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Alumina-Incorporated Polyesteramide from Non-Edible Seed Oils

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To improve the physico-mechanical and chemical resistance properties, lower the curing temperature of annona squamosa and pongamia glabra seeds oils based polyesteramides [ASPEA, PGPEA], as well as to convert the non-edible seed oils into value added products, their respective alumina-incorporated polyesteramides resins [Al-ASPEA, Al-PGPEA] have been synthesized. The resins and their coatings have been tested for their chemical, physico mechanical and chemical/corrosion resistance properties. These properties were compared among the prepared resins and with that of previously reported alumina filled linseed polyesteramide [Al-LPEA]. It was observed that Al-PGPEA-71, which has the highest amount of oleic acid chains, shows the best physico-mechanical and chemical resistance properties.

Keywords non-edible seed oils, alumina-incorporated polyesteramide, coating, corrosion

Introduction

India possesses large forest reserves and farmlands, which have a large variety of edible and non-edible oil bearing seeds, and more than 140 varieties of such seeds have been reported in India. The total production of oil from these seeds is about 30 tons/year (1, 2). However, only 7% of the total production of such seeds are being used in industrial application (1–3). Common seed oils such as linseed, castor, soybean, safflower and sunflower have been used in the synthesis of low molecular weight polymers like alkyds, epoxies, polyurethanes, polyetheramides, polyesteramides, etc. (4–13). Many of these polymers are used extensively in the field of paints and coatings (4, 7, 9–11) (14–16). The meager utilization of non-edible seed oils has prompted the scientist and technologist to develop ways to fully utilize such seed oils (3, 17–21).

Annona squamosa [AS] and *pongamia glabra* [PG] are widely grown trees in India. Their seeds contain 15–40% oil, which possesses a large amount of unsaturated fatty acids (2, 3, 17, 19). *Annona squamosa* is a small, and more or less, evergreen tree

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mainly growing wild in the western part of India. It is now cultivated in most parts of the country (3, 19). *Pongamia glabra* is a shady, medium sized tree, mainly found in coastal areas, tidal forest, rivers and roadsides. Only a small amount of the total production of pongamia seed oil is being used in the soap, leather tanning and lubricant industries (17). *Annona squamosa* and pongamia seeds oils [ASO, PGO] hold potential for the synthesis of low molecular weight polymers like epoxies and polyesteramide (3, 17, 19, 22).

Alkyds are a value added product from seed oils and are well known for surface coating applications (1, 23–25). Oil based polyesteramides are amide modified alkyds that have improved film characteristics over alkyds in terms of drying time, hardness, water vapor resistance, chemical and thermal resistance, as well as durability and other physicochemical properties (26, 27). But their application as coating materials is limited due to their high melting point and high curing temperature (10). It is reported that the incorporation of metals/metalloids (26) and organic moieties like styrene (28, 29), vinyl acetate (28), urethane (30), poly(styrene-co-maleic anhydride) (10, 31) to a polyesteramide reduces the drying time and curing temperature, as well as enhances the thermal stability, scratch hardness and protective efficiency. Earlier, we have incorporated alumina in the backbone of linseed polyesteramide, which has resulted in the improvement in curing temperature, curing time, physico-mechanical characteristics and anticorrosive properties of the resin (32). Since different seed oils have different fatty acids in different amounts, they have a different extent of unsaturation at different positions in the fatty acid chain (33). They are expected to yield polyesteramide resin with different physico-mechanical and anti-corrosive properties (17). *Pongamia glabra* seeds oil (PGO) and *annona squamosa* seed oil (ASO) differ in the type and extent of unsaturation from the linseed oil (LO) (17, 19, 32). However, the former two seed oils are non-edible (3). We therefore, consider it worthwhile to investigate the drying time, curing temperature, thermal stability, physico-mechanical and chemical/corrosion resistance properties of alumina incorporated *pongamia glabra* and *annona squamosa* polyesteramides (Al-PGPEA and Al-ASPEA) to explore a non-edible oil based coating resin, which would hold potential as a candidate for commercial application. A literature survey reveals that no work has yet been reported on this aspect of alumina-incorporated polyesteramides from non-edible oil bearing seeds (17, 19, 30, 32, 34).

In this paper, we report the synthesis, physico-mechanical and physico-chemical properties, protective efficiency and spectroscopic characterization of alumina-incorporated polyesteramides of ASO and PGO with 46% and 71% oleic acid, respectively. We have also compared their properties and with that of linseed oil (22% oleic acid) based alumina-incorporated polyesteramide [Al-LPEA] (33) for the best performing resin as a candidate for coating material from a sustainable resource.

Experimental

Materials

Oils were extracted from *annona squamosa* seed [ASO] and *pongamia glabra* seeds [PGO] (procured from Rajdhani Nursery, Dehradun, India) through a Soxhlet apparatus, using petroleum ether (boiling point range 60–80°C) as a solvent. The fatty acid compositions of these oils were determined by GC (111/8 s.s column. FID detector, Table 2) (17, 19). Phthalic acid, sodium methoxide and aluminium hydroxide [Al(OH)₃] (Merck,

India), diethanol amine and petroleum ether (s.d. Fine Chemicals, India) were of analytical grade.

Synthesis of HEASA and HEPGA

N, N-bis (2-hydroxyethyl) fatty amides of ASO and PGO [HEASA and HEPGA] were synthesized by using a reported method (17, 19).

Synthesis of Annona Squamosa Polyesteramide [ASPEA] and Pongamia Glabra Polyesteramide [PGPEA]

Polyesteramides of ASO and PGO have been synthesized by using our previously reported method (17, 19). The equimolar amount of HEASA/HEPGA, phthalic acid and 50 ml xylene (solvent) were taken in a four-necked round bottom flask fitted with a Dean Stark trap, nitrogen inlet tube, thermometer, and mechanical stirrer. The reaction mixture was heated at $145 \pm 5^\circ\text{C}$ under continuous stirring. TLC (Thin Layer Chromatography) and acid value were used to monitor the progress of the reaction. A calculated amount of water was collected in a Dean Stark trap to obtain the desired acid value. On obtaining the desired acid value, the reaction was stopped and the reaction product was cooled. The xylene was removed from the reaction mixture by a vacuum rotary apparatus under reduced pressure to obtain polyesteramide [ASPEA/PGPEA].

Synthesis of Alumina Incorporated Polyesteramide from ASPEA/PGPEA [Al-ASPEA/Al-PGPEA]

Alumina was incorporated in ASPEA/PGPEA through $\text{Al}(\text{OH})_3$. The above setup was used for the synthesis of Al-ASPEA/Al-PGPEA. The mixture of $\text{Al}(\text{OH})_3$ (0.006 mole) (29) and ASPEA/PGPEA (0.05 mole) in 50 ml xylene was heated at the rate of $10^\circ\text{C}/\text{min}$ up to 60°C . This temperature was maintained for one hour, followed by an increase in temperature up to $145 \pm 5^\circ\text{C}$ at the rate of $5^\circ\text{C}/\text{min}$ and was further heated for 2 h. TLC confirmed the formation of an end product. After the completion of reaction, the end product was cooled and diluted in ether, then washed with 15 wt% aqueous NaCl solution. After washing, the product was dried over anhydrous sodium sulphate and the solvent was removed over a rotary vacuum evaporator under reduced pressure to obtain the pure Al-ASPEA/Al-PGPEA.

Preparation and Testing of Coatings

Coatings of ASPEA/PGPEA and Al-ASPEA/Al-PGPEA resins were prepared by a brush technique using a solution containing 50 wt% of resins in xylene on mild steel strips, $30 \times 10 \times 1$ mm size, for the corrosion resistance test and $70 \times 25 \times 1$ mm size for a physico-mechanical test. The specular gloss was determined at 60° by a gloss meter (model RSPT 20; Digital Instrument, Santa Barbara, CA). Scratch hardness (BS 3900), bending characteristic (ASTM-D3281-84), abrasion resistance (ASTM D 4060-90) and impact resistance (IS: 101part5/Sec.31988) were determined by above-mentioned standard methods. Coated strips were baked at 210°C for 20 min, 220°C for 30 min, and 165°C for 15 min for PGPEA, ASPEA, and Al-ASPEA/Al-PGPEA, respectively. Elcometer (Model 345; Elcometer Instrument, Manchester, UK) was used to measure the coating thickness. The thickness of these coatings were found

between $75 \pm 5 \mu\text{m}$. Corrosion tests were performed in water, acid (5 wt% HCl) and alkali (5 wt% NaOH), by pouring their solutions in 3"-diameter porcelain dishes and dipping the coated samples in the aforementioned media. Periodic examination was conducted until the coatings showed evidence of softening or deterioration (Table 2). A salt-spray test (ASTM B177-94) was also carried out for a period of 10 days in a salt mist chamber.

Results and Discussion

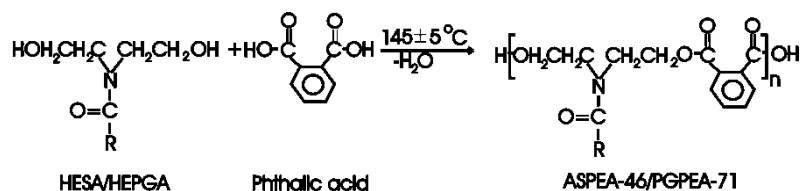
Schemes 1 and 2 illustrate the reaction schemes for the synthesis of polyesteramide and alumina incorporated polyesteramide from annona squamosa and pongamia glabra seeds oils (containing 46% and 71% oleic acid, respectively) carried out by condensation polymerization reaction. The reaction occurs between the hydroxyl groups of the hydroxy fatty amide and carboxyl groups of the phthalic acid resulting into the formation of polyesteramide containing alcoholic hydroxy at the one end and carboxyl hydroxy on the other end of the chain. Alumina was incorporated in the backbone of polyesteramide through a reaction between the carboxyl group of polyesteramide and the hydroxyl group of $\text{Al}(\text{OH})_3$. The presence of alumina, ester and amide bonds in these polymers was confirmed by FTIR (Table 1) and $^1\text{H-NMR}$ spectral analysis (Figure 1). The thermal behavior of these polymers was also studied by TGA and DSC techniques (Figures 2 and 3).

Characterization

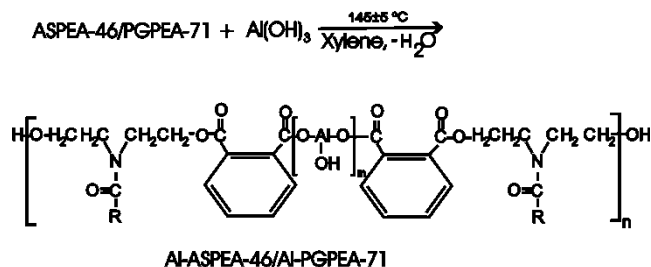
ASPEA/PGPEA and Al-ASPEA/Al-PGPEA were characterized by FT-IR, $^1\text{H-NMR}$, TGA, and DSC. FT-IR spectra of these resins were taken on a Perkin-Elmer 1750 FTIR spectrophotometer (Perkin-Elmer Instruments, Norwalk, CT) using a NaCl cell. $^1\text{H-NMR}$ spectra were recorded on a JEOL GSX 300 MHz FX-1000 spectrometer using deuterated chloroform as a solvent, and tetramethylsilane (TMS) as an internal standard. The thermal stability and curing behavior were studied, respectively by TGA (thermo gravimetric analyzer, TA-57, Instruments USA) and DSC (910 Dupont) in Nitrogen atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$. Iodine value (ASTM D555-6), hydroxyl value, saponification value, specific gravity and refractive index were also determined by standard laboratory methods (Table 2).

Spectral Analysis

The broad structures of HEASA, HEPGA, ASPEA, and PGPEA established by IR and $^1\text{H-NMR}$ spectral analyses have already been reported in our earlier work (17, 19). The



Scheme 1. Synthesis of ASPEA-46/PGPEA-71.



Scheme 2. Synthesis of Al-ASPEA-46/Al-PGPEA-71 and R=Alkyl chain of fatty acid of oil.

structure of Al-ASPEA/Al-PGPEA is confirmed by the presence of characteristic peaks of FT-IR (Table 1) and $^1\text{H-NMR}$ spectra (Figure 2). Table 1 reveals shifting in some of the absorption bands, especially $>\text{C}=\text{O}$ ester (12 cm^{-1}) and $>\text{C}=\text{O}$ amide (6 cm^{-1}) and enhancement in the spread of $-\text{OH}$ band ($3650 \text{ cm}^{-1} - 3120 \text{ cm}^{-1}$) on the incorporation of alumina in ASPEA/PGPEA.

$^1\text{H-NMR}$ spectra (Figure 1) shows peaks of aromatic region at $\delta = 7.2 - 7.8 \text{ ppm}$ in ASPEA/PGPEA, whereas in Al-ASPEA/Al-PGPEA, the peak for aromatic moiety is shifted downfield at $\delta = 7.6 - 8.3 \text{ ppm}$. The peak at $\delta = 8.5 \text{ ppm}$ is assigned to carboxyl proton of ASPEA/PGPEA ($-\text{COOH}$) which is absent in the case of Al-ASPEA/Al-PGPEA (Figure 1).

Table 1
FT-IR spectral assignment of polyesteramide and alumina incorporated polyesteramide

Functional groups	FT-IR assignment (cm^{-1})	
	ASPEA-46/ PGPEA-71	Al-ASPEA-46/ Al-PGPEA-71
$-\text{OH}$	3400–3500	3650–3120
$>\text{C}=\text{O}$ (ester)	1726–1734	1738–1746
$-\text{C}-\text{C}(=\text{O})-\text{O}$ str.	1273–1280, 1170–1175, 1140–1145	1280–1285, 1176–1180, 1141–1147
$>\text{C}=\text{O}$ (amide)	1646–1657	1652–1663
$\text{C}-\text{N}$	1135	1137
$\text{CH}_{2\text{sym}}$	2854–2872	2856–2874
$\text{CH}_{2\text{asym}}$	2920–2938	2930
$\text{C}=\text{C}-\text{H}$ str.	3008	3009
Ar $\text{C}=\text{C}-\text{H}$ str.	3075–3085	3077–3087
Ar $\text{C}=\text{C}$	1610–1615, 1588–1590, 750	1614–1619, 1590–1592, 750, 780

Ar- Aromatic.

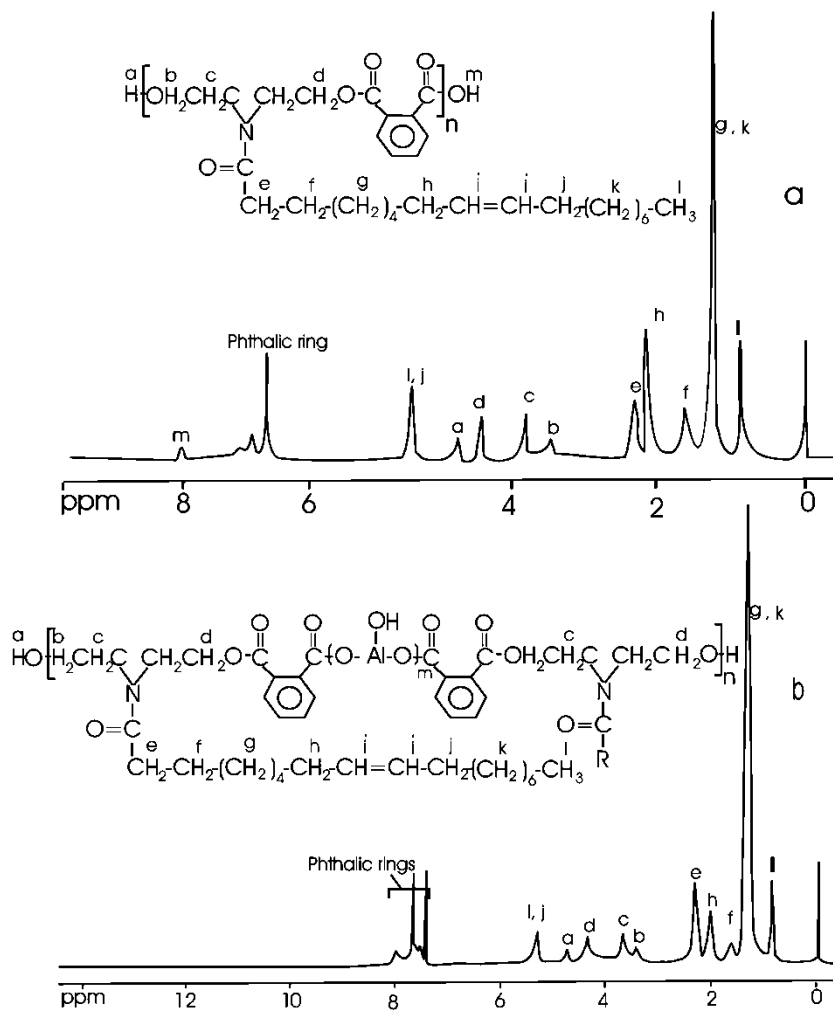


Figure 1. $^1\text{H-NMR}$ spectra of a-ASPEA-46/PGPEA-71, and b-Al-ASPEA-46/Al-PGPEA-71.

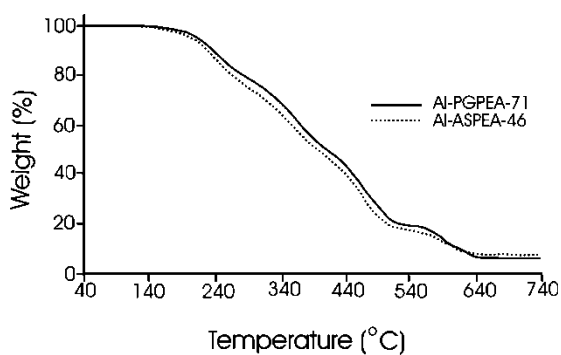


Figure 2. TGA thermogram.

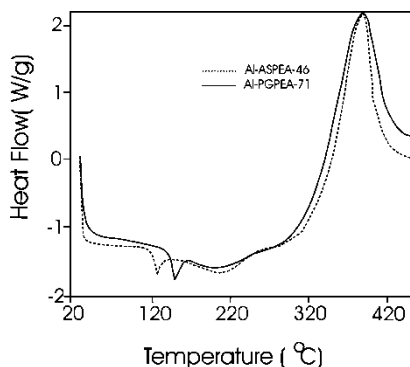


Figure 3. DSC thermogram.

Physico-chemical Characterization

Table 2 shows the values of physico-chemical properties of ASPEA, PGPEA, Al-LPEA, Al-ASPEA, and Al-PGPEA. We notice that with the incorporation of alumina in the polyesteramide, there is a decrease in the saponification and iodine values and an increase in specific gravity, refractive index, and hydroxyl value. This can be attributed to the increases in molar mass of Al-ASPEA/Al-PGPEA. The increase in hydroxyl values from ASPEA/PGPEA to Al-ASPEA/Al-PGPEA is due to the presence of a pendant hydroxyl group attached to aluminum along with terminal hydroxyl of the chain (Scheme 2). With increasing oleic acid contents, the increase in the refractive index is observed which indicates an enhanced linear and dense structure of the resin.

The solubility of polymers has been tested in different solvent, viz., chloroform, methanol, ethanol, water, formaldehyde, DMF (dimethyl formamide), DMSO (dimethyl sulphoxide), toluene, xylene, diethyl ether, acetone, and acetic acid. ASPEA and PGPEA exhibit higher solubility in chloroform, toluene, xylene, diethyl ether, and DMF. In the remaining solvents, it is either insoluble or negligibly soluble. Like Al-LPEA resin, the Al-ASPEA and Al-PGPEA resins show solubility in polar, as well as non-polar solvents except in H₂O and formaldehyde. The higher solubility trend of the latter can be attributed to the presence of polar groups and pendant hydroxyl at aluminum in the resins.

Coatings Properties

Coatings of ASPEA, PGPEA, Al-ASPEA, and Al-PGPEA on mild steel strips were made by brush techniques. Coatings were baked at different temperatures (150–250 °C) and time (5–60 min), to obtain the optimum baking temperature and time at which coatings show the best film properties. The optimum baking temperature and time for ASPEA, PGPEA, and LPEA were found to be 220 °C and 30 min, 210 °C and 20 min, and 220 °C and 10 min, respectively, while the same of Al-ASPEA and Al-PGPEA was found to be 165 °C and 15 min (Table 2). The baking temperature of Al-ASPEA/Al-PGPEA was lower than Al-LPEA (200 °C for 10 min). It was found that the presence of alumina in Al-PGPEA, Al-ASPEA, and Al-LPEA coatings reduced the baking temperature and increased the gloss, scratch hardness and impact resistance over those of PGPEA, ASPEA, and LPEA coatings (32). This can be attributed to the presence of alumina and higher oleic acid

Table 2
Physico-chemical, mechanical, and chemical/corrosion resistance properties of ASPEA-46, PGPEA-71, Al-LPEA-22, Al-ASPEA-46, and Al-PGPEA-71

Resin code*	ASPEA-46 ¹⁹	PGPEA-71 ¹⁷	Al-LPEA-22 ³³	Al-ASPEA-46	Al-PGPEA-71
Physico-chemical analysis					
H.V. (%)	6.02	5.08	14.98	13.05	10.64
S.V. (mg KOH/g)	132.00	106.00	118	120.00	96.00
I.V. (g I ₂ /100 g)	25.00 ¹	28.00	20	13.00	15.00
Sp. Gravity (g/ml)	0.958	1.012	0.976	1.086	1.043
Refractive index	1.507	1.524	1.543	1.543	1.560
Physico-mechanical properties					
Baking temp.**°C	220	210	200	165	165
Baking time** (min)	30	20	10	15	15
S.H. (Kg)	3.0	3.5	2.5	4.0	4.5
I.R. (lb/inch)	100	100	150	200	250
Gloss at 60°C	90	90	135	150	175
Bending (1/8 inch)	passes	passes	passes	passes	passes
Abrasion resistance (wt loss in mg after 500 cycle)	0.7	0.7	–	0.3	0.3
Chemical/corrosion resistance					
H ₂ O (10 days)	e	e	e	e	e
NaOH (5%) 2 hrs	a	a	b ²	b	c
HCl (5%) 10 days	d ³	d ³	e	e	e
NaCl (3.5%) 10 days	d ⁴	d ⁵	e	e	e

H.V.-hydroxyl value, S.V.-saponification value, I.V.-iodine value, Sp. gravity-Specific gravity, S.H.-Scratch hardness, I.R.-impact resistance, a-film completely remove; b-film slightly cracked and partially removed; c-loss in gloss; d-slight loss in gloss; e-unaaffected, 1-after revised analysis, 2-after 1 h, 3-after 48 h,4-after 2 h, and 5-after 72 h.

*Last digit indicates wt% oleic acid in the oil. The coating passes adhesion test without showing any visible damage.

**Optimum baking time and temperature, which resulted in best coating properties.

content in the resins. The presence of alumina increases the chain length of the polymeric resin which results in the formation of crosslinked flexible coating materials with good stiffness and higher scratch hardness (32). While Oleic acid chains, due to low unsaturation, contribute higher flexibility to the resin compared to fatty acid with higher unsaturation, e.g. linoleic, linolenic (17). A greater content of linoleic and linolenic fatty acids (LPEA, Al-LPEA) result in higher crosslinking due to higher unsaturation, producing less flexible and more rigid film, which hampers the physico-mechanical properties (Table 2) of the resins. All these effects result in the reduction in gloss and scratch hardness values with the decrease in oleic acid content (Al-PGPEA > Al-ASPEA > Al-LPEA coatings). The higher scratch hardness, flexibility and gloss values for these systems are also characteristic of oil based polymeric systems. The presence of pendant –OH groups on aluminum in the backbone of polymeric chain

would increase the adhesion between polymeric chain and metal surface (32). The same can be attributed to the higher gloss, scratch and impact resistance values, as well as higher chemical/corrosion resistance properties of Al-PGPEA Al-ASPEA Al-LPEA than those of ASPEA, PGPEA, and LPEA coatings.

Like physico-mechanical properties, the presence of alumina in Al-ASPEA, Al-PGPEA, and Al-LPEA resins also enhances the corrosion protective efficiency of their coating in different corrosive environment viz., aqueous, alkaline, acidic, and saline medium over those of ASPEA, PGPEA, LPEA (Table 2). Coatings of Al-ASPEA and Al-PGPEA are unaffected for 10 days in water, HCl (5 wt%) and the salt spray test in NaCl (3.5 wt) while coatings of ASPEA and PGPEA show a slight loss in gloss in HCl (5 wt%) in 48 h, in NaCl (3.5 wt) in 2 h and 72 h, respectively. In alkaline medium (5 wt%) the Al-ASPEA and Al-LPEA show slight cracks and films are partially removed in 2 h, whereas Al-PGPEA exhibits only a loss in gloss during this period and the cracks in film began after this period. These properties can also be correlated to the incorporation of alumina in the backbone of polyesteramide chains, higher flexibility and greater linearity of the chains and optimum crosslinking in the resin with higher oleic acid content. The hydroxyl group on aluminum in the polymer chains is expected to provide good adhesion between the polymeric coating and metal surface (32). Hydroxyl groups on aluminium that are oriented away from the substrate surface will also provide the extra crosslink sites, which can improve the anti-corrosive properties. From the perusal of Table 2, it is revealed that all the performance parameters comprising of physico-mechanical tests, chemical/corrosion resistance tests, gloss, baking time and temperature are the best in the case of Al-PGPEA.

Thermal Analysis

The initial decomposition temperature for PGPEA, ASPEA, Al-PGPEA, and Al-ASPEA (Figure 2) are found to be almost the same, i.e., around 200°C, while 20 wt% loss is observed at 310°C, 305°C, 290°C, and 286°C, respectively. The 50 wt% loss is recorded at 370°C for both PGPEA and ASPEA, while in Al-PGPEA and Al-ASPEA, the same is observed at 395°C and 390°C, respectively. 80 Wt% loss is found at 415°C, 390°C, 514°C, and 520°C in PGPEA, ASPEA, Al-PGPEA, and Al-ASPEA, respectively. Thermal stability of alumina incorporated polyesteramide resins is found to be higher than that of their virgin polyesteramides. This can be attributed to the presence of alumina in the backbone of the resins.

In the DSC thermogram, (Figure 3) we notice that the melting point of PGPEA is higher than ASPEA by 30°C (17, 19). This is attributed to the greater hydrophobic interaction between the neighboring chains in PGPEA, which has the lowest number of multiple double bonds in the fatty acid chains. In the case of Al-ASPEA and Al-PGPEA, the melting point was found to decrease by almost 30°C from ASPEA and PGPEA. Al-PGPEA melts at 150°C and Al-ASPEA at 130°C. The decrease in the melting points of Al-ASPEA and Al-PGPEA resins from PGPEA and ASPEA can be correlated to the comparatively longer molecular chains of the former two resins that will enhance the flexibility of the chains causing lowering of melting in these resins. A shallow endothermic peak is observed in ASPEA in the temperature range 170°C to 260°C, due to the curing reaction occurring in the resin. A similar endothermic peak has been observed in Al-PGPEA and Al-ASPEA in the temperature range 170°C to 255°C and 150°C to 250°C, respectively. TGA thermograms (Figure 2) show the initiation of sluggish decomposition from 210°C, followed by faster decomposition beyond 275°C.

The DSC thermogram of Al-ASPEA and Al-PGPEA (Figure 3) also exhibits similar decomposition events around 255°C. From these observations, it can be concluded that a slight weight loss is accompanied along with curing of the resin that can be attributed to the evaporation of entrapped solvent during the high temperature curing of the resins. The TGA thermogram (Figure 2) shows a similar trend of decomposition of both PGPEA and ASPEA, and Al-PGPEA and Al-ASPEA.

Conclusions

It can be finally concluded that alumina can be incorporated into polyesteramides synthesized from AS and PG seed oils. Al-incorporated-polyesteramides were found to possess higher physico mechanical and chemical resistance properties than the corresponding neat polyesteramides. The amount of unsaturation in the fatty acid chain influences the physico-mechanical and chemical resistance properties of Al-PGPEA-71, Al-ASPEA-46, and Al-LPEA-22 coatings. Al-PGPEA-71, having the largest amount of oleic acid chains was found to exhibit superior physico-mechanical and chemical resistance properties compared with Al-ASPEA-46 and Al-LPEA-22. Al-PGPEA-71 can further be more rigorously evaluated as a candidate for anticorrosive coating material from a sustainable resource.

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