

# Interpenetrating Polymerization of Styrene in Polypropylene and Poly(Ethylene Terephthalate) Using Microwave Irradiation

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## Abstract

The preparation of interpenetrating polymer is one of the most efficient methods for modifying or improving polymer properties. In this work, interpenetrating polymerization of styrene in polypropylene (PP) and poly(ethylene terephthalate) (PET) using microwave irradiation was investigated. The immobilization percentage of polystyrene as a function of the initiator/monomer ratio, reaction time, and microwave power was studied. Immobilization percentages up to 30 and 16 % in PP and PET, respectively, were achieved in less than 20 min of microwave time. The interpenetrating polymers showed superior chemical stability in both acidic and basic media for several days over their pristine counterparts while their thermal and mechanical properties were comparable, indicating that the new materials reported in this research can be used as substitutes for various applications. Furthermore, the microwave method developed in this work provides efficient, versatile, and simple method for the preparation of chemically stable interpenetrating polymers, and can be used to modify the structure and properties of polymeric substrates.

**Keywords:** interpenetrating polymer; poly(ethylene terephthalate); polypropylene; polystyrene; microwave irradiation

## 1. Introduction

Polymer hybrids consisting of two or more polymers have emerged as multi-functional materials that can be used in applications such as compatibilizers, templates, etc. Polymer hybrids can be produced by a number of methods, including preparation of polymer blends (Lloyd, 2007; Herranz, *et al.*, 2018) and

interpenetrating network (IPN) polymers (Shivashankar, *et al.*, 2012; Gupta, *et al.*, 1994; Obiweluzor, *et al.*, 2018), and surface grafting (Memetea, *et al.*, 1979; Chen, *et al.*, 1998; Hochart, *et al.*, 2003; Sun, *et al.*, 2018). Polymer blends are a physical mixture of two (or more) polymers. Although easy to prepare, blending of some pairs of polymers may lead to phase

separation, due to the incompatibility of the pairs. This produces undesired properties and the deterioration of the blended materials (Zhang, *et al.*, 2013). Graft copolymers consist of main chains of one monomer type and branches of the others (Bhattacharya, *et al.*, 2004). However, preparation of graft copolymers involves modification of the polymer structure via grafting reactions. Grafting of inert polymers can be difficult and may destroy the polymer structures when harsh conditions are employed. Another interesting method of creating polymeric hybrid materials is the preparation of IPN polymer. IPN is a hybrid material comprising two or more polymer networks that are partially or totally intertangled, but not bonded to each other. In contrast with grafting techniques, the chemical structures of IPN polymers are not modified, as the polymers are not chemically bonded. Compared with polymer blends, IPN polymers are much more stable and will not phase separate, because the polymer chains are intertangled (Madhumitha, *et al.*, 2012).

Free radical polymerization is generally utilized to prepare IPN polymers, typically requiring high temperatures and long reaction times. In previous studies, preparation of IPN polymers was mostly performed via photo-initiated polymerization or the use of a normal heating source. The use of microwave irradiation is rare (Cheng, *et al.*, 2013; Meena, *et al.*, 2014; Bajpai, *et al.*, 2015; Kamboj, *et al.*, 2015; Tally, *et al.*, 2015). In this study, we investigated the use of microwave irradiation for interpenetrating polymerization. The microwave method is fast

and supplies high constant thermal energy. Microwave irradiation has been used in step-growth polymerization, ring-opening polymerization, and radical polymerization. Pielichowski *et al.* used microwave irradiation to synthesize poly(aspartic acid) from maleic anhydride and increased the reaction rate increased by a factor of ten without affecting the yield (Polaczek, *et al.*, 2005). Faghihi and Hagibeygi used a domestic microwave oven to synthesize polyamides containing azo-benzene moieties with higher yields and faster reaction time than the conventional methods (Faghihi, *et al.*, 2003).

In this research, the microwave method was used to achieve interpenetrating polymerization of styrene in polypropylene (PP) and poly(ethylene terephthalate) (PET). PP and PET were chosen as the polymeric substrates as they are low-cost and non-toxic thermoplastic polymers that are flexible and resilient. They are widely used to fabricate commodity products including plastic bags, containers, and bottles (Paszun, *et al.*, 1997). They have also been used in a variety of advanced technological applications ranging from electronics (Hota, *et al.*, 2012; Konieczna, *et al.*, 2010; Li, *et al.*, 2005) to biological devices (Dhahri, *et al.*, 2011; švorčík, *et al.*, 2006). Various IPN polymers have been combined PET with polyacrylamide (Rudenja, *et al.*, 2010), polyamides (Liu, *et al.*, 2010), poly(2-hydroxyethyl methacrylate) (Gloria, *et al.*, 2011), poly(acrylamide-co-ethylene glycol) (Park, *et al.*, 2000), N-halamine (Zhao, *et al.*, 2011b), butynyl monomer (Li, *et al.*, 2012), and oils (Barrett, *et al.*, 1993a; Barrett, *et al.*, 1994;

Barrett, *et al.*, 1993b). PP has been combined with N-isopropyl acrylamide (NIPAAm) (Muñoz-Muñoz, *et al.*, 2012), acrylic acid (AAc) (Muñoz-Muñoz, *et al.*, 2012), polyacrylamide (PAM) (Zhao, *et al.*, 2011a), and styrene (St) (Tsai, *et al.*, 2011; Li, *et al.*, 2002). Nonetheless, to the best of our knowledge the microwave method has not previously been used to combine styrene with PP or PET. The immobilization efficiency of styrene as a function of initiator/monomer ratio, reaction temperature, and reaction time was studied. The molecular weights of free polymers in the solution were measured. The thermal and mechanical properties of the samples were determined. This study demonstrated a fast, simple interpenetrating polymerization method using microwaves to create new materials based on PS, PP, and PET. These new materials will be attractive in a range of applications.

## 2. Materials and Methods

### 2.1 Materials

All chemicals were purchased from Sigma-Aldrich unless otherwise noted. Styrene

was purified by passing through silica gel columns prior to use. Benzoyl peroxide (BPO) (75 %, Acros) was recrystallized using methanol. Hexane, acetone, dichloromethane, diethyl ether (Labscan), xylene, and trifluoroacetic acid were used as received. Deionized (DI) water was used throughout the experiments.

### 2.2 Substrate preparation

PET and PP samples (0.75 x 0.75 cm<sup>2</sup> and 0.1 mm thick) were cleaned in hexane, acetone, and DI water using an ultrasonic bath (10 min) and dried in an oven at 70 °C overnight.

### 2.3 Interpenetrating polymerization by microwave irradiation

The polymer samples were immersed in a solution of styrene and BPO and then placed in a domestic microwave oven (Electrolux EMS2327S) for a specified time and microwave power. The samples were then cleaned by ultrasonication in dichloromethane (4 x 10 mL, 15 min) and dried overnight at 70 °C. The immobilization percentage was defined as the percentage by weight of polystyrene to the polymer substrate, which was calculated using equation (1).

$$\text{Immobilization percentage} = [(w_2 - w_1) \div w_1] \times 100 \quad (1)$$

where w1 and w2 are the weights of sample before and after polymerization, respectively. The immobilization percentage was averaged from at least five samples in each condition.

### 2.4 Extraction of polystyrene from interpenetrating polymers

The interpenetrating PS was extracted using a trifluoroacetic acid solution in dichloromethane (5 % v/v) for the PET and xylene for the PP. All samples were dissolved in the solution and stirred at room temperature for 20 min. The mixtures were then precipitated in methanol and filtered. The powder was

ultrasonicated in dichloromethane ( $4 \times 10$  mL, 15 min) to extract the polystyrene. The solution was then concentrated and precipitated in methanol and the solid polystyrene was filtered and dried at  $70\text{ }^{\circ}\text{C}$  overnight.

### 2.5 Stability test in acidic and basic media

An acidic solution (5 wt% trifluoroacetic acid in dichloromethane) and basic solution (1 M potassium hydroxide in water) were used to test the stability of the samples. After immersion in the solution and gentle blotting of the surface liquid, the samples were weighed at each immersion time.

### 2.6 Characterization

Thermogravimetric analysis (TGA) of the polymers was carried out using an SDTA851e Mettler Toledo analyzer. The samples were heated at a rate of  $10\text{ }^{\circ}\text{C min}^{-1}$  under a nitrogen atmosphere. The glass transition temperatures of the polymers were obtained using a DSC822e Mettler Toledo differential scanning calorimeter (DSC). The samples were heated at a rate of  $10\text{ }^{\circ}\text{C min}^{-1}$  under a nitrogen atmosphere. Polymer molecular weights and polydispersities were obtained using a Waters e2695 gel permeation chromatograph (GPC), equipped with a Viscotek 3580 refractive index detector. Tetrahydrofuran was used as eluent with a flow rate of  $1.0\text{ mL min}^{-1}$ . The molecular weights and polydispersities of the polymers were calculated using polystyrene standards. Scanning electron microscopic (SEM) images were taken at a magnification of 500x using a Hitachi S-3400N

scanning electron microscope. The mechanical properties of the polymers were determined using a universal testing machine (Instron 55R4502, S/N H3342) following ASTM D638 at a gauge length and rate of 65 mm and  $50\text{ mm min}^{-1}$ , respectively.

## 3. Results and Discussion

In this work, interpenetrating polymers were simply prepared in a domestic microwave oven using two types of polymeric substrate: polypropylene (PP) and poly(ethylene terephthalate) (PET). These substrates were subjected to interpenetrating polymerization using styrene as the monomer and BPO as the initiator. Since styrene and BPO are hydrophobic, they can polymerize within the PP and PET substrates, forming an intertangled structure that cannot be easily separated (Madhumitha, *et al.*, 2012; Paoprasert, *et al.*, 2014). The immobilization percentage was used to determine the amount of polystyrene inside each polymeric substrate. To systematically control the structure and properties of the interpenetrating polymers, we investigated the effects of the reaction conditions on the immobilization percentage as follows.

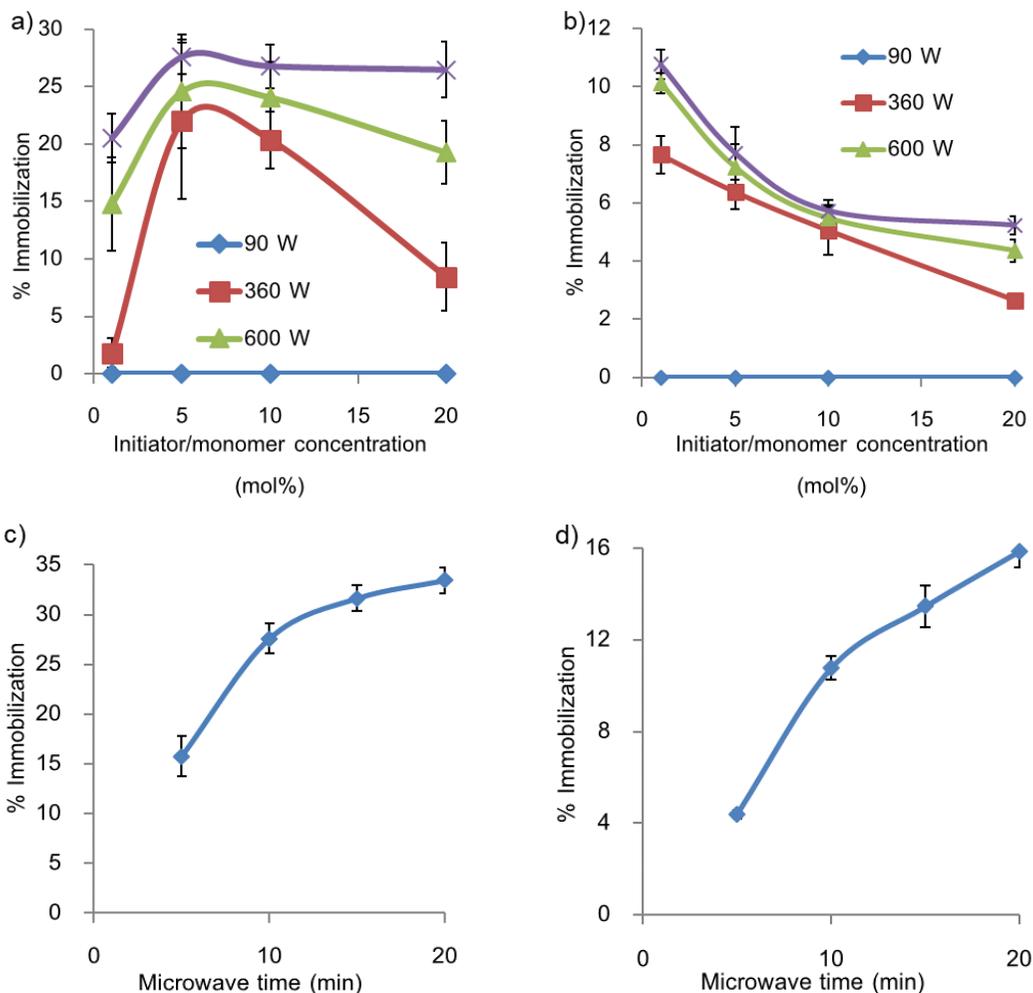
### 3.1 Effect of microwave power, initiator /monomer concentration, and reaction time

First, the effect of the microwave power level on the immobilization percentage of styrene in PET and PP was investigated (Fig. 1a and 1b). For both substrates, increasing the level increased the immobilization percentage at all concentrations. However, at a microwave power

of 90 W no polymerization was observed. This is possibly because the thermal energy supplied was insufficient to dissociate the BPO. The optimum temperature for BPO is approximately 70-100 °C (Odian, 2004).

Second, the effect of initiator/monomer concentration on the immobilization percentage was investigated. It was found that BPO concentrations of 5 and 1 wt% yielded the highest immobilization percentage for the PP

and PET substrates, respectively. Beyond these maxima, further increases in initiator/monomer concentration reduced the immobilization percentage. Clearly, the amount of polystyrene in produced PP was higher than that in PET under the same conditions. This is possibly due to the lower density and crystalline nature of PP, and greater compatibility between styrene and PP.



**Figure 1** Immobilization percentage of polystyrene as a function of microwave power, initiator/monomer concentration, and reaction time in PP (a and c) and PET (b and d).

The effect of microwave time on interpenetrating polymerization of styrene in PP and PET was next studied (Fig. 1c and 1d). The initiator/monomer concentrations of 5 wt% and 1 wt% were employed for PP and PET, respectively, at a microwave power of 800 W. It was found that, as the microwave time was increased, the immobilization percentage of polystyrene increased. However, the immobilization percentage started to plateau after 10 min of reaction time, indicating the saturation of polystyrene inside the substrate.

### 3.2 Stability in acid and base media

The stability of interpenetrating polymers in acidic and basic media was investigated and compared with those of unmodified PP and PET. The results showed that the shapes and weights of all PP samples were unchanged in either media. This is due to the fact that PP and PS are hydrocarbon polymers that are nonpolar and do not contain sensitive functional groups. In contrast, the unmodified PET samples lost their weight at a faster rate than the PS-interpenetrating samples, especially in the basic medium (Fig. 2). PET is a polyester, and its ester backbone is therefore subject to hydrolysis in both acid and base (Yoshioka, *et al.*, 2001; Holmes, *et al.*, 1993). The presence of PS retards the hydrolysis of the ester bonds, thereby slowing the hydrolysis rate. This study thus demonstrated that these PS-interpenetrating PET can be used in applications that require better durability in aqueous solutions.

### 3.3 Mechanical Properties

The mechanical properties of the PS-interpenetrating polymers were characterized and compared with those of the pristine substrates. It was found that the Young's modulus of both the PS-interpenetrating PP and PET were similar to their pristine counterparts, but the elongation of the interpenetrating samples was much lower (Table 1). The presence of PS, which is less elastic than PP and PET, reduced the elasticity of the interpenetrating polymer samples (Mark, 2009).

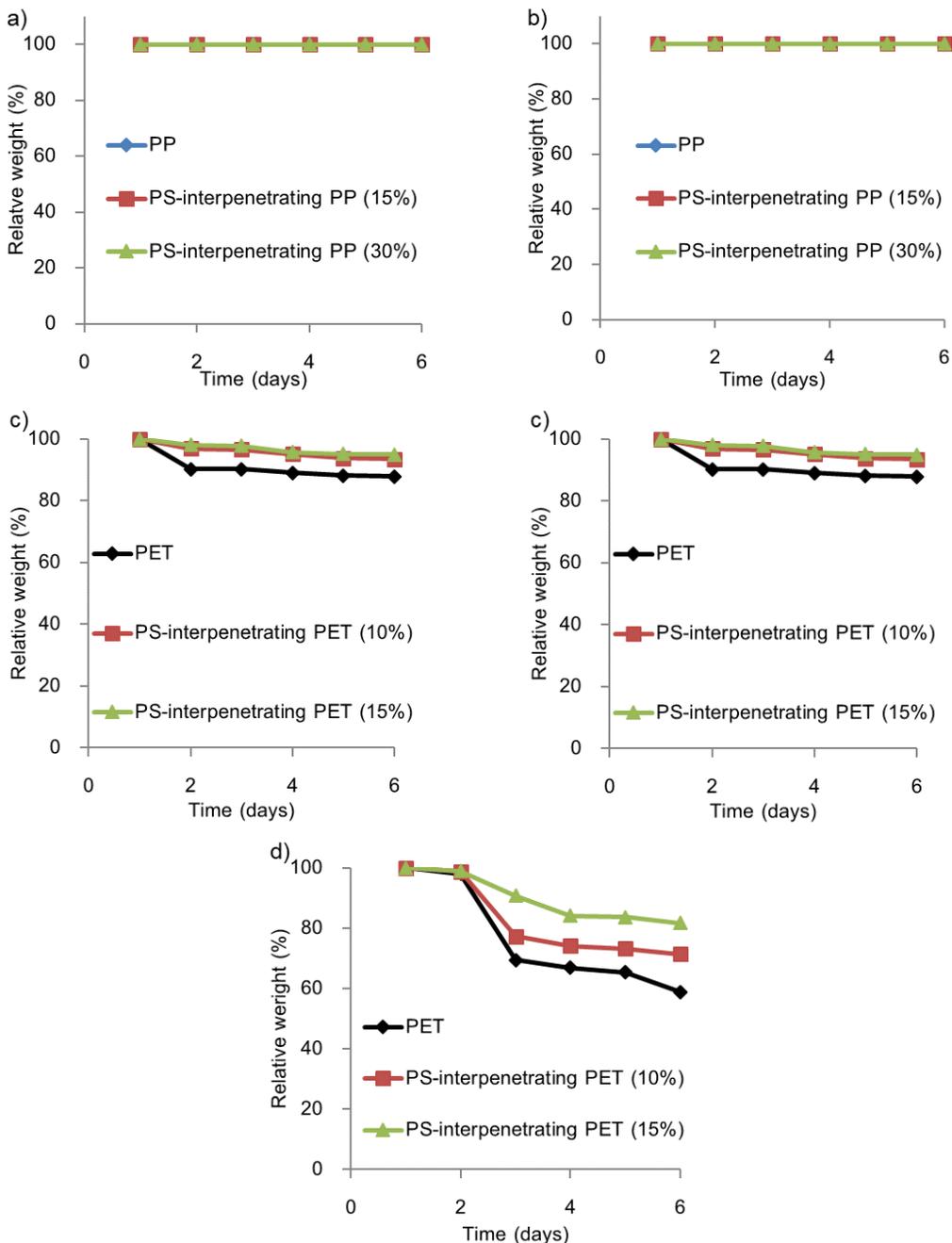
### 3.4 Thermal properties

The thermal stability of the interpenetrating polymers and pristine polymers was analyzed using TGA (Fig. 3a and 3b). The PS-interpenetrating PP showed an onset decomposition temperature at 274 °C, whereas the PS and PP decomposed at 365 and 423 °C, respectively. The onset decomposition temperature of both the PET and interpenetrating PET was 385 °C. The presence of PS did not affect the thermal stability of the PET.

The thermal transition of the polymer samples was analyzed using DSC. The DSC thermograms of the PS samples showed a glass transition temperature at 85 °C (Fig. 3c and 3d). PP and PS-interpenetrating PP showed a glass transition temperature and melting temperature of approximately 127 and 165 °C, respectively. The PET and PS-interpenetrating PET showed endothermic transition at 250 °C and 254 °C, respectively, corresponding to the melting point of PET. The presence of PS did not significantly affect the thermal transitions of the PP or PET, possibly because PP and PET continued to

constitute the majority of the materials in the interpenetrating polymers. This result thus showed that the thermal properties of inter-

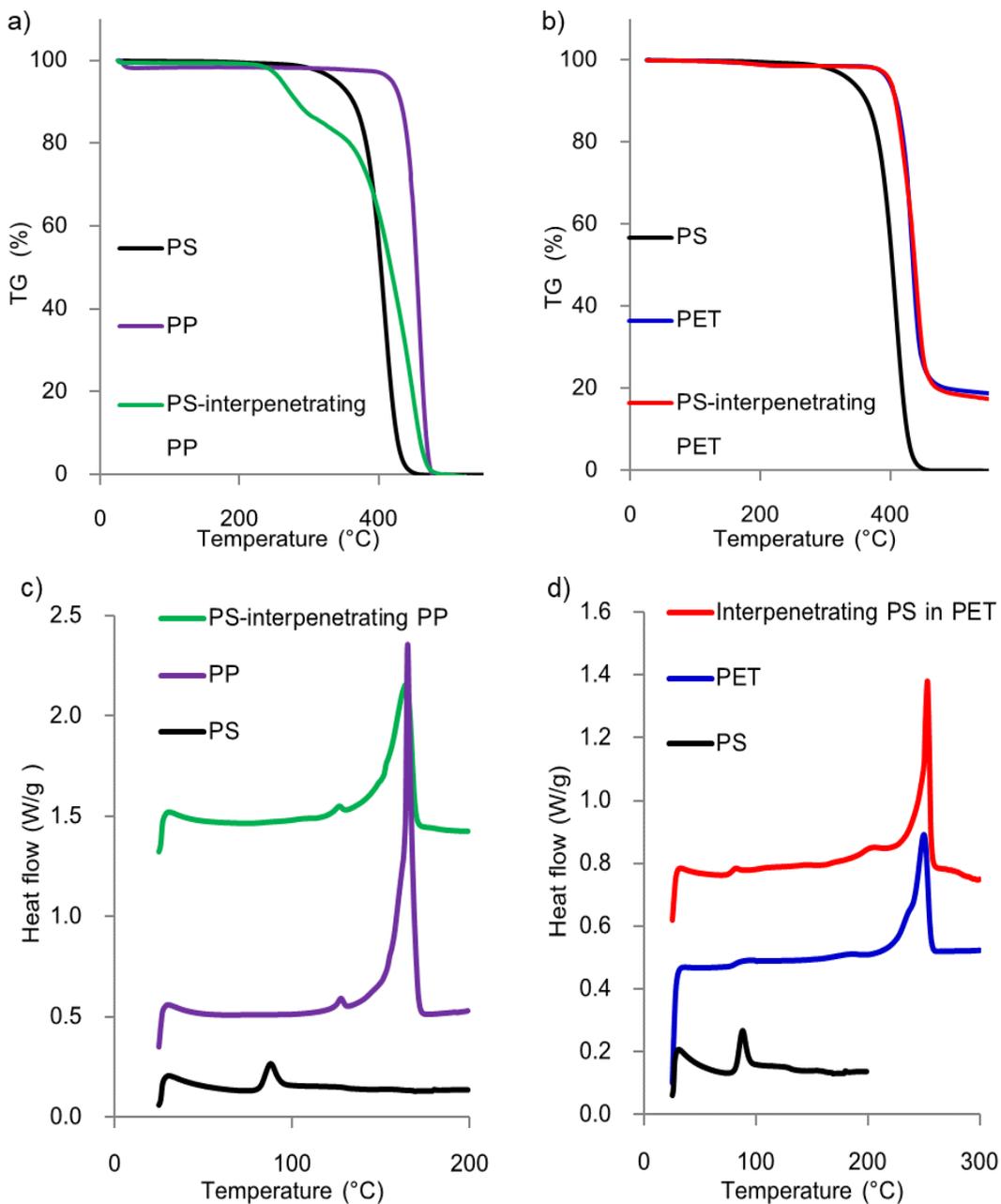
penetrating polymers were largely preserved, making them attractive as substitutes to their pristine polymers.



**Figure 2** Stability of unmodified and modified PP (a and b) and PET (c and d) in acidic (a and c) and basic (b and d) solutions.

**Table 1** Mechanical properties of interpenetrating polymers and pristine PP and PET.

| Parameter             | PP    | PS-interpenetrating PP | PET   | PS-interpenetrating PET |
|-----------------------|-------|------------------------|-------|-------------------------|
| Young's Modulus (MPa) | 1,772 | 1,990                  | 3,422 | 3,423                   |
| % Elongation          | 483.3 | 3.2                    | 6.1   | 2.7                     |

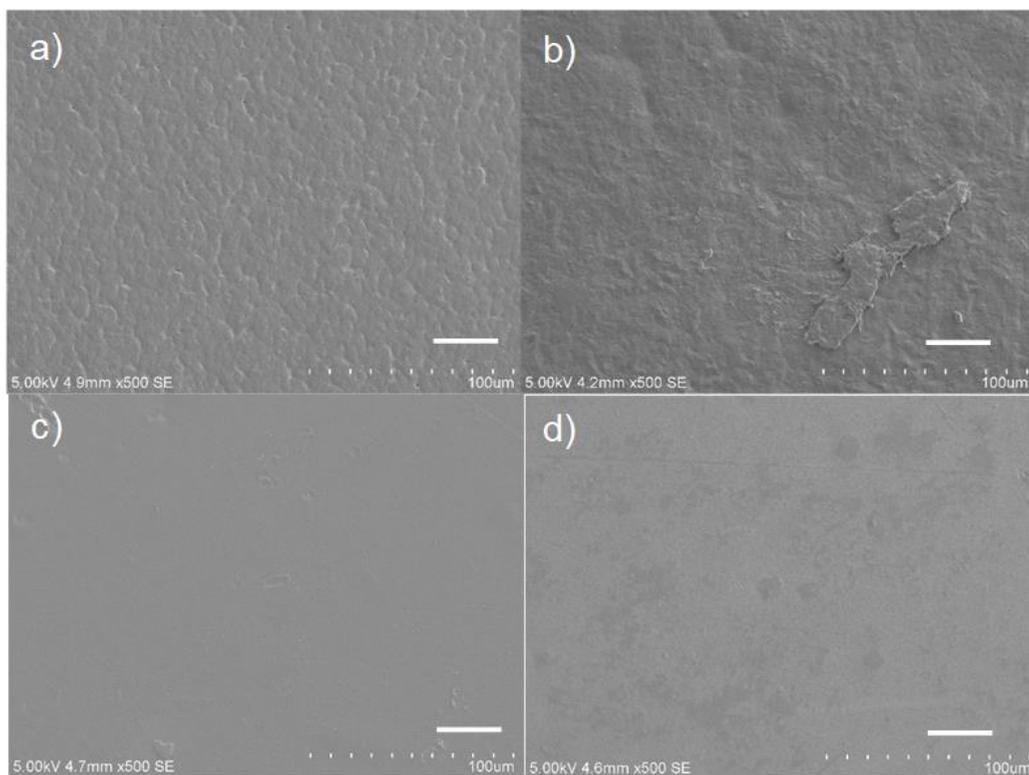


**Figure 3** TGA and DSC of PS, PET, and PS-interpenetrating PET samples.

### 3.5 Surface morphology

The surface topography of the interpenetrating samples was characterized and compared with that of unmodified substrates using SEM (Fig. 4). The interpenetrating polymers showed similar surface morphology to

the pristine counterparts. This preservation of morphology confirmed that the use of microwave irradiation did not destroy and impair the polymers. Our developed technique can be thus applied to improve their properties without negatively affecting their appearances.



**Figure 4** SEM images of a) PP, b) PS-interpenetrating PP, c) PET, and d) PS-interpenetrating PET. Scale bar is 30  $\mu\text{m}$ .

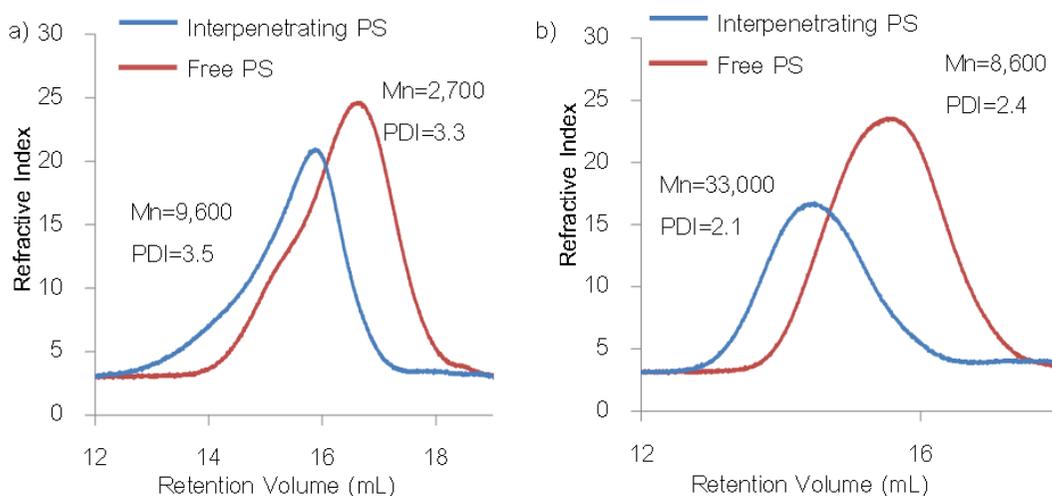
### 3.6 Molecular weight measurement

Since no crosslinker was used in the synthesis, no network structure was formed. This allowed polystyrene to be separated and extracted from the PP and PET upon dissolution for molecular weight characterization. This is consistent with previous studies, which reported no reaction between vinyl monomers and PET

when no crosslinker was employed (Paoprasert, *et al.*, 2014; Xue, *et al.*, 1995). After extraction, the molecular weight of the polystyrene was measured and compared with that of free polystyrene obtained from the reaction medium. As can be seen from Fig. 5, the free polystyrene was much shorter than the interpenetrating polystyrene. This maybe because the monomer/

initiator ratio inside the PP and PET substrates was higher than that in the solution, as the larger BPO molecules could not penetrate the PP and PET substrates as effectively as they could compared to the smaller styrene. In many previous studies, the molecular weights of free polymers have been used to describe those of immobilized polymers in various systems (Chen, *et al.*, 2006; Fan, *et al.*, 2006; Tomoaki, *et al.*, 2009), because in most cases it is not

convenient or possible to extract immobilized polymers for molecular weight measurement. This study demonstrated that the assumption that immobilized polymers and free polymers have the same molecular weights cannot be made for interpenetrating polymer systems. This is consistent with previously reported results (Behling, *et al.*, 2009). The polydispersity was above 2, which is typical for classical free radical polymerization.



**Figure 5** GPC chromatograms of free polystyrene and interpenetrating polystyrene extracted PP and PET.

#### 4. Conclusions

This paper reports a simple, versatile method for the preparation of interpenetrating polymers from PP and PET using microwave irradiation. The microwave power, reaction time, and initiator/monomer ratio were all shown to affect the immobilization percentage of styrene in both PET and PP. Immobilization percentages up to 30 and 16 % in PP and PET, respectively,

were achieved in less than 20 min of microwaving. The interpenetrating polymers showed better chemical stability in both acidic and basic media for several days over their pristine PP and PET. The technique proposed in this study allows the modification of PP and PET, creating interpenetrating polymers with superior properties for potential applications as compatibilizers for polymer blends, flexible

fabrics or commodity products that require chemical stability. In future work the applications of this strategy to the preparation of multi-interpenetrating polymers by simultaneous immobilization of multiple monomers should be investigated.

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