

Production of Sequential Interpenetrating Polymer Networks from Ximenia americana Seed Oil-based Polyurethanes and Polystyrene

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ABSTRACT: Interpenetrating polymer network are combinations of two or more polymer in network form. The objective of this paper was to evaluate the production and characterization of sequential polyurethane-polystyrene interpenetrating polymer networks from different diisocyanates and varied styrene content using Ximenia americana seed oil as base material. The polymer networks were characterized for their Tensile, Swelling and Thermal properties. The tensile strength and tensile modulus for MDPU-1.50-PS polymer networks, 57.86 ±5.42 -422.85±15.25 MPa and 2.26±0.91 - 11.08±4.21 MPa respectively are higher than the values represented for HDPU-1.50-PS and TDPU-1.50-PS polymer networks, but the latter polymer networks are higher in values for elongation at break than the former. This is also corroborated by the swelling mass ratio (q_m) with values for HDPU-1.50-PS and TDPU-1.50-PS networks higher than those for MDPU-1.50-PS, consistent with lower polystyrene crosslinks in the former polymer networks. Thermal studies present HDPU-1.50-PS-20 as the most stable network at 10% degradation, but at higher degradation temperatures MDPU-1.50-PS-20 polymer network shows stability up to 600°C with 19.40g residual weight of char polymer. This study shows that derivatised Ximenia americana seed oil is suitable as starting material for preparation of an interpenetrating polymer network.

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One of the important routes for modifying the properties of polymers to meet specific needs is the physico-chemical combination of two or more structurally different polymers. The properties of such multicomponent polymeric systems depend on the properties of individual polymers and the ways in which they are combined Chenal and Widmair, (2005); Quinzler et al., (2010). Generally, mixing of polymers results in a multiphase morphology because of their thermodynamic incompatibility. This is due to relative small gain in entropy upon the mixing of polymers, as long chain length imparts contiguity restrictions Naveed et al., (2022). The degree of phase

the polymers, varies widely for different interpenetrating polymer networks Lipatov and Tatiana, (2007). Production of interpenetrating polymer networks is highly significant and interesting because it is the only way of intimately blending two crosslinked polymers. The use of naturally functionalized triglyceride oils in interpenetrating polymer networks has gained conspicuous importance Frisch et al., (2004); Guner et al., (2006). Triglyceride chains are derivatised to introduce polymerizable moieties that react easily with bi or poly-functional monomers to produce a step-growth polymeric

separation, which depends mainly on the miscibility of

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backbone which in the presence of an initiator and a vinyl monomer forms sequential or simultaneous interpenetrating polymer networks Valero et al., (2009). The dynamic mechanical properties of polystyrene-polyurethane blends have been studied Theocaris and Kefalas (1991). The effect of styrene content on the properties of polyurethane-polystyrene interpenetrating polymer networks based on transesterified castor oil has shown the dependent on the properties of polymer networks on styrene contents Dave and Hasmukh, (2017). The effects of temperature and molecular structure variation on gas through polyurethane-polystyrene transportation interpenetrating polymer networks, has been reported Doo et al., (1991). Ramis et al., (2001) reported the thermal and dynamic mechanical thermal behavior of polyurethane-unsaturated polyester interpenetrating polymer networks. The structure-property relationships of simultaneous interpenetrating polymer networks of polyurethane from pentaerythritol-modified castor oil and polystyrene have been presented Valero et al., (2009). Samanthi et al., (2004) studied the synthesis, chemical, mechanical, thermal and morphological properties of interpenetrating polymer networks based on polyol modified castor oil polyurethane and poly (2ethoxyethyl metacrylate). This study aimed to produce and characterized a sequential polyurethanepolystyrene interpenetrating polymer networks from different diisocyanates and varied styrene content, using Ximenia americana seed oil as base material collected in Yola, Adamawa State, Nigeria.

MATERIAL AND METHODS

Collection and preparation of Ximenia americana seeds: The seeds of *Ximenia americana* were collected in Yola, Adamawa State, Nigeria. The seeds were shelled by cracking to remove the kernel. The kernel were washed with water, sun-dried for two weeks (14 days) and milled into paste with a previously cleaned blender (Binatone, Japan, model BLG-400). The paste was then packed in polyethylene bag and stored in a refrigerator prior to oil extraction process.

Extraction of Ximenia americana seed oil: Oil was extracted from the paste with petroleum ether (40-60 °C) using a soxhlet extractor (Eromosele and Eromosele, 2002). 100 g of ground *Ximenia americana* seeds were packed in a soxhlet thimble and 500 ml solvent (petroleum ether) was poured into the 1000 ml soxhlet round bottom distillation flask. A condenser was connected to the top of the extractor. The heating mantle was switched on (set at 50 °C) and heat was supplied to the distillation flask. Vaporized solvent were generated and condensed as hot liquid solvent in the packed thimble. This process continued

for 3 hours and resulted in the extraction of the oil through various siphoning of oil plus solvent into the distillation flask.

Glycerolysis of Ximenia americana seed oil: Ximenia americana seed oil and glycerol; molar ratio of 1:3 were poured into a 500 ml three- neck reaction round bottom flask equipped with a stirrer, a thermometer and nitrogen inlet tube. The mixture was stirred (300 rpm) and heated on a thermostatic heating mantle to a temperature of 200°C for 45 minutes. At this point, calcium hydroxide (0.1 wt % of the oil) was added as catalyst and the temperature raised to 230°C and maintained at this temperature for 3 h, after which an aliquot was withdrawn from the reaction mixture and tested for its solubility in methanol. After, cooling the reaction mixture it was dissolved with diethyl ether and washed first with 0.1 M HCl solution and then with distilled water to remove the catalyst and the residual glycerol. The ethereal layer was then dried over sodium sulphate for 24 h. The residual solvent was removed with the aid of a rotary evaporator. The ensuing product (Ximenia diol) was recovered and dried for 48h over silica gel.

Preparation *polyurethane--polystyrene* ofnetwork: interpenetrating polymer The interpenetrating polymer network was prepared via grafting/crosslinking of styrene monomer onto the polyurethane backbone, by free radical polymerization initiated by benzoyl perioxide (BPO). A calculated amount of 1, 6 -hexamethylene diisocyanate (HDI) corresponding to 1.50 isocyanate index was put into a 500 ml three-neck round bottom flask. The flask was fitted with a stirrer, thermometer, reflux condenser and a nitrogen inlet tube. Ximenia americana diol previously dried over silica gel for 24h was added along with 0.002 wt. % of dibutyltindilaurate compared to the weight of diol. The flask and its accessory were then mounted on a thermostatic heating mantle. The temperature of the mantle was set at 80° C and the polymerization was maintained for 90 minutes with continuous stirring (100 rpm) under nitrogen atmosphere. This resulted, into а polyurethane resin (HDPU) in form of a viscous liquid. 20 ml of toluene was added and stirred (200 rpm) for 10 minutes. The initiator 1% compared to the weight of styrene (Sharma and Kundu, 2006) was added slowly, over a period of 5 minutes into the reaction mixture, the temperature of the mantle was adjusted to 120° C and stirred (200 rpm) for 20 minutes to enable the homolytic cleavage of benzoyl peroxide into a highly reactive phenyl radical and CO₂ (Penezek, et al., 2005). Calculated amount of styrene as formulated (table 1) was added to the reaction mixture, with the temperature maintained at 120° C for

30 minutes under nitrogen atmosphere. Finally, the mixture was poured into a Teflon mould, covered to prevent moisture and allowed to cool to ambient temperature. The mould and its content were then cured in an oven set at 130° C for 6 h. The interpenetrating polymer sheets was later removed from the mold and stored at ambient temperature for seven (7) days. This sample produced from 1, 6-

hexamethylene diisocyanate, at 1.50 isocyanate index and 10% styrene content was coded HDPU-1.50-PS-10. The other polymer network samples containing toluene diisocyanate (TDI) and 4'4 diphenyl methylene diisocyanate (MDI) and varying content of styrene as shown in Table 1 were prepared by the same procedure as outlined above.

Table 1: Formulation for	r Polyurethane-I	Polystyrene In	iterpenetratir	g Polymer Networks.
Sample code	Styrene (g)	BPO (g)	Diol (g)	Diisocyanate (g)
HDPU-1.50-PS-10	2.739	0.027	13.338	11.313
HDPU-1.50-PS-15	4.109	0.041	12.597	10.685
HDPU-1.50-PS-20	5.478	0.055	11.856	10.056
HDPU-1.50-PS-25	6.848	0.068	11.115	9.428
HDPU-1.50-PS-30	8.217	0.082	10.374	8.799
TDPU-1.50-PS-10	2.779	0.028	13.338	11.673
TDPU-1.50-PS-15	4.169	0.042	12.597	11.025
TDPU-1.50-PS-20	5.558	0.056	11.856	10.376
TDPU-1.50-PS-25	6.948	0.069	11.115	9.728
TDPU-1.50-PS-30	8.337	0.083	10.374	9.079
MDPU-1.50-PS-10	3.344	0.033	13.338	16.758
MDPU-1.50-PS-15	5.016	0.050	12.597	15.827
MDPU-1.50-PS-20	6.688	0.067	11.856	14.896
MDPU-1.50-PS-25	8.360	0.084	11.115	13.965
MDPU-1.50-PS-30	10.032	0.100	10.374	13.034

Initiator (g) = 1% of styrene (g)

Determination of tensile properties of polyurethanepolystyrene: Cured polyurethane-polystyrene polymeric networks were conditioned in a dissicator (containing silica gel) for 48h. According to ASTM-D412 specification, tensile properties were determined from stress-strain curves, with an Instron Tensile Test Machine (Model M500-25KN), at a strain rate of 10mm min⁻¹ using rectangular specimens (50mm × 25mm × 2mm) at ambient temperature. Readings were taken in triplicate for each polymer network. The values reported are the mean and standard deviation respectively.

Swelling studies of interpenetrating polymer network: The swelling ratio of the polymeric network were assessed in four different solvents; Toluene (organic solvent), 4% NaOH (alkaline solution), 4% H₂SO₄ (acidic solution) and 4% NaCl (saline solution). The sorption of solvent by the interpenetrating polymer network was determined by the pat-and-weigh method (Fieldson and Barbari, 1993). Network sheets (50 mm×25mm×2mm) after measuring their dry weight were placed in the solvents contained in a standard test-tube and stopped with aluminum foil, supported on a rack and maintained at room temperature (30 $\pm 2^{\circ}$ C). The sheets were taken out at regular intervals (24h) and solvent adhering to the surface was wiped off with cotton wool, weighed immediately and replaced in the solvent. This procedure was carried-out for three samples from each polymer network. The swelling mass ratio (q_m) after each interval was evaluated using the equation 1;

Swelling mass ratio(qm) =
$$\frac{W}{W}$$
 (1)

Where W = mass of swollen polymer network and w = mass of dry polymer network

FT-IR spectroscopy: The structural components of *Ximenia* polyurethane-polystyrene interpenetrating network was studied with a Bruker fourier transform infrared spectrophotometer (Model 12028429). The sample was mixed with KBr cells and the spectrum was recorded, scanned in the range, 3500 to 500 cm⁻¹.

Thermal Analysis of polymer networks: The thermal stabilities of *Ximenia americana* aliphatic polyurethane-polystyrene and aromatic polyurethane-polystyrene networks were studied with a Perkin Elmer Thermal Analyzer. For each study the sample was heated under nitrogen atmosphere from 30°C at an incremental rate of 10°C/min up to 950°C.

RESULT AND DISCUSSIONS

Figure 1 presents the structural make-up of the polymer networks and the mechanical (Tensile Strength, Tensile Modulus and Elongation at break) properties for the polymer networks are presented in the Table 2.



Fig 1: FTIR spectrum of Ximenia polyurethane-polystyrene network

Fable 2: Tensile properties of Ximenia-based interpenetrating polymer networks
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Polymer sample	Tensile Strength (MPa)	Tensile Modulus (MPa)	Elongation @ Break (%)
HDPU-1.50-PS-10	53.68±0.33	2.34 ± 0.15	26.13±0.27
HDPU-1.50-PS-15	57.09±0.61	2.79 ± 0.09	16.87±0.20
HDPU-1.50-PS-20	64.94±0.66	2.96 ± 0.07	10.06±0.30
HDPU-1.50-PS-25	73.13±2.77	2.81 ± 0.04	11.96±0.18
HDPU-1.50-PS-30	55.21±0.70	2.47±0.13	13.25±0.24
TDPU-1.50-PS-10	55.07±0.87	2.41 ± 0.04	2.45±0.02
TDPU-1.50-PS-15	61.99±1.66	3.41 ±0.01	2.16±0.01
TDPU-1.50-PS-20	79.35±4.65	3.82±0.03	1.94 ± 0.01
TDPU-1.50-PS-25	76.17±3.52	3.67±0.01	2.38±0.04
TDPU-1.50-PS-30	73.13±2.77	3.34 ± 0.02	2.48 ± 0.01
MDPU-1.50-PS-10	57.86±5.42	2.26±0.91	10.72 ± 1.61
MDPU-1.50-PS-15	69.76±4.18	3.48 ± 1.80	7.58±1.54
MDPU-1.50-PS-20	126.27±6.99	$4.14{\pm}1.56$	5.48 ± 1.21
MDPU-1.50-PS-25	220.81±15.53	5.96±3.33	4.72±1.00
MDPU-1.50-PS-30	422.85±15.25	11.08 ± 4.21	3.64±0.98

Figure 1 shows the spectrum of Ximenia polyurethanepolystyrene interpenetrating polymer network. The characteristic peak at 3337 cm⁻¹ indicates the presence of urethane group. The band at 1017 cm⁻¹ indicates the presence of aromatic ring of polystyrene (Dave and Hasmukh, 2017), corroborated by the 2853 cm⁻¹ band for methylene stretch of polystyrene (Dave and Hasmukh, 2017). The tensile strength and tensile modulus for HDPU-PS and TDPU-PS networks increase with styrene content (10-20 wt.%) and then decreases at higher styrene content with a corresponding increase in elongation at break. For the MDPU-PS polymer networks the tensile strength and tensile modulus increase with styrene content in the range 10-30 wt.% with corresponding decrease in elongation at break. The decrease in tensile strength and tensile modulus for HDPU-PS and TDPU-PS polymer networks at higher styrene content may be associated with increased formation of styrene homopolymer and attendant phase separation between the hydrophobic polystyrene and polyurethane in the interpenetrating polymer network Kundu and Larock, (2005). Hence, this macroheterogeniety causes a decrease in stress transfer and result in low tensile properties and increase in elongation of the two phases

owing to poor networking within their boundaries Li and Larock, (2003); Li et al., (2003); Mathew et al., (2003). The highest tensile properties and low values of elongation at break recorded for MDPU-PS interpenetrating polymer networks with styrene content in the range 10-30wt.% compared to HDPU-PS and TDPU-PS networks may be attributed to the following: the amorphous nature of MDPU polymer, which favors the diffusion of more graft initiating phenyl radical and styrene monomer into the polyurethane matrix Li and Larock, (2003). The preponderance and proximity of crosslinking sites due to diallylic (-CH=CHCH₂CH=CH-) carbon that may be present in the pendant fatty acid chain occasioned by the high level of polyene present in linoleic and linolenic fatty acids of Ximenia seed oil Eromosele and Eromosele, (2002): Hiemenz and Lodge, (2007); allylic carbon on the methylene link of methylene diphenyl diisocyanate monomer Penezek et al., (2005), and allylic site on the allophanates crosslinks Can et al., (2001). All these Suggest, more crosslinking reactions in this network, initiated by reactive phenyl radical which results in shorter rigid polystyrene crosslink chains. Under stress, the shorter rigid crosslink chains act in concert to transfer and hold-up stress longer before failure, thereby, exhibiting higher tensile properties and lower elongation.



Fig 2: Swelling mass ratio of HDPU-PS polymer networks in various media



Fig 3: Swelling mass ratio of TDPU-PS polymer networks in various media

The tensile strength for HDPU-PS is higher by several orders than the 2.9-34 MPa reported for castor-based polyurethane crosslinked with 2-ethoxy ethyl methacrylate Samanthi *et al.*, (2004). Thus, the higher tensile strength and tensile modulus for *Ximenia*-based interpenetrating polymer networks may be linked to the high content of linoleic and lenolenic fatty acids Eromosele and Eromosele, (2002), which provide more sites for crosslinks occasioned by vinyl groups endowed on the pendant fatty acid chain of the polyurethane backbone. This is corroborated by the observation also that castor oil-based polyurethane

interpenetrating polymer network from MDI crosslinked with methylacrylate showed lower tensile strength (0.43-1.87 MPa) and higher elongation (8.4-10.2 %) compared to the corresponding values for *Ximenia*-based polymer network having values for tensile strength and elongation in the ranges 57.21-422.85 MPa and 3.64-10.72 % respectively.



Fig 4: Swelling mass ratio of MDPU-PS polymer networks in various media

The plots of swelling mass ratio (q_m) against styrene content are shown in Figures 2 to 4. The swelling mass ratios for the interpenetrating polymer networks in all the solvents generally decreased with increase in styrene content consistent with increasing crosslinks. However, for HDPU-PS and TDPU-PS networks, in toluene and 4% NaOH, the decrease is characterized by a minimum at 20 wt.% styrene content beyond which there is a marginal increase in swelling mass ratio. This is not the case for MDPU-PS network. In all the solvents, the swelling mass ratio is in the order toluene>4%NaOH>4%H2SO4>4%NaCl. Hence, the maximum swelling is observed in toluene. This may be attributed to toluene being a good solvent for the interpenetrating polymer networks having solubility parameter, (δ) 8.9 (cal/cm³)^{1/2} which is close to that of polystyrene, $(9.1 \text{ (cal/cm}^3)^{1/2})$ (Barton, 1991). The closeness of the solubility parameters is expected to facilitate ready diffusion of toluene molecules into the polymer networks and solvation of polystyrene crosslinks. The other media are aqueous and polar in nature and show less penetration into a relatively nonpolar network Sharma and Kundu, (2008). The low swelling of the interpenetrating polymer networks in polar solvents clearly suggests the overriding influence of the hydrophobic polystyrene in the polymer networks. The swelling mass ratios for HDPU-PS and TDPU-PS interpenetrating polymer

crosslinks.



Table 3: Thermogravimetric data for polyurethane-polystyrene polymer networks							
Polymer Sample	T ₁ (°C)	T _{10%} (°C)	T _{50%} (⁰ C)	T _{max} (°C)	Residual weight at 600 °C (%)		
HDPU-PS-20	296	341	422	556	11.20		
TDPU-PS-20	255	320	448	580	8.90		
MDPU-PS-20	247	299	466	600	19.40		

Figures 5-7 and Table 3 shows the thermograms and thermogravimetric data respectively for thermal degradation of polyurethane-polystyrene interpenetrating polymer network, prepared at 20 wt.% styrene content. From the onset degradation temperature (T_1) , it is evident that the relative stabilities of the polymer networks is in the order HDPU-PS-20 > TDPU-PS-20 > MDPU-PS-20 i.e, 296, 255 and 247°C respectively. The weight loss at these temperatures is less than 10% and may be attributed to decrosslinking of allophanates and decomposition of urethane groups. Crystallization of symmetrical monomer chains in aliphatic

polyurethane backbone of HDPU-PS-20 interpenetrating polymer network may account for its relative stability compared to the others Petel and Suther, (1987); Prashantha et al., (2001). However, from T_{max}, The order of stability of the interpenetrating polymer network is MDPU-PS-20 > TDPU-PS-20 > HDPU-PS-20. This suggests a higher degree of polystyrene crosslinks in MDPU-PS-20 expected to enhance stability at high temperatures.

Fig 7: thermogram for MDPU-PS

Polyurethane-polystyrene Conclusion: interpenetrating polymer networks have been produced from Ximenia americana seed oil as starting material and characterized. The effect of diisocyanate

type and styrene content on the tensile, swelling and thermal properties of the polymer networks is a clearcut. *Ximenia americana* seed oil could find application as a bioresource material for the polymer industry.

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