Effect of Nano-Susceptor Material Addition on the Microwave Sintering of Polypropylene

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Abstract

The use of microwave heating technology for processing of polypropylene was investigated with key research focus on the use of susceptor materials utilized and on the pre-mixing technique employed before microwave processing. Two susceptor materials (carbon black and polyaniline) were investigated for this purpose. Fourier transform infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA), density measurement, hardness testing, scanning electron microscopy (SEM) and optical microscopy were employed to study the efficiency of each susceptor material and to characterize the properties of the composites formed after microwave processing. The results indicated a higher efficiency for carbon black susceptor material as compared to polyaniline and manifested the enhancement in the properties and characteristics of the composites by using a low viscosity premixing technique.

Key Words: *microwave, sintering, susceptor, dielectric, composites, processing*

1. Introduction

Microwaves are extensively used in the processing of foods, ceramics and polymers. Polymerization, vulcanization and joining of plastics parts transpire by using microwaves. The frequency of microwaves start from 300 MHz and goes up to 300 GHz [1]. Microwave ovens having fixed frequencies of 915 MHz, 2.45 GHz, 5.8 MHz, or 24.125 GHz were commonly used in past. But now, microwave ovens operating at variable frequency are effortlessly available [2]. Microwave processing mainly depends on the dielectric properties of materials. Processing of every material is not practicable using microwaves. Presence of polar groups is necessary to process materials using microwaves. In polar materials, heat is generated from high vibrational motion of polar molecules due to absorbance of microwaves [1,2].

When subjected to microwaves materials may behave as transparent, reflectors and absorbers. Microwaves can easily pass through transparent materials without physically affecting them. Polymers, glass, and ceramics are common examples. From reflectors, incident waves reflect without making any change in material's characteristics, this behavior is manifested by most of the metals. Materials which absorb electromagnetic waves are termed as absorbers. Microwave processing of transparent and reflector material is practicable by some modifications in material. Some microwave sensitive sites can be

easily generated by using 'susceptor', to make them responsive toward microwaves. By using appropriate susceptor, microwave processing of ceramic and metals have been achieved successfully [1].

Most polymers such as polyethylene, polypropylene [3] and ABS [4] etc. are transparent to microwaves. So, their microwaves processing is not straight forward. The response of materials toward electromagnetic fields is dominantly based upon two fundamental properties of materials that are the dielectric constant (ε) and dielectric loss (ε"). The permittivity or dielectric constant (ε') determines the electric energy hoarding ability of a material, while the dielectric loss (ε) ^{*}) determines the ability of the material to transform the electric energy into heat [1]. Susceptors are microwave sensitive materials and can be used to process transparent materials. Microwaves have been used for the sintering process in the field of metals and ceramics [5]. Sintering is chiefly a process of densification of powdered ceramic, metal or other material by heating [6].

The heat treatment of most polymers for various applications is somehow obligatory as polymeric materials have many potential applications from household items to hi-tech aerospace industry. Polymers are light weight and easily process able with noticeably low energy consumption as compared to metals. Presently, these materials are being processed by injection molding, compression molding, extrusion molding, calendaring etc. In all these processing techniques, an extruder is used to melt the solid polymers. The two crucial limitations in processing polymers in extruder are non-uniform heating and non-uniform shearing which results in different kind of failures in the end product. On the basis of origin, waxes can be divided into two types natural and synthetic[7]. Synthetic waxes are further divided into types, like non-polar polyethylene waxes, polar polyethylene waxes, polar polypropylene waxes, non-polar polypropylene waxes, grafted PO waxes and copolymers. Waxes are used to manufacture simple candle and as additives for inks. Waxes have application in hot melt adhesives, coatings, and in investment casting [8,9].

Waxes are commonly used as phase changing materials (PCMs). But the absorbance of heat in PCMs results in undesired loss of mechanical properties because the increase in heat absorbing capacity of PCMs unfortunately results in the loss of mechanical properties. Ideally PCMs should absorb maximum heat without significant loss in mechanical strength. Waxes are common phase changing materials as they have high potential to store heat energy e.g. paraffin wax [10]. These waxes are generally low molecular weight and have low melting points and high flow rates in molten form. The softening point of waxes ranges from 30° C to 130 °C [11]. In order to overcome the loss of mechanical properties, it is desirable to blend waxes with polymers that have superior mechanical properties. Much work has been reported in literature where significant improvements in mechanical properties were made by blending waxes with LDPE, HDPE and PP [3,10,12]. Many research papers have been published in recent years on the addition of nanofillers in polymer matrix [13,14,15,16,17,18].

The performance of blends mainly depends upon the quality of mixing. The most commonly used method is hot melt mixing. But the fact to be noticed is that hot melt mixing is a time taking process when compared with time efficient and energy efficient microwave processing. Novelty of work can be expressed in terms of pre-mixing methodology adopted where susceptor was first coated on substrate surface by sonication followed by solution blending. After this microwave were used to melt susceptor coated polypropylene substrate. Moreover, utilization of polyaniline as a susceptor has been rarely reported in literature. Thus, new pre-blending technique and using polyaniline as a susceptor in comparison with carbon black are novel aspects of this study. In literature microwave technology, has been widely used for welding of polymers and fastest joining have been achieved even in 2 seconds [19]. In this study, microwaves have been utilized to melt and process PP, for its application in blending with waxes. Pre-melting of PP based composites can shorten the processing time. In this article, carbon black and polyaniline are used as susceptor materials to process polypropylene through microwave oven.

2. Experimentation

2.1 Materials

Ammonium persulphate (APS, reagent grade) purchased from *SIGMA-ALDRICH Chemicals* was used as oxidizing agent in preparation of polyaniline. Hydrochloric acid (HCl, reagent grade) and aniline (reagent grade) purchased from *DAEJUNG Chemicals* were used as received for the synthesis of polyaniline (PANI) Polypropylene *Homopolymer, Repol, Reliance Industries Limited, India* was purchased in the form of pellets. Cabot Carbon Black N-220 was also used as susceptor material after grinding with mortar and pestle. Reagent grade toluene from *DAEJUNG Chemicals* was used as solvent for density measurements.

Fig. 1: PP-powder preparation using liquid nitrogen

2.2 Preparation of Polyaniline

Polymerization of aniline using APS has been successively reported in literature from last few years because of production of PANI in noteworthy bulk amount using ammonium persulphate [20]. Ammonium persulphate (APS) as an oxidizing agent was used for the preparation of polyaniline. 1000mL hydrochloric acid solution was prepared by dissolving 83.3mL (1 molar) HCl in distilled water. HCl solution was then distinctly poured into two beakers, each of 500mL. 60.53 grams (0.65 molar) of aniline was then added in 500mL solution of HCl and 136.9 grams (0.6 molar) of ammonium persulphate (APS) was added in the remaining 500mL HCl solution. APS solution was added drop wise in aniline solution

with continuous stirring at $0^{\circ}C - 5^{\circ}C$ and the mixture was stirred for 3 hours using magnetic stirrer. The solution was filtered and then washed with distilled water. The moisture content was utterly evaporated by heating washed sample at 90° C in drying oven.

2.3 Preparation of PP-powder

In order to facilitate the premixing process, polypropylene pellets were grinded to powder by cryofracture grinding technique. Cooling and freezing of PP pellets were accomplished using liquid nitrogen. Pellets after freezing were grinded to prepare powder using 3 phase (2 hp) grinder. The grinded pellets were then sieved through 40 mesh size sieves to separate the particles of 420 microns (0.420 mm) sizes. Remaining large sized particles were treated again with liquid nitrogen and grinded to reduce particles up to desired size. A scheme of the process utilized for the preparation of PP powder is shown in Fig. 1.

2.4 Preparation of PP-composites

Carbon black as susceptor material was added in polypropylene to introduce the dipole sites (heat sensitive sites) in polypropylene. Carbon black of required concentration was first dispersed in definite proportion of distilled water using sonicator. Sample was sonicated for 30 minutes. On the other side, PP powder of 0.420 mm particle size was well dispersed in water in the presence of heat using magnetic stirrer. Heat was provided for proper dispersion of PP powder as the density of polypropylene at room temperature is noticeably lower than that of distilled water. Dispersed samples of both carbon black and PP were thoroughly mixed, and mixture was heated constantly to accomplish evaporation. The sample was then proliferated on tray and moisture content was allowed to evaporate in the drying oven. The overall process is summarized in Fig. 2. Same procedure was adopted to prepare PANI-PP composites through sonication technique.

2.5 Microwave sintering of PPcomposites

Dawlance Microwave Oven with frequency of 2.54 GHz was adequate according to our requirements and the higher heating rates required for the microwave sintering of polypropylene composites were achieved using it. Polypropylene filled polyaniline composite sample PP-PANI (90:10) and various polypropylene filled carbon black composite samples i.e. PP-CB (85:15), PP-CB (90:10), PP-CB (95:5), PP-CB (97:3), and PP- CB (99:1) were examined for their melting time and temperature under the application of electromagnetic waves. Electromagnetic waves were absorbed by the heat sensitive sites created by the susceptor material in the substrate polymer. Energy was transferred electromagnetically which resulted in an increase in temperature of PPcomposite samples. Both exterior and interior homogeneous surface heating was achieved using microwave sintering and heat was dissipated as a whole in composite sample.

3. Characterization

3.1 Fourier Transformation Infrared Spectroscopy (FTIR)

Analysis was performed using JASCO FT/IR-4100 analyzer in ATR mode by preparing the samples in crystalline form and the spectra were recorded. FTIR spectra of polypropylene filled polyaniline and polypropylene filled carbon black composites at various concentrations were studied.

3.2 Thermogravimetric Analysis (TGA)

TGA analysis was accomplished using SHIMADZU TGA-50 Thermogravimetric Analyzer in nitrogen atmosphere at the flow rate of 30 mL/min. 4-5 grams of polypropylene filled carbon black and polyaniline composite samples were placed in the aluminum pan within temperature range of 30° C -500 $^{\circ}$ C at the rate of 10° C/min.

3.3 Scanning Electron Microscopy (SEM)

SEM was performed using JEOL USA (JSM 5910) scanning electron microscope. Morphological attributes of pure polyaniline, polypropylene filled carbon black and polypropylene filled polyaniline composites were examined. Samples were attached onto the aluminum stage using double sided carbon tapes and the analysis was carried out at an accelerating voltage of 15 kV. Micrographs for various samples at specific resolutions were observed.

3.4 Optical Microscopy (OM)

The morphological features of pure PANI, PP-PANI composites, and PP-CB composites of various compositions were observed using Olympus STM-6 measuring microscope (Olympus Corporation, Tokyo, Japan). Composite samples were microwaved and compression molded prior

to optical microscopy and digital impressions were obtained to observe the dispersion of susceptor material in base polymer. Micrographs obtained by optical microscopy manifest some large and small sized particles ranging in various micrometers.

3.5 Density measurements

The precise density measurements for PP-CB and PP-PANI were accomplished using KERN ARS 120-4 density meter. Specific gravity method for density measurements was utilized to calculate the density values of specimen. Toluene having a density of 0.864 g/cm³ was used as a reference solvent. Masses of samples in both air and solvent were observed and the calculated values were simply added in equation 1 mentioned below for accurate density measurement.

Density
\n
$$
= \frac{Mass \text{ in air}}{Mass \text{ in air} - Mass \text{ in solvent}}
$$
\n
$$
\times \text{ Density of solvent}
$$
\n(1)

3.6 Hardness measurements

The specimen's ability to resist permanent indentation was determined using GIBITRE Electronic Durometer instrument using shore D at room temperature.

Fig. 2: Coating of susceptor material on polypropylene

4. Results and Discussion

4.1 Morphology of PANI

SEM analysis was performed to observe the morphological attributes of polyaniline. Granular morphology is ordinarily observed when polyaniline in the form of powder is synthesized under highly acidic conditions [21]. SEM micrograph in Fig. 3 (a) revealed aggregated structure of PANI synthesized using ammonium persulphate (APS). The micrographs although manifested the reasonably smaller sizes of PANI particles which are appropriate for dispersion in base polymer but discrimination of individual particles of PANI is arduous as the adhesion of particles can clearly be observed throughout the micrograph.

4.2 Morphology of PP-composites

Granular morphological attributes of polypropylene coated with polyaniline and carbon black individually were analyzed through scanning electron microscopy. Fig. 3(b) shows SEM micrograph of polypropylene coated with PANI and Fig. 3(c) shows SEM micrograph of polypropylene coated with carbon black. Proper dispersion of PANI and carbon black nanoparticles can be observed in SEM micrographs. The adequate particle sizes as observed in micrographs resulted in highly appropriate dispersion of susceptor materials in substrate polymer. Particle adhesion was also observed due to complete dispersion of polyaniline and carbon black in polypropylene. Well dispersed susceptor material can result in the appropriate generation of heat sensitive sites in the transparent substrate polymer.

4.3 Optical Microscopy

Optical micrograph of PANI-PP (90:10) composite is shown in Fig. 4(a) and micrograph of CB-PP (90:10) composite is shown in Fig. 4(b). Optical micrographs manifest the availability of susceptor material over the entire surface of the compression molded composite sample which ensures the availability of heat sensitive sites in the composite. A direct comparison between the two micrographs reveals larger particle size of the PANI in comparison to CB particles at same weight percentage in PP. Such morphology of susceptor materials in the form of dispersed particles ranging in various micrometers can lead to different heating rate of the composite when exposed to microwaves.

4.4 Microwave Sintering

Microwave sintering of PP composites was performed in the microwave oven and after exposure to the microwaves the average surface temperature of the composites was recorded based on four different location temperature readings. The average temperature achieved and the total melting time in microwave exposure for different susceptor (CB) concentration is shown in Fig. 5. The physical appearance of PP-CB (99:01) after 10 minutes, PP-CB (97:03) after 07 minutes, PP-CB (95:05) after 4 minutes and PP-CB (90: 10) after 02 minutes of microwave sintering is shown in Fig. 6.

Fig. 4: OM Micrograph of a. PANI coated PP (90:10), b. carbon black coated PP (90:10)

The highest average temperature was observed for PP-CB (85:15) which was about 225.75°C with melting time of 1 minute. The reason for achieving high temperature is the higher amount of susceptor material in polypropylene. Melting started at inceptive stages of sintering but also resulted in thermal degradation of PP-CB (85:15) sample. The most effective PP:CB ratio was observed to be PP-CB(90:10) which achieved the temperature of 206.50°C (temperature 20-25°C above the melting point of PP) in 2 minutes without any notable degradation of sample at higher temperatures. The low melting time without any degradation of sample indicates that microwave sintering is comparatively time and energy efficient process than that of tardy conventional polymer processing techniques [19].

Fig. 5 shows higher values of temperatures and low melting time for higher amounts of susceptor material due to increase in the dipole sites introduced in the substrate polymer. Rotation of introduced dipole sites resulted in the increase in temperature from center to surface as heating of sample was accomplished [22]. In order to compare the effectiveness of CB as a susceptor material, PP-PANI (90:10) sample was also microwave sintered. The results obtained for PANI based composite were 186 average temperature achieved for 8 minutes of melting time in microwave. The microwave sintering data comparison for PP-CB (90:10) and PP-PANI (90:10) indicates the effectiveness of utilizing CB as a susceptor material because of its better dispersion and distribution on the surface of PP as revealed by SEM results in Fig. 3.

4.5 FTIR analysis

ATR analysis of unfilled and filled polypropylene composites was carried out to study the interactions between polypropylene and the susceptor carbon black. ATR spectra of filled and unfilled specimen are shown in Fig. 7. Characteristic absorption peaks of polypropylene were observed near 2917 , 1454 and 1374 cm⁻¹ that represents C-H stretch, $CH₂$ and $CH₃$ deformation respectively while the band near 1033 cm⁻¹ represents isotactic polypropylene bend. When

ATR spectrum of carbon black filled polypropylene composites were analyzed small peaks near 1740, 1580 were observed that were absent in ATR spectra of unfilled polypropylene specimen. This indicates the deposition of carbon black on polypropylene powder. Variation in intensities of polypropylene and carbon black peaks indicates the interference of susceptor (carbon black) with polypropylene and slight degradation of polypropylene when exposed to microwaves. Further no new peaks were observed in spectra of filled composites which suggest the absence of any chemical networking between susceptor and polypropylene matrix. However physical networking is evident from the spectra given in Fig. 7.

PP-CB (99:01)-10 min

PP-CB (97:03)-07 min

PP-CB (95:05)-04 min

PP-CB (90:10)-02 min

Fig. 6: Physical appearance of (a) PP-CB (99:01) after 10 minutes, (b) PP-CB (97:03) after 07 minutes, (c) PP-CB (95:05) after 4 minutes and (d) PP-CB (90: 10) after 02 minutes of microwave sintering

4.6 TGA analysis

Susceptor materials such as carbon black and polyaniline have noticeably significant effects on the thermal stability of substrates as the increase in thermal stability of substrate polymers is reported in literature due to increase in ratio of carbon black suspector. Unfilled composites degrade at relatively lower temperatures than that of susceptor filled composites. [23]. It was observed that small mass loss of PP-CB (90:10) w/w % composite started at 280° C and then the

sample began to degrade thermally. This initial weight loss could be due to loss of moisture or other volatiles present in sample. The onset and endset values for thermal degradation of PP-CB (90:10) sample are 360.14° C and 452.83° C respectively with total mass loss of 92.63%.

Degradation of PP-PANI (90:10) w/w % composite sample was started at comparatively higher temperatures than PP filled with carbon black. Onset and endset points are 383.76°C and 459.16° C respectively. Higher weight loss of 98.21% was achieved in comparison to PP-CB(90:10). It was concluded that the composite samples of PP-CB composite and PP-PANI composite were thermally stable at higher temperature of about 300° C without showing any thermal degradation. The thermal stability of PP-PANI (90:10) was observed to be relativity higher than PP-CB (90:10) as the thermal degradation of PP-PANI (90:10) was carried out at higher temperatures.

Fig. 7: FTIR spectra of PP and PP-CB composites.

4.7 Density analysis

Density measurements are crucial for evaluation of strength-weight ratio of material. Density of material has noteworthy effects on the crystallinity of material and also the physical changes in the material are related to density therefore the precisely measured density values are significant. Density measurement using specific gravity method was carried out for various polypropylene samples coated with susceptor material. Toluene as a solvent was used as a reference fluid. Fig. 9 represents the density values of filled and unfilled samples at various susceptor loadings. The highest density value of 0.954 $g/cm³$ was measured for PP-CB (85:15) and the lowest

value of 0.902 $g/cm³$ for virgin polypropylene was observed because of absence of susceptor material. Density values of pure PP usually ranges from 0.91-0.94 g/cm³ [24]. In case of PP-PANI (90:10) specimens the shore D hardness value of 83.24 was observed. It is fascinating to notice that all the measured density values of pure and composite samples lie within the density range of pure PP which manifests great properties due to its low density.

Fig. 8: (a) TGA thermograms for PP-CB (90:10), (b) TGA thermograms for PP-PANI (90:10) composites

4.8 Hardness analysis

Shore D hardness of microwaved and compression molded composites was measured. In case of PP:CB specimens an increasing trend was observed as concentration of susceptor was increased. Fig. 9 represents the hardness values of filled and unfilled CB samples at various susceptor loadings. Samples were observed to be reasonably hard and were flexible up to some extent. The higher values of hardness at high susceptor loading leads to brittleness of material so adequate values of hardness are required to avoid brittleness. Hardness values for pure PP sample were observed to be 80.51shore D and the highest hardness of 85.68 shore D was observed for PP-CB (85:15). In case of PP-PANI (90:10) specimens the shore D hardness value of 83.24 was observed. All other samples represented relatively similar hardness values. The reasonable hardness of PP-CB and PP-PANI composites indicated the dimensional stability of materials under loading.

Fig. 9: Density (g/cm^3) and Hardness (Shore D) of PP-CB composite specimens

5. Conclusion

Carbon black and polyaniline behaved as susceptor elements as revealed by thermal and microwave sintering analysis. SEM micrographs showed appropriate dispersion of susceptor in polymeric substrate. FTIR analysis also supports SEM analysis as physical networking between polypropylene and carbon black was evident. Hardness was increased at high susceptor loading. Thermal analysis revealed specimen were stable up to 300° C. Hence the premixing techniques employed were successful in deposition of susceptors on polypropylene substrate.

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