for preheating in blow molding process for bottle manufacturing. The technique can be applied to any form of polymeric material or any form of materials in general. The results obtained using the methodology is found to be in good agreement with published results. The new methodology has three major advantages: (a) the material can be used ''as is'' in any geometry, (b) any type of polymer can be tried, (c) no mathematical calculations are necessary. The method shows that the sample holder used for the characterization of the dielectric characterization of PET preforms can also be used for microwave preheating. It will enable the microwave power technology to be successfully used in preheating of preform in ISBM process.

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