

Pongamia glabra Seed Oil Based Poly(urethane–fatty amide)

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ABSTRACT: Poly(urethane–fatty amide) (PUPGFA) was developed to use nonedible and nondrying *Pongamia glabra* seed oil. The resin was synthesized by the reaction of *N,N*-bis(2-hydroxyethyl) *P. glabra* fatty amide (HEPGFA) and tolylene-2,4-diisocyanate through a one-shot technique with a minimal amount (8–10%) of organic solvent. The structural elucidation of the resin was carried out by Fourier transform infrared (FTIR), ¹H-NMR, and ¹³C-NMR spectroscopic techniques, whereas the curing mechanism was confirmed by FTIR spectroscopy and differential scanning calorimetry. The solubility of the resin was studied in different polar and nonpolar solvents. Thermal analysis was carried out by thermogravimetric analysis and differ-

ential thermal analysis techniques. Antibacterial studies of HEPGFA and PUPGFA were performed in the presence of Gram-negative *Salmonella* and Gram-positive *Staphylococcus aureus* bacteria by an agar diffusion method. Physicochemical, physicochemical, and corrosion-resistance tests were performed by standard laboratory methods. PUPGFA had good physicochemical properties, excellent chemical/corrosion resistance, and moderate antibacterial activities. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 117: 1245–1251, 2010

Key words: coatings; FT-IR; polyurethanes; renewable resources; resins

INTRODUCTION

Pongamia glabra is a widely grown, medium-sized deciduous tree, found mainly along coasts and riverbanks. Generally, the tree produces oil-rich seeds (30–40% oil content) twice in a year.¹ The oil is thick, viscous, yellow–orange to brown in color, and nonedible and nondrying in nature possesses about 71% oleic acid.^{1,2} It has found application in the fields of medicine, leather tanning, soap industry, biodiesel, and polymeric materials.^{3–6}

India has the potential to produce over 150,000 tons of *P. glabra* seeds and about 50,000 tons of its oil.¹ Out of this, hardly one-fourth is used, whereas the rest goes to waste.^{1,2} The application of this oil in the synthesis of low-molecular-weight polymers will provide an opportunity to use it as an alternative source of energy because it is expected that petroleum-based resources will be exhausted by the end of the 21st century.^{7,8} Furthermore, an increase in the prices of petro-based chemicals has forced sci-

entists and technologists to study the application of oils in the synthesis of various polymers, such as epoxies, polyols, alkyds, polyesteramides, and poly(urethane–amide)s.^{9–18}

Poly(urethane–fatty amide) (PUPGFA) is a class of polymer that has both urethane and amide linkages in the same polymeric chain; hence, it possesses the synergistics of both. The incorporation of urethane groups in the polymeric chain enhances the abrasion resistance, toughness, and ability for outdoor services along with the chemical resistance;^{18–20} the polymer can then be used as an elastomer, adhesive, thermoset, or high-performance coating.^{18,21–23} The incorporation of amide groups in the polymeric chain enhances the thermal stability, alkali resistance, and tensile strength.^{24,25} In view of this, we report the synthesis, characterization, and anticorrosive and antibacterial properties of *P. glabra* oil based PUPGFA by urethanation of *N,N*-bis(2-hydroxyethyl) *P. glabra* fatty amide (HEPGFA). The anticorrosive properties of the PUPGFA coatings were also compared with those of *P. glabra* oil based baked polyesteramide (PGPEA).¹

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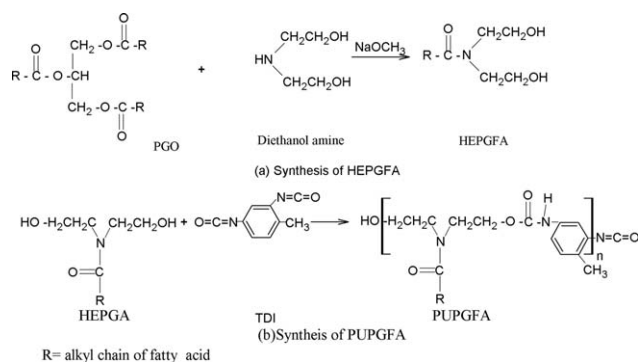
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EXPERIMENTAL

Materials

Oil was extracted from *P. glabra* seeds (procured from a local market) through a Soxhlet apparatus with petroleum ether (boiling range = 60–80°C) as a solvent. The fatty acid composition of the oil was



Scheme 1 Synthesis of PUPGFA from *P. glabra* oil.

determined by gas chromatography (111/8 s.s. column, FID detector) (Perkin-Elmer 8700 Gas Chromatograph, Shelton, CT).¹ Diethyl ether and tolylene-2,4-diisocyanate (TDI; Merck, Mumbai, India) and diethanol amine and xylene (S. D. Fine Chemical, Mumbai, India) were analytical grade.

Synthesis of HEPGFA

HEPGFA was synthesized according to our previously reported method.¹

Synthesis of PUPGFA

PUPGFA was prepared with a one-shot technique (a single-stage process). HEPGFA (1.0 mol) and TDI (1.0–1.5 mol, solution in 8–10% xylene solvent) were placed in a four-necked, flat-bottom flask fitted with a dropping funnel, a thermometer, and a condenser (Scheme 1). The reaction mixture was continuously

stirred under a nitrogen environment at room temperature. The progress of the reaction was monitored by thin-layer chromatography and also by the determination of the hydroxyl values at regular intervals of time. When it reached the desired hydroxyl value, the reaction was stopped. Xylene was removed through a rotary vacuum evaporator (PSAW, Ambala Cantt, India) to obtain the pure PUPGFA resin with a reddish brown color.

Characterization

The structural elucidation of PUPGFA was carried out by Fourier transform infrared (FTIR), ¹H-NMR and ¹³C-NMR spectroscopic techniques. FTIR spectra of these polymers were taken on a PerkinElmer 1750 FTIR spectrophotometer (PerkinElmer Instruments, Norwalk, CT) with an NaCl cell. ¹H-NMR and ¹³C-NMR spectra were recorded on a JEOL GSX 300 MHz FX-1000 spectrometer (Switzerland) with deuterated chloroform (CDCl₃) as the solvent and tetramethylsilane as an internal standard. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were performed with a PerkinElmer Diamond analyzer (Shelton, CT) from ambient temperature to 750°C under a nitrogen atmosphere at a 10°C/min flow rate. Differential scanning calorimetry (DSC) was done with a DSC-10 instrument (TA Instruments, USA) from 30 to 300°C under a nitrogen atmosphere at a 10°C/min flow rate. We tested the solubility of the resin in various polar and non-polar solvents by taking 50 mg of resin in 10 mL of different solvents in a closed test tube and setting it

TABLE I
Experimental Data

Resin code	PGO	HEPGA	PUPGFA-1.0	PUPGFA-1.2	PUPGFA-1.5
Physicochemical characterization					
Hydroxyl value (%)	0.20	8.3	5.9	4.04	1.42
Iodine value (g of I ₂ /100 g)	80	44	26.5	24.0	22.0
Specific gravity (g/mL)	0.8652	0.8873	0.91273	0.91285	0.91305
Inherent viscosity (dL/dg)	—	—	0.756	0.763	0.801
Refractive index	1.471	1.475	1.504	1.506	1.508
Physicomechanical characterization					
Gloss at 45°			43	46	44
Scratch hardness (g)			500	1900	700
Impact resistance (150 lb/in.)			Pass	Pass	Pass
Bending (1/8 in.)			Pass	Pass	Pass
Drying time (min)			15	12	10
Coating thickness (μm)			102	106	108
Chemical resistance tests					
5% NaOH (6 days)			C	D	C
5% HCl (5 days)			B	D	C
3.5% NaCl (10 days)			B	D	B
Xylene (15 days)			E	E	E
Water (15 days)			E	E	E

A, Film completely removed; B, Film cracked and partially removed; C, Loss in gloss; D, Slight loss in gloss; E, film unaffected.

PGO, *Pongamia glabra* oil.

aside for a day. The iodine value, hydroxyl value, inherent viscosity, and refractive index were determined by standard laboratory methods. The experimental data given in Table I are the averages of these parameters with a 0.5% error.

Preparation and testing of the coatings

Coatings of the PUPGFA resin were prepared by a brush technique with a solution containing 50 wt % resin in xylene on mild steel strips. Physicomechanical tests were performed on $70 \times 25 \times 1$ mm³ strips. The specular gloss was determined at 45° by a gloss meter (model RSPT 20; Digital instrument, Santa Barbara, CA); the scratch hardness (BS 3900), bending (ASTM D 3281-84), and impact resistance (IS: 101 part-5/sec.3, 1988) were determined by standard methods. An elcometer (model 345; Elcometer Instruments, Manchester, England) was used to measure the coating thickness. We performed corrosion-resistance tests on $25 \times 25 \times 1$ mm³ coated mild steel strips by dipping them in water, acid (5 wt % HCl), alkali (5 wt % NaOH), and xylene. Periodic examination was conducted until the coating showed evidence of softening or deterioration. Salt spray tests in 3.5 wt % NaCl solution were carried out as per ASTM B 177-94.

Antibacterial activity

The antibacterial activities of HEPGFA and PUPGFA were tested against *Staphylococcus aureus* (Gram-positive) and *Salmonella* (Gram-negative) bacteria. The antimicrobial activities were evaluated with respect to Amekasin (amino glycoside) as a standard controlled drug in the nutrient by the agar diffusion method.²⁶ One loopful of bacteria was inoculated in 10 mL of nutrient broth (peptone = 5 g/L, pH 6.8) and incubated at 37°C for 28–30 h in a test-tube shaker at 100 rpm. The actively growing bacterial cells were used for inhibition studies. The nutrient agars (20 mL) with different percentages of PUPGFA and HEPGFA were poured in to sterile Petri dishes and allowed to solidify at room temperature. After the solidification, 0.1 mL of the bacterial culture was spread on the nutrient agar. A circular well (diameter = 9 mm) was made with a sterilized steel borer. Then, 0.1 mL of solution of the said resin in xylene (xylene does not show any antibacterial activity against the aforementioned bacteria)²⁷ was poured into the well and incubated at 37°C for 24 h. After incubation, the zone of inhibition was measured in millimeters.

RESULTS AND DISCUSSION

Synthesis

HEPGFA was synthesized by the aminolysis of *P. glabra* oil with diethanolamine in the presence of

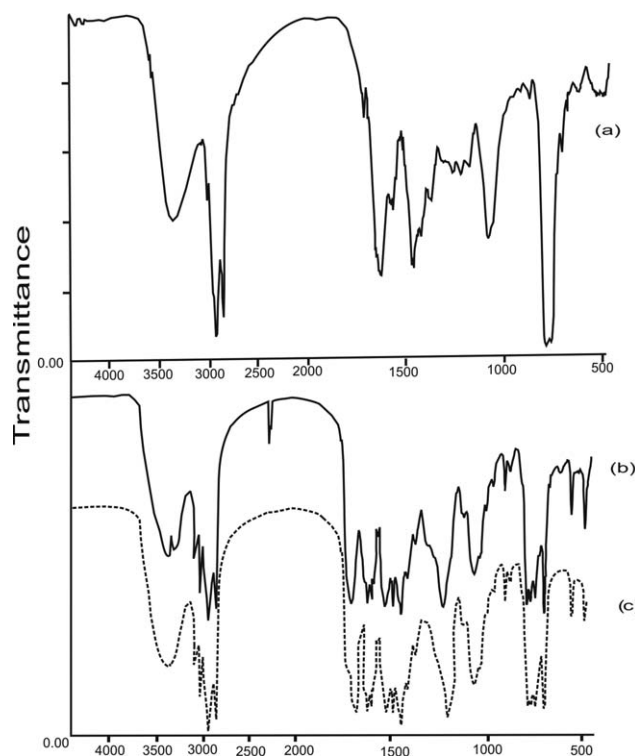


Figure 1 FTIR spectra of (a) HEPGFA, (b) PUPGFA-1.2 resin (before curing), and (c) PUPGFA-1.2 (after curing).

sodium methoxide, which acted as a catalyst. The reaction was carried out via a base-catalyzed nucleophilic acyl substitution bimolecular (S_N2) reaction mechanism.²⁸ The PUPGFA resin was synthesized by the addition polymerization reaction of $-OH$ groups of the HEPGFA with $-NCO$ groups of TDI.¹⁸ To obtain the optimum composition of PUPGFA, TDI was used in different molar ratios. Beyond a 1.5-mol TDI loading, a lumpy aggregate was formed. This was correlated to the reaction of the isocyanate groups of TDI with the hydroxyl groups of HEPGFA to form the linear chain of urethane (primary reaction); furthermore, the hydrogen of the urethane linkage reacted with the isocyanate group of TDI to form allophanate (secondary reaction). This led to the formation of a higher cross-linked network structure in the form of a lumpy aggregate.¹⁰

Spectral analysis of PUPGFA

The formation of urethane linkages and hydrogen bonding in PUPGFA was confirmed by comparison of the FTIR and ¹H-NMR spectra of PUPGFA with the reported spectra of HEPGFA.¹ The FTIR spectra of PUPGFA (Fig. 1) showed characteristic absorption bands at 3338 cm⁻¹ (NH stretching of urethane linkages), 3296 cm⁻¹ (residual hydroxyl groups of the resin), 1715.3 cm⁻¹ ($\text{C}=\text{O}$, urethane groups), 1620.5 cm⁻¹ ($\text{C}=\text{O}$ amide groups), 1229.5 cm⁻¹

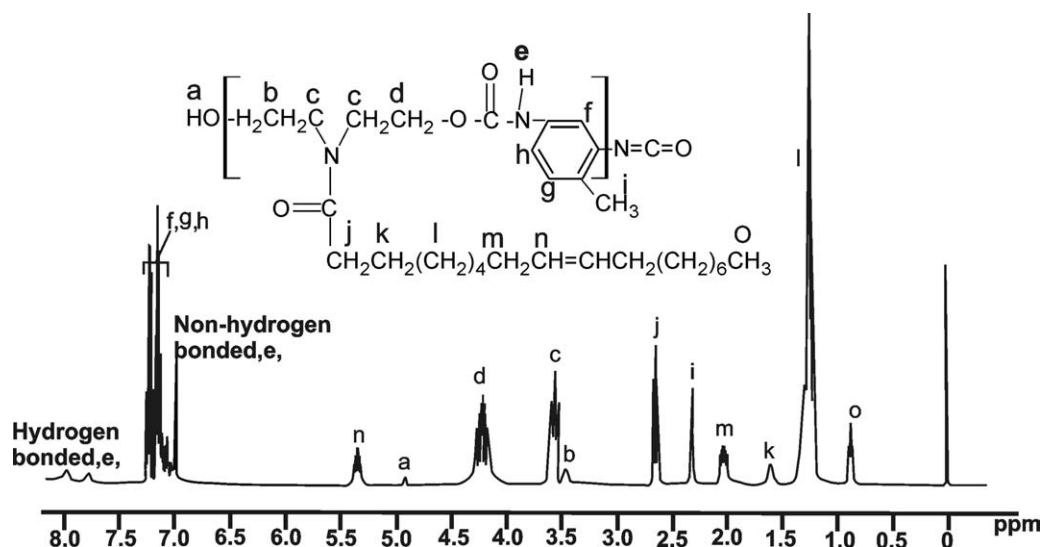


Figure 2 ^1H -NMR spectrum of PUPGFA.

(C–N stretching, urethane groups), 1495.8 cm^{-1} (–NH bending, urethane), 615.8 cm^{-1} (–NH deformation), 697.5 cm^{-1} (–NH wagging, urethane), 2271 cm^{-1} (free –NCO groups), 3085.3 cm^{-1} (ArC=C–H stretching), and 1605 , 1538.2 , and 787.9 cm^{-1} (ArC=C stretching and bending). The presence of these peaks led to the formation of urethane linkages in the resin. The ^1H -NMR spectra of PUPGFA (Fig. 2) showed low-intensity peaks at 4.8 and 3.6 ppm for residual –OH groups. –CH₂ attached to those –OH groups confirmed the consumption of –OH groups in the formation of urethane, and the appearance of a peak at $\delta = 4.3\text{ ppm}$ for –CH₂ attached to the urethane groups further confirmed the reaction of the –NCO groups of TDI with the –OH groups

of HEPGFA. Additional characteristic peaks were observed at 2.23 ppm for –CH₃ of TDI and 7.12–7.06 ppm for other ring protons. The ^{13}C -NMR spectra (Fig. 3) showed peaks at 67 and 134 ppm for –CH₂ groups and aromatic rings attached to urethane groups, whereas there were peaks at 144 and 155 ppm for carbonyls of urethane and free NCO groups. The other aromatic ring carbon peaks were observed at 136, 137, 125.7, 126.5, and 116.2 ppm, whereas a peak at 17.0 ppm appeared for –CH₃ of TDI.

Close examination of the spectra of HEPGFA and PUPGFA showed a slight lower field shifting of the amide carbonyl band from 1622.4 cm^{-1} (HEPGFA) to 1620.50 cm^{-1} (PUPGFA) and C–N stretching,

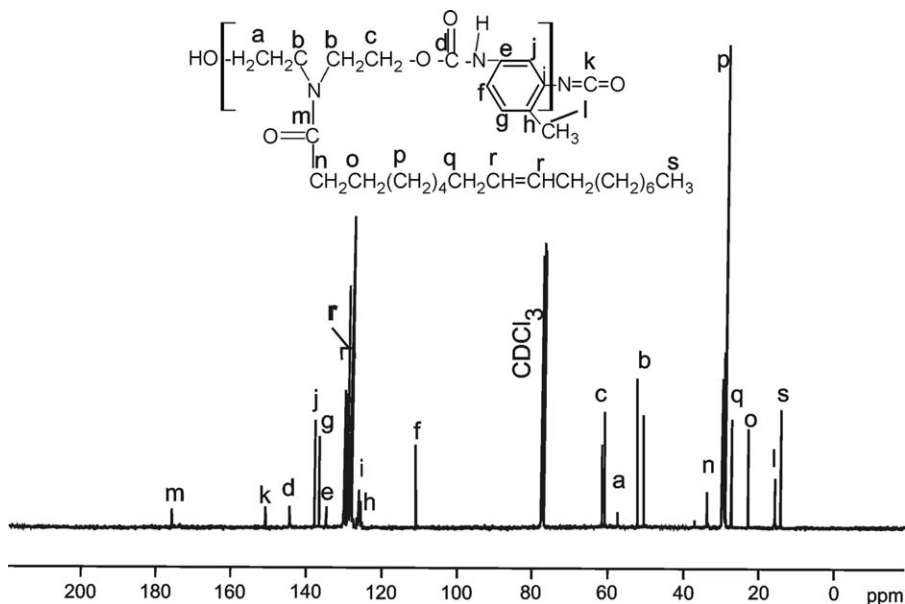


Figure 3 ^{13}C -NMR spectrum of PUPGFA.

with the amide band from 1450 cm^{-1} (HEPGFA) to 1455 cm^{-1} (PUPGFA). This slight shifting of the band was correlated to the presence of hydrogen bonding between the oxygen atom of the carbonyl and the hydrogen atom of the NH group in PUPGFA.³⁰ It was further observed that a broad —OH band was present in PUPGFA at 3354.3 cm^{-1} , whereas the same band was observed at 3296 cm^{-1} in HEPGFA. The occurrence of peaks in $^1\text{H-NMR}$ of PUPGFA at 7.1–6.9 and 7.99–7.82 ppm for non-hydrogen-bonded and hydrogen-bonded —NH groups of the urethane further supported the presence of hydrogen bonding and the formation of urethane linkages in the system.²⁹

Physicochemical characterization

Physicochemical characterization (Table I) indicated that the decrease in the hydroxyl value from HEPGFA to PUPGFA was correlated to the reaction of the —OH groups of HEPGFA with the —NCO groups of TDI, whereas the decrease in the iodine value and increase in the values of specific gravity, refractive index, and inherent viscosity were correlated with the increase in the molar mass of PUPGFA. We also observed with increased loading of TDI in PUPGFA further increases in the specific gravity, refractive index, and inherent viscosity and a decrease in the iodine value (Table I).

Solubility

The solubility of the PUPGFA resin was tested in various solvents. The resin was completely soluble (80–100%) in xylene, CDCl_3 , carbon tetrachloride, dimethylformamide, dimethyl sulfoxide, tetrahydrofuran, ethyl methyl ketone, and ether; slightly soluble (20–40%) in methyl alcohol, and ethyl alcohol; and almost insoluble in water. The presence of long fatty acid hydrocarbon chains and polar groups in the resin was responsible for the solubility trend.²⁷

Curing of PUPGFA

The two-stage room-temperature (28°C for 15–10 min) curing of the PUPGFA coatings was observed. During the first stage, solvent evaporation occurred, which was a physical phenomenon, followed by the second-stage curing, which was a chemical process.¹⁰ In the later process, free —NCO groups of the resin reacted with the residual —OH groups of the other chain of the resin along with the atmospheric moisture to form urethane and amino groups through an addition reaction mechanism. These groups further reacted with the —NCO groups present in another molecule of the resin to form a cross-linked structure.¹⁰ An important electrostatic process

of interaction also took place through hydrogen bonding; this resulted in further crosslinking of the resin. The possibility of the involvement of residual unsaturation in the curing reactions was negligible as *P. glabra* oil possessed only one double bond, which was insufficient for autoxidation.³⁰ The comparison of the FTIR spectra of the uncured resin [Fig. 1(b)] and cured resin [Fig. 1(c)] also helped to explain the curing mechanism of the coating. Characteristic peaks at 3338, 3296, and 2271 cm^{-1} were observed in the uncured resin that could be ascribed to —NH urethanes, residual —OH groups, and free —NCO groups, respectively, although in the cured resin, only a broad peak of —NH appeared and the other two peaks completely disappeared. A shifting in the carbonyl and —C—N stretching peaks of the urethane groups was also observed from 1715 to 1705 cm^{-1} (-10 cm^{-1}) and 1229.5 to 1216 cm^{-1} (-13.5 cm^{-1}), respectively, which led to the formation of hydrogen bonding between the —NH of urethane and >C=O groups. The shifting in these peaks and the disappearance of the —OH and —NCO peaks in the cured resin confirmed the involvement of these groups in the curing reaction of the resin.²⁹ Close examination of the spectra showed that the peak at 3008 cm^{-1} of —C=C—H stretching (residual unsaturation) remained present in both spectra, which further confirmed that the curing did not occur through residual unsaturation.

Coating properties

To determine the optimum composition for the coating that provided the best performance of the PUPGFA coatings, coatings of different compositions were prepared with different loadings of TDI (1.0–1.5 mol). Coating compositions with less than 1.0 mol of TDI became soft even after 1 week; this was attributed to the insufficient crosslinking and curing of the film. Although coating compositions beyond 1.5 mol of TDI became brittle and showed cracks after a few minutes, this was correlated to excessive crosslinking. Therefore, the composition of the resin was optimized by loading of TDI from 1.0 to 1.5 mol, that is, 1.0, 1.2, and 1.5 mol of TDI. The coatings performances in terms of the physicomechanical properties and chemical/corrosion resistance of these three compositions are given in Table I. The thickness and gloss (at 45°) of the coatings were found to be 102–108 μm and 43–46, respectively. The maximum value of scratch hardness for the PUPGFA was exhibited by the coatings with a 1.2-mol loading of TDI, whereas the lowest value was observed with a 1.0-mol loading of TDI. This was correlated to the development of maximum adhesion between the coating material and the surface of the specimen through the uniform distribution of

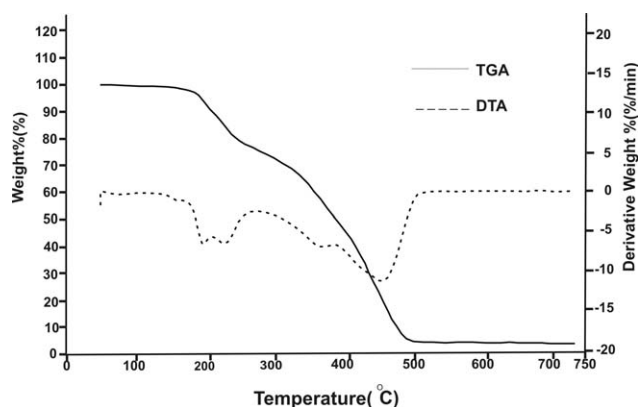


Figure 4 TGA and DTA thermograms of PUPGFA-1.2.

pendent polar groups present in the polymeric chain of the resin and to the optimum crosslink density in case of the 1.2-mol loading of TDI. Beyond this loading, excessive crosslinking and entanglement of the polymeric chains may have occurred, which produced an internal strain in the film that may have been responsible for the deterioration of the scratch hardness values. All of the coatings systems passed the bending (1/8-in. conical mandrel) test as a characteristic of oil-based polymers and the impact-resistance test (150 Lb/inch), which was associated with the polar nature of PUPGFA linkages, which provided adhesion and toughness to the coatings.¹⁸ Like those of the physicochemical properties, the chemical/corrosion-resistance tests (Table I) also revealed PUPGFA-1.2 resin with 1.2-mol loading of TDI showed best performance among the three compositions, as they remained unaffected even after 15 days of immersion in xylene and water, whereas they showed slight loss in gloss after 10 days in the salt spray test in NaCl solution. When immersed in 5% NaOH and 5% HCl, the resin revealed a slight loss in gloss after 6 and 5 days, respectively. This was attributed to the homogeneous, uniform, and well-adhered crosslinked structure of the coatings, which prevented the penetration of corrosive ions into the coating framework. We also observed that the PUPGFA coating systems showed superior alkali-resistance performance as compared to that of the PGPEA baked coatings;¹ this was due to the presence of urethane groups in the PUPGFA coatings, which are less hydrolyzable in nature than the ester groups present in the PGPEA coating system.¹

Thermal analysis

Figure 4 shows the TGA thermogram of PUPGFA-1.2. In the thermogram, two onsets of two degradation temperatures were observed, first, at 177°C and,

then, at 357°C. The 5% weight loss occurring at 200°C was attributed to entrapped solvent and moisture. The degradation at 225°C corresponded to 10% weight loss and may have been due to the cleavage of urethane linkages, which depended on the type of substituents present on the isocyanate and polyol sides.³¹ The 50 and 80% weight losses occurred at 390 and 455°C, respectively, and could be correlated to the degradation of the aromatic ring and aliphatic alkyl group chain of the fatty acid, respectively. The DTA thermogram of PUPGFA (Fig. 4) showed five endotherms. The first endotherm at 160°C was due to the restructuring of segments, which were considered to be the glass-transition temperature (T_g) of PUPGFA.³² The second and third endotherms were observed at 205 and 225°C and could be attributed to the melting of the polymer and the breaking of urethane bonds, respectively. The last two endotherms were observed at 370 and 460°C and could be associated with the dissociation of the amide linkages and hydrocarbon chain, respectively.³² We observed that most of the decomposition temperature of TGA matched that of the DTA endotherm's temperature. The DSC thermogram of PUPGFA (Fig. 5) revealed that the T_g of the same was between 160 and 170°C. This value also coincided with the DTA of the same polymer. The high value of T_g was correlated with the presence of a pendent fatty acid chain, a polar group, and hydrogen bonding in the polymeric chain.³¹ The thermogram also showed endothermic peaks ranging from 40 to 190°C (centered at 90.58°C), 175 to 190°C (centered at 184°C), and 210 to 230°C (centered at 225°C) followed by the decomposition of the resin; the former peak (40–190°C) was related to the onset of curing by urethane linkages, which underwent curing at ambient temperature.^{8,10} This led to configurational changes in the polymeric chains. The second and third endotherms were correlated to the melting of polyurethane and polyamide or second-stage curing.³¹ These endotherms matched the DTA of the same.

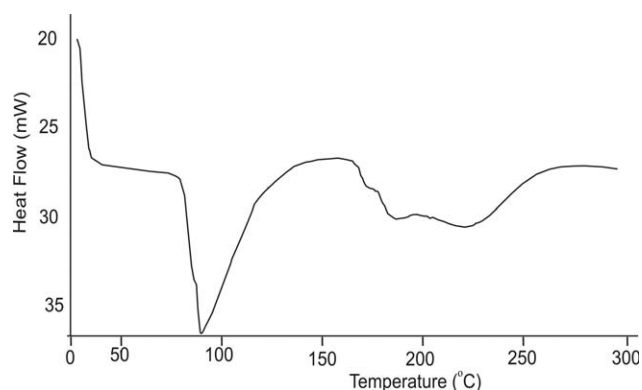


Figure 5 DSC thermogram of PUPGFA-1.2.

Antibacterial activity

The antibacterial results reveal that HEPGFA did not show antibacterial activity against *S. aureus* or *Salmonella*, whereas PUPGFA showed a moderate effect (inhibition zone = 18 mm) only against *Salmonella*. The antibacterial activity of PUPGFA was correlated to the presence of urethane and free —NCO groups. The cell wall of *Salmonella* is composed of a thin layer of peptidoglycon, which is not protected by teichoic acid, as in the case of Gram-positive bacteria. Therefore, the —NCO and —OCONH groups of PUPGFA interacted with —NH and —OH groups of peptidoglycon, which ultimately resulted in the formation of a crosslinked structure, which led to the degradation of the bacterial cell wall.²⁶

CONCLUSIONS

Nonedible and nondrying *P. glabra* oil can be successfully used for the preparation of room-temperature-cured PUPGFA resin through a one-shot technique. The PUPGFA-1.2 system showed good physicomechanical properties and excellent chemical/corrosion resistance and moderate antibacterial performance. The resin showed superior alkali-resistance performance compared to the baked coating of PGPEA. Thus, this resin could safely be used up to 200°C as a protective eco-friendly coating material.

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