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Journal of Macromolecular Science, Part A

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597274

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To cite this Article Ahmad, Sharif , Ashraf, S. M. , Sharmin, Eram and Alam, Manawwer(2005) 'Ambient Cured Tartaric Acid Modified Oil Fatty Amide Anticorrosive Coatings', Journal of Macromolecular Science, Part A, 42: 6, 751 — 764

To link to this Article: DOI: 10.1081/MA-200058648

URL: http://dx.doi.org/10.1081/MA-200058648

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Journal of Macromolecular Science®, Part A: Pure and Applied Chemistry, 42:751–764, 2005

ISSN 1060-1325 print/1520-5738 online
DOI: 10.1081/MA-200058648

Ambient Cured Tartaric Acid Modified Oil Fatty Amide Anticorrosive Coatings

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A novel tartaric acid modified fatty amide diol (TAFA) was synthesized through the condensation polymerization of N,N-bis(2-hydroxy ethyl) linseed oil fatty amide and tartaric acid (TA).The structural elucidation of the TAFA resin was carried out by FT-IR,^{1}H-NMR, and ^{13}C-NMR spectroscopic techniques. The physico-mechanical and physico-chemical characterization of the resin were done by standard methods. TAFA, when further reacted with butylated melamine formaldehyde (BMF) in different phr (part per hundred part of resin) (TAFA-BMF) was found to cure at room temperature. The TAFA-BMF cured system was subjected to spectroscopic analysis to ascertain the structure and curing scheme of the same. The thermal studies of these resins were carried out by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The physico-mechanical properties and anticorrosive performance of TAFA-BMF coatings were evaluated by standard methods. The effect of TA and BMF on thermal stability, physico-mechanical and anticorrosive properties of resins was also investigated.

Keywords fatty amide, tartaric acid, coatings, anticorrosive properties

Introduction

In recent years, there has been growing concern for the conservation of the quickly depleting petroleum stock. The escalating prices of petro-based chemicals have further motivated the researchers and industrialists to substitute sustainable resources as an alternative to petroleum resources (1–4). Vegetable seed oils, a sustainable resource, are considered important due to their functional attributes (2, 4). They have been actively utilized to develop low molecular weight polymers viz. alkyds (5), polyesters (6), polyepoxies (2, 7, 8, 29), polyurethanes (2, 3, 9–12), and monomers such as fatty amide diols (13–17).

Fatty amide diols are obtained by the base catalyzed aminolysis of seed oils with diethanolamine (15). They have been reportedly synthesized from linseed (15–19), soybean (3, 9), pongamia glabra (13), castor oil (17), annona squamosa oil (14) and argemone oil (20). The modification of fatty amide diols has been reported with styrene and acrylic monomers (18, 21). Exhaustive work has been carried out on polyester modification of the oil based fatty amide diols through their reaction with dicarboxylic acids

Received September 2004, Accepted October 2004

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such as maleic acid, phthalic acid, isophthalic acid, fumaric acid, sebacic acid, terphthalic acid, adipic acid, succinic acid and their anhydrides (15, 16, 22) by using suitable curing agents such as isocyanates and melamine formaldehyde that are capable of reacting with hydroxyl moieties (10, 17, 21).

Melamine resins are characterized by good quality hardness, resistance to water, alkali and other solvents, along with adequate thermal stability (23). It is reported that melamine formaldehyde resins have been used for the curing of polymeric coatings and paints (24). Generally, curing reactions of oligomers and polymers with melamine occur at elevated temperature (25). To achieve the room temperature curing, an attempt has been made for the modification of linseed oil fatty amide with tartaric acid, a dihydroxy dicarboxylic acid resulting in the formation of tartaric acid modified fatty amide diol (TAFA). It was further cured with BMF in different phr. As expected, the terminal hydroxyls, as well as the dihydroxyl groups of fatty amide backbone and tartarate of TAFA, respectively, may react with BMF and facilitate crosslink formation through ether linkages rendering the polymer curable at room temperature.

A literature survey reveals that the modification of oil fatty amide diol using tartaric acid in the above manner has not been reported (26). The present work deals with the modification of fatty amide diol through the incorporation of ester and ether linkages with special reference to the coating properties of BMF cured TAFA resin. Earlier workers have reported the use of BMF in polyester modification of oil fatty amides. However, the significant role played by melamine moieties and ether linkages, in the enhancement of the thermal, physico-mechanical and anticorrosive properties of the resin have not been highlighted (15–17, 21).

The work further reports a comparative study of TA modified fatty amide diol (TAFA) with previously reported phthalic acid modified (PEA) (28). The stipulated reaction schemes for the synthesis and curing of the resin were confirmed by FT-IR, ^{1}H-NMR and ^{13}C-NMR spectroscopic techniques. Thermal analysis was carried out by TGA and DSC techniques. The idea of the present work possesses prospects for incorporating ether, ester and amide linkages, along with melamine moieties collectively in the resin to improve coating properties of the resin.

Experimental

Materials

Linseed oil was procured from a local market. The fatty acid composition of the oil was determined by gas chromatography (GC: 111/8ss column, FID detector) (28) diethanolamine, xylene (Merck, India), tartaric acid (Sarabhai Chemicals, India), sodium methoxide, butylated melamine formaldehyde (Synthetic and Polymer Industries, Gujarat, India) were of analytical grade.

Synthesis

Synthesis of N,N-Bis(2-hydroxy ethyl) Linseed Oil Fatty Amide (HELA). HELA was prepared according to a previously reported method (28).

Synthesis of Tartaric Acid Modified Fatty Amide (TAFA). HELA (0.03 mol) and tartaric acid (0.025 mol) were dissolved in xylene (50 ml) and were placed in a four-necked round bottom flask fitted with Dean Stark Trap, nitrogen inlet tube, thermometer and mechanical stirrer. The reaction mixture was heated at 145 ± 5°C and refluxed until the calculated amount of water was collected in a Dean Stark trap. The progress of the reaction was monitored by acid value, as well as thin layer chromatography (TLC) at regular intervals. After the completion of the reaction, xylene was removed from the product under reduced pressure to obtain TAFA.

Curing Reaction of TAFA with BMF. TAFA was dissolved in xylene and treated with BMF (in different phr) in a four-necked round bottom flask fitted with a Dean Stark trap, nitrogen inlet, thermometer, and magnetic stirrer. The reaction was carried out under stirring at 120°C in the presence of a few drops of phosphoric acid used as a catalyst. The progress of the reaction was monitored by hydroxyl value and TLC. The solvent was removed from the resin in a rotary vacuum evaporator.

Characterization

TAFA and TAFA-BMF were characterized by FT-IR, ^{1}H-NMR, and ^{13}C-NMR spectroscopies. FT-IR spectra of these polymers were taken on a Perkin-Elmer 1750 FT-IR spectrophotometer (Perkin-Elmer Cetus Instruments, Norwalk, CT) using a NaCl Cell.^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker Spectrospin DPX 300 MHz using deuterated chloroform as a solvent and tetra methyl silane (TMS) as an internal standard. Thermal analysis of the polymer was carried out by Thermogravimetric Analysis (TGA) TA2000, TA Instrument, USA and Differential Scanning Calorimetry (DSC) (TA Instrument, USA) in nitrogen atmosphere. The inherent viscosity of TAFA and TAFA-BMF in xylene 0.5 g/100 ml at 25°C was determined by a Ubbelodhe viscometer. The physico-chemical properties such as iodine value (ASTM D5556) hydroxyl value, acid value, saponification value, specific gravity and refractive index were determined by standard laboratory methods (Table 2).

Preparation and Testing of Coatings

The 40 wt% solution of TAFA and TAFA-BMF in xylene were applied by a brush on commercially available mild steel strips, 30 × 10 × 1 mm size, for chemical resistance and 70 × 25 × 1 mm size to determine the specular gloss at 45° by a Glossmeter Model RSPT 20, (Digital Instrument, Santa Barbara) and to evaluate the physico-mechanical properties, such as scratch hardness (BS 3900), impact resistance (IS: 101 part5/sec.3, 1988) and bend test (ASTM D 3281–84). Coating specimens of TAFA were baked at 170°C for 10 min while those of TAFA-BMF were left to dry at room temperature. The dry to touch (DTT) and dry to hard (DTH) times of the latter was also noted. The thickness of these coatings was found to be between 78–50 μm as determined by a Elcometer Model 345 (Elcometer Instrument, Manchester, UK). Corrosion tests were performed in acid (5 wt% HCl), alkali (5 wt% NaOH), water and xylene by placing them in 3 inch diameter porcelain dishes in the aforementioned media. Periodic visual examination was conducted until the films showed evidence of softening or deterioration (Table 2).

Results and Discussion

Reaction schemes for tartaric acid modification of fatty amide (TAFA) and its curing reaction with BMF (TAFA-BMF) have been given in Schemes 1(a and b). The structures of TAFA and its cured counterpart (TAFA-BMF) have been confirmed by spectroscopic analysis as given below.

N,N-Bis(2-hydroxy ethyl) Linseed Oil Fatty Amide (HELA)

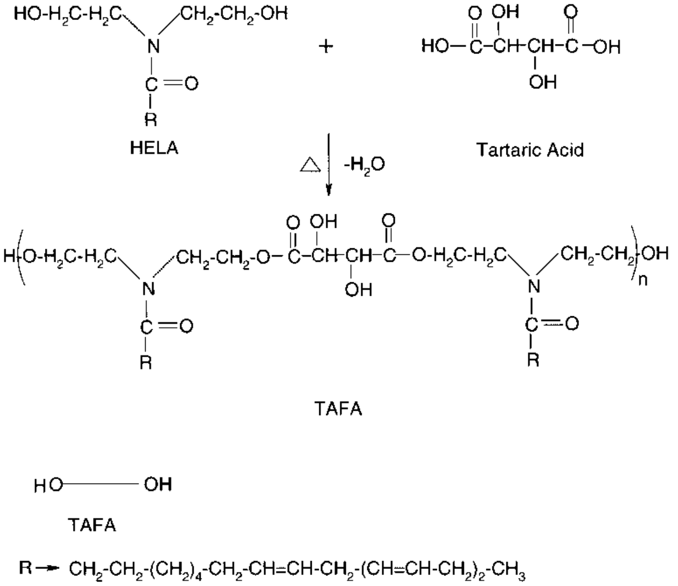
The structure of HELA was confirmed by FT-IR, ^{1}H-NMR spectral analysis according to our earlier reported paper (28).

Tartaric Acid Modified Fatty Amide (TAFA)

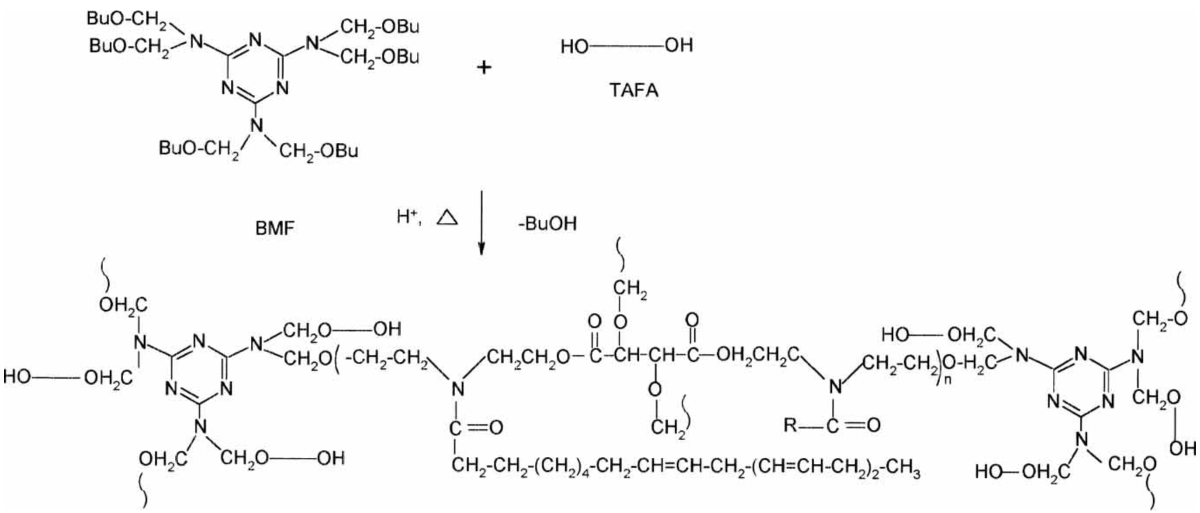
In the FT-IR spectra of TAFA, the broad band of hydroxyl group occurs at 3422 cm^{-1}. $-CH_2$ symmetric and asymmetric stretching vibrations occur at 2926 cm^{-1} and 2854 cm^{-1}. The ester carbonyl and amide carbonyl bands were found at 1740 cm^{-1} and 1648 cm^{-1}, respectively. The C–C (=O)–O and O–C–C bands for ester appear at 1250,1175. and 1135 cm^{-1}. The absorption bands for C–N of amide and those for –CH– of unsaturation occur at 1458 cm^{-1} and 3011 cm^{-1}, respectively.

The above mentioned bands were also observed in the spectra of TAFA-BMF along with some additional bands found at 1634 cm^{-1} for –CN– stretching of melamine (27) and at 1560–1557 cm^{-1}for in-plane stretching of s-triazine ring; the latter is normally the most intense band of melamine resin (25). Additional bands were distinctly observed at 1230–1171 cm^{-1} and at 1084 cm^{-1} for ether linkages formed by the curing reaction of TAFA with BMF.

The ^{1}H-NMR and ^{13}C-NMR spectra of TAFA and TAFA-BMF have been given in Figures 1–4 (27).



Scheme 1a. Synthesis of tartaric acid modified fatty amide (TAFA).



Scheme 1b. Curing reaction of TAFA with BMF.

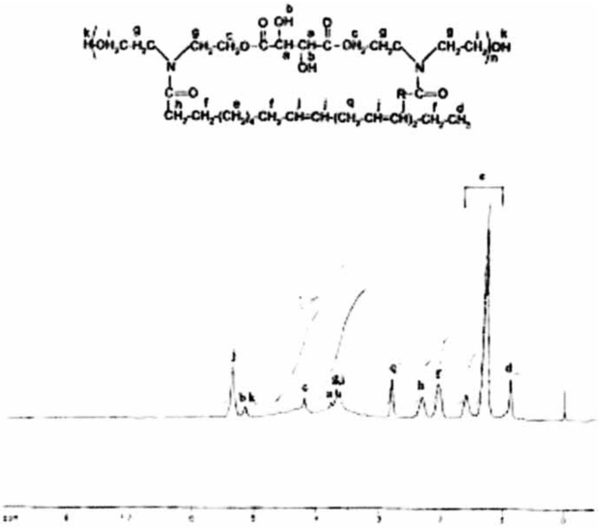


Figure 1. ^{1}H NMR spectra of TAFA.

Physico-chemical Characterization

Table 1 provides information about the physico-chemical characterization of PEA, TAFA and TAFA-BMF. It is observed that the hydroxyl value, saponification and iodine values decrease from PEA to TAFA and further decrease with increased loading of BMF in

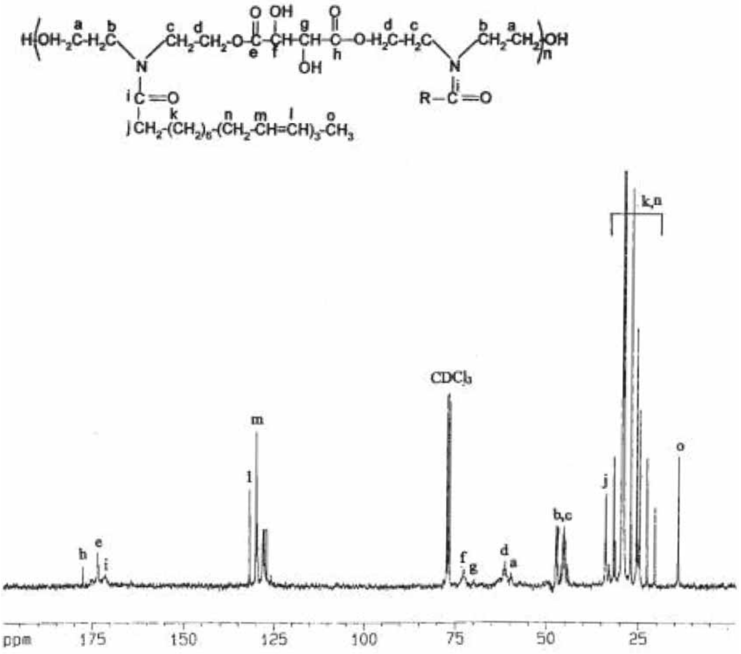


Figure 2. ^{13}C NMR spectra of TAFA.

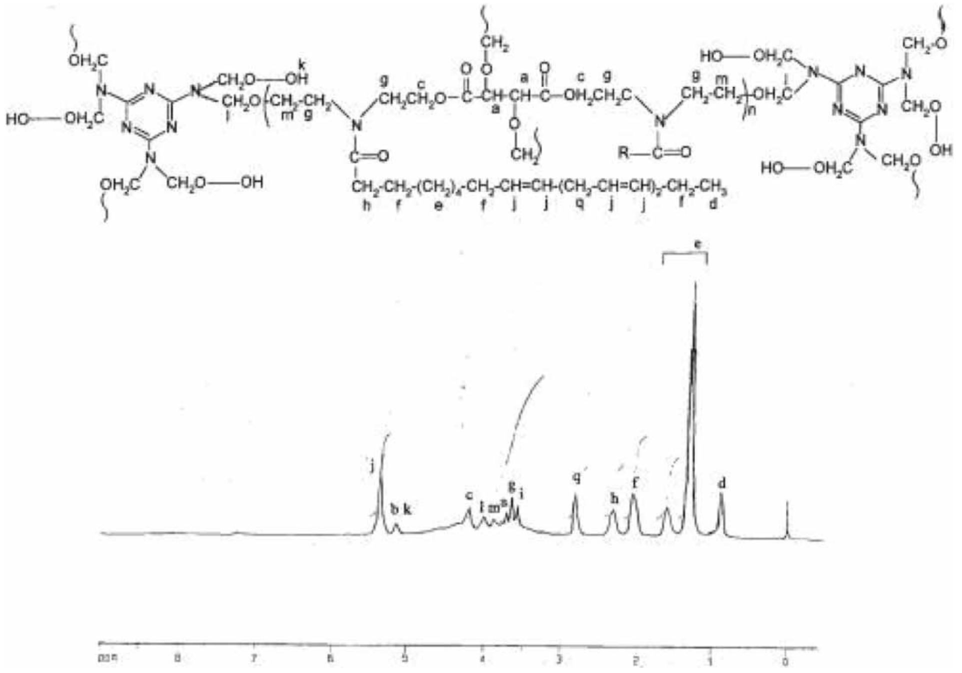


Figure 3. ^{1}H NMR spectra of TAFA-BMF.

TAFA. The specific gravity, refractive index and inherent viscosity increase at the same time. These trends emphasize the increase in molar mass of the resins from PAFA to TAFA and TAFA-BMF systems (18, 19, 23, 28, 30).

Coating Properties

Table 2 reveals the physico-mechanical and anticorrosive properties of PEA, TAFA and TAFA-BMF systems. A considerable improvement was observed in the baking temperature of TAFA coatings in comparison to PEA. Room temperature curing was achieved in the case of TAFA-BMF systems. The TAFA resin was formed by the esterification reaction between HELA and tartaric acid. The reaction scheme for the synthesis of TAFA (Scheme 1a), based on the spectroscopic analyses, shows that there are at least ten crosslinking sites per unit molecule of the resin, viz., four hydroxyls in the backbone of TAFA and six unsaturation sites in the pendant fatty amide chains to undergo etherification reaction with BMF and auto-oxidation, respectively. The additional hydroxyls attached to the tartarate moieties of TAFA may also undergo esterification reaction with carboxyls of another tartaric acid. It is, thus, assumed that the TAFA system when baked at 170°C (Table 2) may itself attain a high crosslink density and molecular weight. The latter assumption is further supported by the results of physico-chemical analysis, which indicate an increase in molar mass from PEA to TAFA to TAFA-BMF (Table 1). These factors, an increase in cross link density and high molar mass may be attributed to the lowering of baking temperature (from 220°C to 170°C) (9, 18, 19, 25, 30) in the case of TAFA compared to PEA. The spectroscopic analysis of the resin also confirms that there are two important functionalities, which

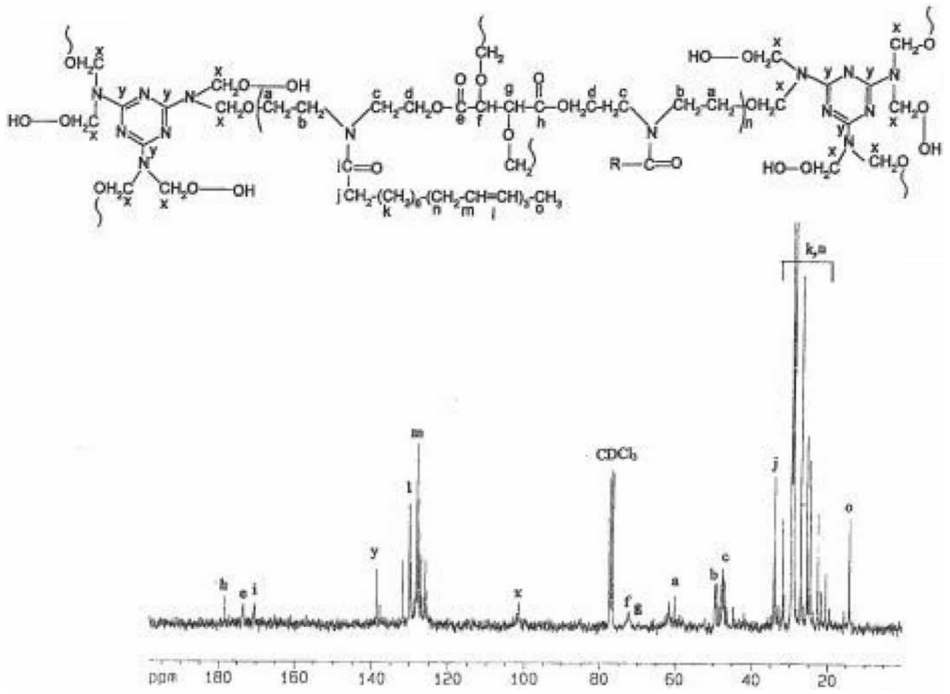


Figure 4. ^{13}C NMR spectra of TAFA-BMF.

participate in curing process-both the terminal and internal hydroxyls of TAFA, and the unsaturation present in the pendant alkyl chain.

With the successive addition of BMF, the drying times of TAFA-BMF decrease (Table 2). The possibility arises that with the increased loading of BMF, most of the cross-linking sites of TAFA and BMF participate in the curing process. The systems become denser and more compact (9, 18, 19, 25, 30). The etherification reaction

Table 1
Physico-chemical characterization of PEA (28), TAFA and TAFA-BMF resins

Resin code[a]	Refractive index	Sp. gravity	Inherent viscosity	Iodine value	Saponification value	Hydroxyl value (%)
PEA	1.5070	0.9380	0.6520	48	128	7.87
TAFA	1.5190	0.9421	0.6532	47	126	8.01
TAFA-10	1.5215	0.9431	0.6544	44	123	6.23
TAFA-20	1.5254	0.9440	0.6545	41	121	5.89
TAFA-30	1.5273	0.9443	0.6550	39	118	4.22
TAFA-40	1.5300	0.9447	0.6553	35	117	3.57
TAFA-50	1.5321	0.9450	0.6557	32	114	3.01

PEA,TAFA and TAFA-BMF Resin.
[a] Last digit of resin code indicates the amount of BMF loading (in phr).

Table 2
Physico-mechanical and anticorrosive properties of PEA (28), TAFA and TAFA-BMF systems

						Chemical resistance			
Resin code[a]	DTT (min)	DTH (hrs)	S. hardness [Kgs]	I. resistance [lb/in]	B. test [inch]	H_2O (7 days)	5 wt% HCl (7 days)	15 wt% NaoH (3 hr)	Xylene (7 days)
PEA[b]	—	—	2.0	100	1/4	e	f	a*	d
TAFA[c]	—	—	1.9	150	1/8	e	f	a	e
TAFA-BMF10	50	50	1.5	100	1/4	e	c	b	e
TAFA-BMF20	44	48	1.6	150	1/8	e	d	b	e
TAFA-BMF30	32	39	1.9	150	1/8	e	e	c	e
TAFA-BMF40	25	31	1.7	150	1/4	e	d	b	e
TAFA-BMF50	20	25	1.5	100	1/4	e	d	b	e

S. Hardness: Scratch Hardness, I. Resistance: Impact Resistance, B. Test: Bend Test DTT: Dry to touch, DTH: Dry to hard.

a: film completely removed, b: film cracked and partially removed, c: loss in gloss and weight, d: slight loss in gloss, e: unaffected, f: loss in gloss, a*: film completely removed in one hour.

[a] Last digit of resin code indicates the amount of BMF loading in phr.

[b] Baked at 220°C for 10 minutes.

[c] Baked at 170°C for 10 minutes.

between the terminal hydroxyls of TAFA, as well as the internal pendant hydroxyl of tartarate of TAFA, causes an excessive crosslinking density and increase in molecular weight. It is observed that in the matter of a few hours (Table 2), the coatings become DTH. The reaction scheme for curing of TAFA with BMF (Scheme 1b) also reveals that the cured TAFA-BMF system is highly cross-linked which facilitates their coatings to dry at room

Curing of the reaction may be assumed as a two-stage process. In the first stage, partial curing occurs through the etherification reaction of TAFA and BMF by SN_2 mechanism with the liberation of butanol (25). On application over the substrate, at this stage, the coatings become DTT. The second stage curing involves auto-oxidation wherein the coatings become dry to hard (DTH). Drying through auto-oxidation is reportedly a slow process (25). In contrast, in the present case, drying (DTH) occurred in a matter of a few hours. We assume that the high crosslink density, due to additional hydroxyls of TA, facilitates the formation of a compact network to be augmented further by auto-oxidation, resulting in a comparatively shorter DTH time (Table 2).

The scratch hardness values of TAFA and PEA were found to be comparable (1.9 Kgs and 2.0 Kgs, respectively). The same decrease was noticed from TAFA to TAFA-10, presumably due to the hard nature of melamine introducing brittleness to the systems. It is further observed that the scratch hardness was found to increase from TAFA to TAFA-30 beyond which it decreased again. The trend may be attributed to the increase in melamine content, which is followed by an increase in the ether linkages that confer flexibility and adhesion to the polymeric resin. We assume that the etherification (curing) reaction of TAFA with BMF provides a good combination of properties, which has a cumulative effect on the coating properties of the resin. Beyond TAFA-30, a further increase in melamine resin and crosslink density brought about by the curing reaction exceeds the optimum required limit and produce brittleness in the system causing lowering of scratch hardness. Impact resistance and bend test results also follow the same trend. Impact resistance and bending ability (flexibility) of a resin depend largely upon the adhesion of the polymeric resin to the substrate. The residual hydroxyl groups of tartarate moieties of TAFA contribute to the good adhesion of the resin to the substrate than PEA and consequently, result in the better impact resistance and bend test values/bending ability. However, beyond TAFA-30, as explained earlier, the system turns brittle lowering the impact resistance and bend test values, at the same time. Among all TAFA resins, TAFA-30 shows better properties compared to its other counterparts due to the formation of optimum crosslinks and ether linkages at this amount of BMF loading. Gloss values were found to be comparable among all resins (54–58 at 45°). This indicates that the dense structure formed in all the cases contributes to the good gloss of the resins.

The chemical resistance tests (Table 2) indicate that TAFA shows better performance than PEA under all corrosive media, in particular, against an alkaline environment. The coatings of the former are completely removed in 3 h, and the latter, in 1 h in a 5% NaOH solution. Table 2 shows that up to TAFA-30, the coatings perform satisfactorily under various corrosive media, presumably due to the polar residual hydroxyls and ether linkages rendering a well-adhered coating system. Beyond 30 phr loading of BMF, like physico-mechanical properties, anticorrosive properties also follow the same trend. Beyond TAFA-30, due to the hard nature of melamine resin and high crosslink density, the coatings are highly strained; they become brittle and lose their adhesion. This provides an easy passage for corrosive ions to penetrate through the coatings causing damage to the coating framework.

Thus, among all resin systems, TAFA-30 gives the best performance under alkaline and acidic environments. These studies reveal that the factors that govern the physico-mechanical properties directly affect the anticorrosive performance in various corrosive media.

Thermal Analysis

The effect of the BMF content on thermal stability of TAFA systems was investigated by TGA studies (Figures 5 and 6) In the TGA thermograms of TAFA and TAFA-BMF, the onset of degradation occurs in the range of 150°C to 175°C, which may be attributed to the evaporation of the trapped solvent. The 10 wt% loss occurred at 215°C and 235°C, while 50 wt% loss was observed at 349°C and 475°C in TAFA and TAFA-30, respectively. It was found that at initial temperatures of decomposition, the weight loss (10 wt%), in either case, occurred with a difference of 20°C. As the temperature for TGA is increased, the 50 wt% loss occurred with a difference of 126°C between TAFA and TAFA-30. These results highlight the fact that the important role played by BMF in determining the thermal properties of the resins become evident only at higher temperatures of degradation in TGA.

Thus, the TAFA-BMF system clearly possesses a higher thermal stability than the TAFA system itself. Melamine resins possess adequate thermal stability provided by the s-triazine ring. It is also well known that an increase in crosslink density increases the thermal stability of a resin (18). The higher thermal stability of TAFA-BMF may be attributed to the presence of melamine moieties forming a highly crosslinked network, both by the etherification reaction of melamine and through auto-oxidation at unsaturation of fatty amide chains.

DSC thermograms of TAFA and TAFA-30 show an endothermic peak at 126°C and 148°C. This peak may be attributed to the melting of the resin. An increase in flexibility causes reduction in the melting point of the resin and vice versa. The higher melting point of TAFA-BMF may be correlated to the presence of hard melamine content of the resin (9).

The thermal studies confirm higher thermal stability of TAFA-30, which may be safely used up to 200°C.

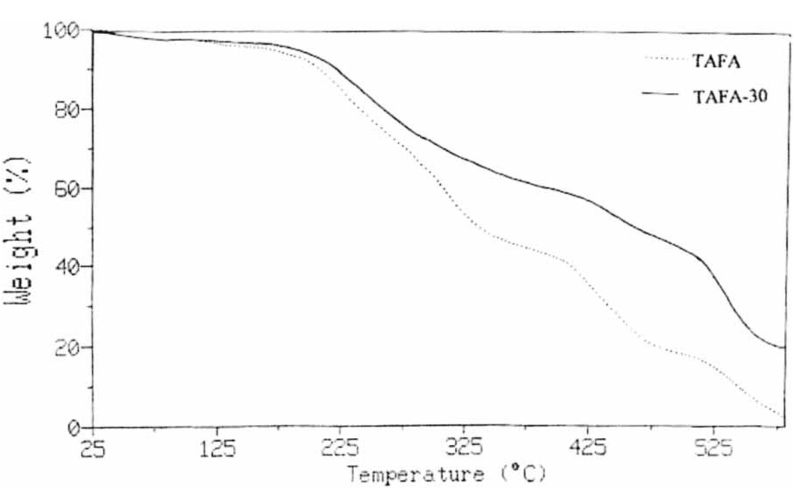


Figure 5. TGA thermogram of TAFA, TAFA-30.

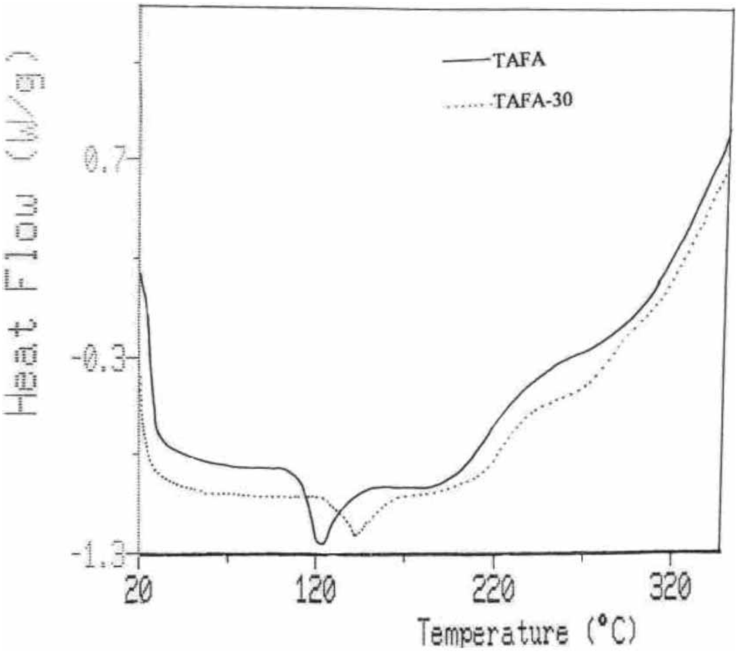


Figure 6. DSC thermogram of TAFA, TAFA-30.

Conclusion

The new tartaric acid modified fatty amide systems (TAFA and TAFA-BMF) showed a remarkable improvement in the drying property over PEA. The physico-mechanical and anticorrosive performance evaluation presents satisfactory results. A novel combination of properties of amide, melamine, ether and tartaric acid could be achieved by this approach. It offers a less time and energy consuming curing process, and also highlights the significant role played by tartaric acid and BMF in improving the coating properties of the systems. The studies reveal that TAFA-30 shows the best performance among all the resin samples developed and could be safely used up to 200°C.

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