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RESEARCH ARTICLE

Synthesis and Thermal Characterization of new interpenetrating polymer network (IPN) Silicone- Epoxy Resins

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Abstract

Polymers are widely used in industry and in our daily life because of their diverse functionality, light weight, low cost and excellent chemical stability. However, on some applications such as heat exchangers and electronic packaging, the low thermal conductivity of polymers is one of the major technological barriers. Interpenetrating polymer network (IPN) is a novel type of polymer hybrids, which possess physicochemical properties suitable for high performance coating. Heat resistance IPN have been prepared from immiscible resins , epoxy and silicones using a cross-linking agent and a catalyst. The products were analyzed by, FTIR, TGA, DSC, UV-Vis and SEM studies .It was different from those of the individual resins. Silicone micro domains could be seen uniformly distributed in epoxy regions.

Keywords: *Silicone-Epoxy Resins, Interpenetrating polymer network (IPN), Polymer.*

Introduction

Polymers have become an important class of materials in recent years, generally due to their favorable properties, comparatively low cost and easy accessibility. However, the insufficient thermal stability and flammability of these materials still constitute a serious problem which may limit their potential multidirectional applications [1]. Moreover, well-known and effective halogenated flame retardants have been gradually prohibited as they are environmentally toxic [2].Thus, special efforts are being made to develop a new class of flame retardants for polymeric materials as an alternative for the commonly used halogenated additives.

Many studies have already demonstrated that silicones may improve the thermal stability of polymeric materials and that they could be explored as potential environmentally friendly flame retardants [3-6]. ordinarily available coating systems are heat resistant up to 333 K. As the operating temperature increases, the deterioration becomes rapid. Inorganic binder-based formulations perform well at high temperatures and protect the structure even at 1273K if suitably pigmented [7]. Interpenetrating polymer network (IPN) are a novel type of polymer blends. This is a combination of chemically dissimilar polymers in which the chain of one polymer is completely entangled with those of another. The entanglement is permanent due to the homo-cross-linking of the polymers [8]. Preparation of high-temperature IPN composition from vinyl/acrylic copolymer with polystyrene/styrene–butadiene is known [9].

The present work deals with IPN in which two resins (epoxy and silicone resins), which are immiscible with each other, were combined by using a cross-linking agent and a catalyst. The influence of siloxane resins on thermal stability and fire behavior of the epoxy matrix is discussed. (Composites containing 50 wt % of epoxy were prepared by the melt blending method. The thermal stability of neat siloxane resins, as well as of the obtained composites was examined by the thermogravimetric (TG) analysis in both inert and oxidative environments [10,11]

Experimental

Materials

Epoxy resin was a FOSROC Co. Product (nitofill EP L-V), Jordon. The density of epoxy resin was 1.04 gm.cm-3, silicone resin was manufactured commercially, Turkey. Hardener, The ratio between resin and hardener for this study was **5**:1

Instruments

FTIR 8400S, Fourier Transform infrared spectrophotometer, SHIMADZU, Japan), (Oven, Trivp International Crop. Italy), (UV-Visible 1800,Shimadzo). SEM (Bruker, Nano GmbH model Germany),(Thermogravimetry analysis (TGA) , using a heating rate of 10ºC/min in Argon atmosphere within the temperature range of 25-600ºC)(differential thermal analysis (DSC) measurement using apparatus (DSC) in the college of Education for pure scieneses Ibn al-Haitham/ University of Baghdad). Shaking Incubator (Heidolph unimax 1010\ Germany), pH-meter(Hanna\ USA).

Interpenetrating polymer network (IPN) were prepared by using 50g resin solution of an air-drying silicone resin,50g epoxy resin 10g Aromatic amine MPDA hardener, . The IPN were prepared from these resin solution after refluxing the resin composition with a catalyst and a cross-linking agent at 353K for 1h .A description of the IPNs as well as thickness and drying time the resin and the IPN were coated on sand-blasted mild steel substrate and dried for 7 days.

The baking type silicone resin-coated panels were baked in an air oven for 15 min at 453 k. after complete drying, the resin and IPN-coated panels were characterized by FT-IR , TG, DSC,UV-Vis and SEM. The coated panels were also subjected to salt spray tests. Their heat resistance properties and electrochemical impedance and mechanical properties were also investigated.

Table 1: Shows the materials used

System	Weight
Silicone resin	50 g
Epoxy resin	50 g
Hardener	10 g
Catalyst	0.15 g
Crosslinking agent	1 g

Results and Discussions

Characterization of Resins and IPN

Fourier Transform-Infrared Spectroscopy (FTIR)

In the Figure (1) FT IR spectra of IPN, The peaks of the epoxy and silicone resins are nearly similar except for the absence of the peak at 1110–1107 cm^{-1} in the IPN. This is maybe due to the interlinking point of silicone and the epoxy resin in the formation of IPN. The presence of broad band between 1041 and 1134 cm^{-1} confirms a Ti–O linkage followed by ester group, indicating an effective participation of the cross-linking agent during the formation of IPN. The characteristic terminal epoxy group peaks remain unaltered in these IPN. These epoxy groups further react with the amine hardener.

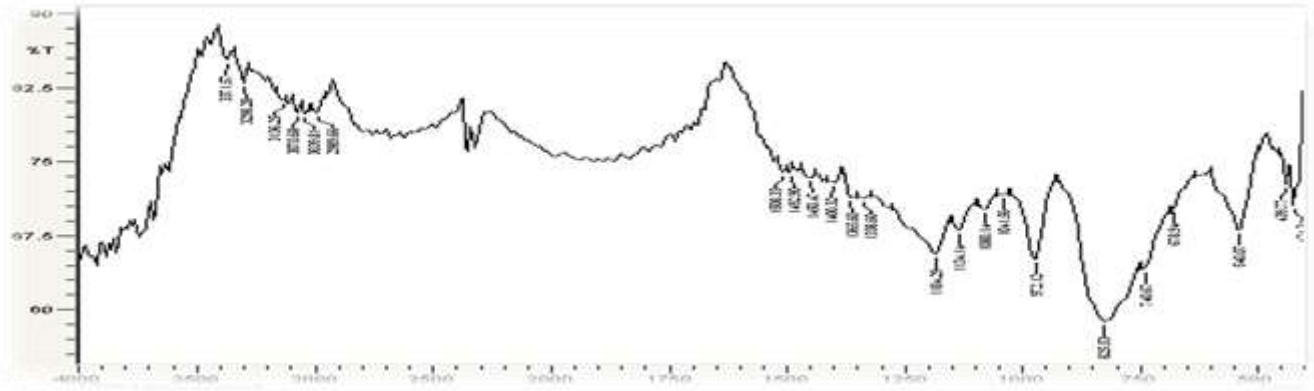

Figure1: FTIR spectrum of IPN

Thermal Properties

Thermo Gravimetric Analysis (TGA) Study

Thermo gravimetric Analysis (TGA) involves determining changes in mass as a function of temperature. It is usually used to research degradation temperatures, absorbed content of materials, levels of in organic and organic parts contained in a material and analyses solvent residues. It employs a sensitive electronic balance from which the sample is suspended in a furnace controlled by a temperature programmer. The thermal properties of samples of these polymer were investigated by means of thermo gravimetric analysis (TGA) in Argon atmosphere at heating rate of 10 °C/min and the results such as T_i, T_{op} , T_f , $T_{50\%}$, % Residue at 600 °C , and char yields at 500°C are summarized in (Table 2).The temperatures of 50% weight loss of(IPN) as a standard indication for thermal stability of polymers were all from 500 to 600°C, The char yields of Polymer at 500°C are 58% until 66% in Argon atmosphere , which indicate they could meet high temperature resistant requirements as some specific materials in modern aerospace.

Table 2: Thermal behavior data of polymer

Polymer	DT/°C				$T_{50\%}$	Residue at °C600	Char % *at 500°C*
	T_i	T_{op1}	T_{op2}	T_f			
Polymer	144.14	256.91	304.34	422.69	384.43	91%	69%

DT: *Decomposition temperature.*
T_i : *Initial decomposition temperature.*
T_{op} : *Optimum decomposition temperature.*
T_f : *Final decomposition temperature.*
$T_{50\%}$: *Temperature of 50% weight loss, obtained from TGA.*

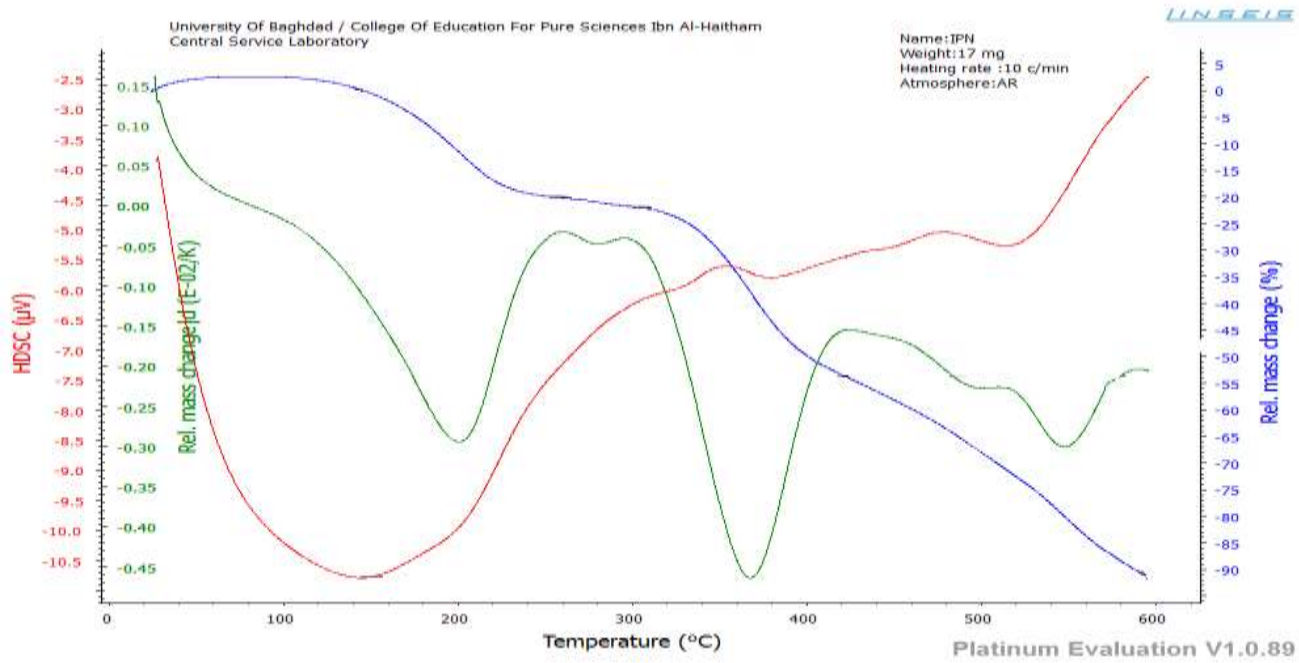

Figure 2: TGA and DSC for IPN

Differential Scanning Calorimeter Analysis (DSC) Study

Differential Scanning Calorimetry, is a technique of thermal analysis that investigates how material's heat capacity (Cp) is transformed by temperature. A known mass sample is heated or cooled and the variations in its heat capacity are observed as alterations in the heat flow. This allows to reveal transitions such as melts glass transitions (Tg), and the melting point (Tm) the degree of crystallization (Tc) This test was applied to the prepared samples , Shows the Figure(2) for the sample of Polymer, the results showed the value of the glass transition (Tg) of the mixture (80°C), referring to obtain the flow temperature and then increases endothermic

the sample to reach the melting point (Tm) at (520°C) completely dissolve and then less the sample heat absorption rate and by a curved appointed degree of crystallization (Tc) of the mixture was (150°C).

Table 3: shows the degree of glass transition, melting point and the degree of crystallization in the differential thermal analysis of Polymer

Samples	Tg (°C)	Tm (°C)	Tc (°C)
INPs	**80**	**520**	**150**

Tg : Degree glass transition
Tm : Melting Point
Tc : Degree of crystallization

Surface Morphology of IPNs

SEM images of IPN is shown in Fig. [3] and [4] . respectively. The silicone microdomains are distributed all over the epoxy region of IPN. But it is very clearly seen from these figures that the degree of entanglement, since the microdomains are more uniformly distributed in the former. it shows both the spherical and some fibrous clusters of IPN structures.

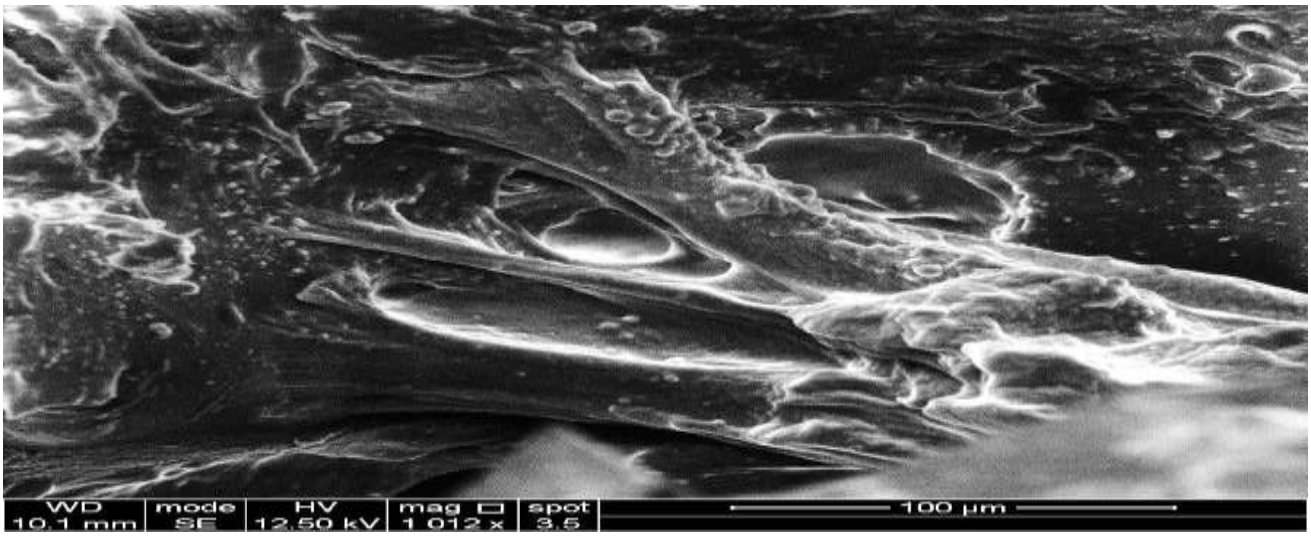

Figure 3: SEM images for IPN

Figure 4: SEM images for IPN

The Optical Characteristics for Polymeric Solutions

Absorption

The UV-Vis absorption spectra of the interpenetrating polymer network (IPN) region of the electromagnetic spectrum (215-310) nm which called the fundamental absorption edge of the polymeric blends. These optical absorptions are associated with the electronic transitions from highly occupied molecular orbital (HOMO) π-band to lowly unoccupied molecular orbital (LUMO)

π*-band of electronic states. The absorbance at 215 nm band is due This is due to the large number of reactive polar groups present in the baking type silicone resin formulation as shown in Fig [5] . The absorbance at λ= 390 nm band increases with the increase of IPN in which two resins (epoxy and silicone resins), which are immiscible with each other, were combined by using a cross-linking agent and a catalyst. Thus, more polymerization is takes place. The result is in agreement with the recent study on silicone micro domains could be seen uniformly distributed in epoxy regions absorption bands were at 320-800 nm wavelengths [13].

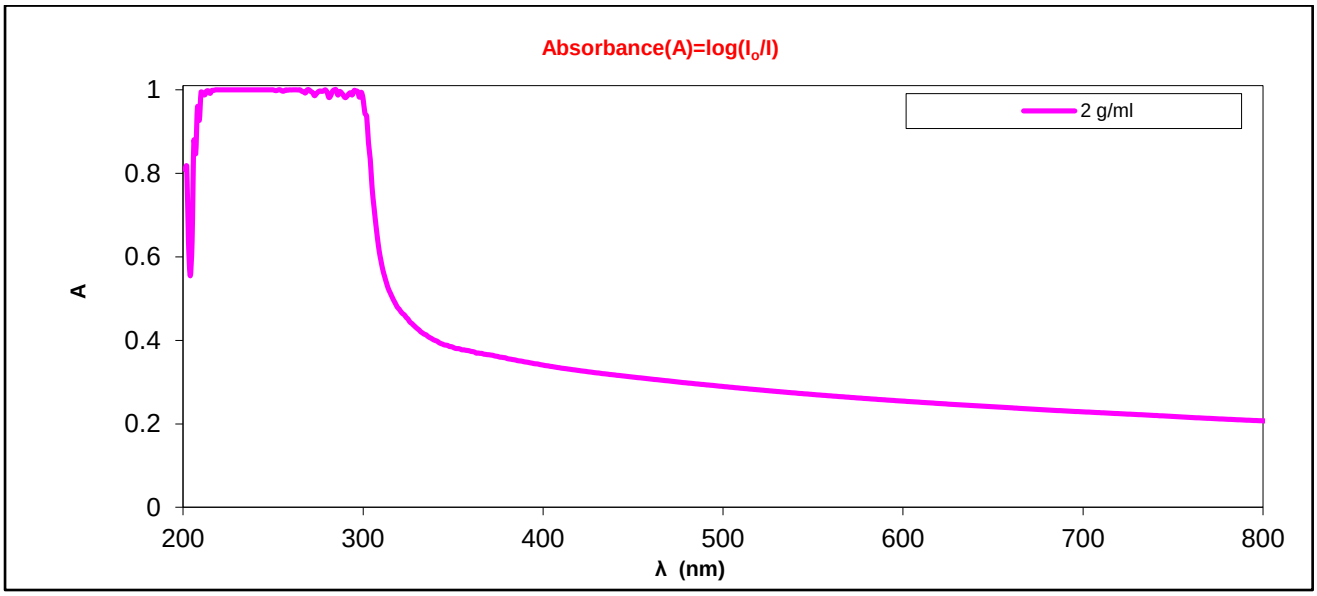

Figure 5: The absorbance as a function of wavelength for (IPN)

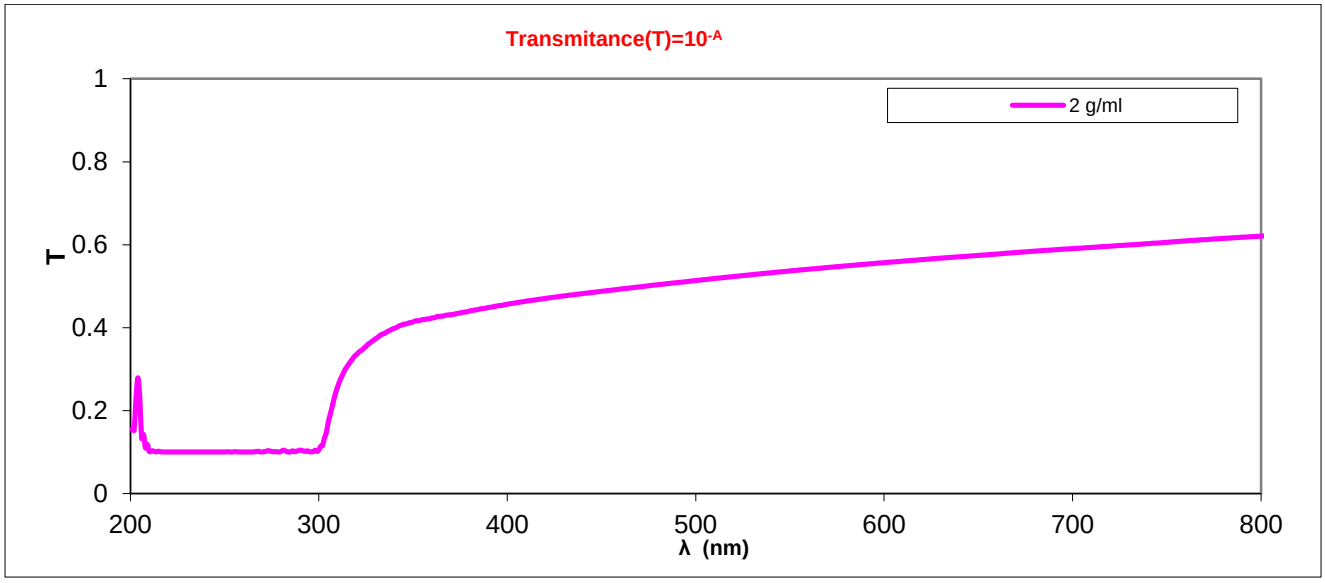

Figure 6: Transmittance as a function of wavelength for (IPN)

Transmittance

The optical transmittance spectra is measured for the interpenetrating polymer network (IPN) in the wavelengths rang (200-800) nm as shown in Figure (6).

From the figure it can be noticed that transmittance increase with the wavelength due to the decrease absorbance with the wavelength (13) .

Conclusion

- Heat resistant interpenetrating Polymer networks (IPN) were prepared from epoxy and silicone resin with Aromatic amine as hardener.
- The formation of IPN is confirmed by FT-IR, UV-Vis, TGA, DSC, and SEM analysis.
- The FT-IR spectra show the presence of Ti-O linkage in the IPN molecules.

- The TG/DSC and heat resistance studies confirm the superior heat resistance properties of the IPN as compared to the individual resin.
- The SEM images suggest that the silicone grains are uniformly distributed throughout the epoxy resin confirming the formation of IPN.

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