

# Improving coating properties of shellac–epoxidised-novolac blends with melamine formaldehyde resin

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## Abstract

**Purpose** – The purpose of this study is to improve the coating properties of shellac–epoxidised novolac blends by treatment with melamine formaldehyde resin (MF) at ambient temperature for its use as a coating material.

**Design/methodology/approach** – Epoxidised-novolac resin was synthesised by epoxidation of novolac resin with epichlorohydrin. Novolac resin was synthesised by reaction of phenol with formaldehyde in acidic medium. Shellac was blended with the epoxidised-novolac resin in solution in varying ratios and treated the blends with MF resin in fixed ratio. Coating properties of the treated compositions were studied using a standard procedure. The compositions were characterised with Fourier transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA) and scanning electron microscopic (SEM) spectroscopy.

**Findings** – Treatment of shellac–epoxidised-novolac blends with MF resin improved water and alkali resistance of the blends, besides enhancing gloss. Gloss in all the blends was uniformly increased on treatment with MF resin. Water resistance of the blends tremendously improved after treatment with MF resin. Contact angle of the blends against water increased while decreased against ethylene glycol and dioxane. The compositions were more resistant to polar solvent than non-polar ones, suggesting that the compositions shifted to hydrophobic (lipophilic) nature on treatment with the MF resin.

**Research limitations/implications** – A specified concentration of MF resin was used in the study. Different concentrations of the MF resin can also be tried for treatment of shellac–epoxidised-novolac blends to see the effect of the resin on the blends.

**Practical implications** – Treatment of shellac–epoxidised-novolac blend with MF resin improved the coating properties of the blends. The formulation SeNB-64 is the best with high gloss, good impact, scratch hardness and water resistance, and hence can be used as coating material for metal surfaces.

**Originality/value** – Blending of shellac with epoxidised-novolac resin and treatment of the blends with the MF resin was done for the first time. The formulation SeNB-64 can be used as coating material for metal surfaces.

**Keywords** Resins, Coating, Synthetic resins, Lac resin

**Paper type** Research paper

## Introduction

Shellac is the refined commercial form of natural resin lac which is secreted by the tiny insects called *Kerria lacca* (Kerr.). It has been used extensively for decorative and protective purposes since ancient times. Shellac possesses a unique combination of properties, valued for its smooth, hard and glossy films and adhesion to a variety of surfaces. Its use is restricted because of its low softening point, poor water resistance, brittleness and insolubility in cheaper hydrocarbon solvents (Baboo and Goswami, 2010).

Modification of shellac was attempted from time to time to improve upon its drawbacks and for its suitable application. Shellac was reacted with various chemicals (Venugopalan,

1934; Venugopalan *et al.*, 1934) to improve the softening point of shellac. The resultant showed higher softening point but with increased brittleness. The brittleness was tackled with the use of plasticisers that led to further decrease in the softening point of shellac. Shellac was subjected to a variety of synthetic resins such as urea formaldehyde (Kumar and Sankaranarayanan, 1965), acrylics (Ansari and Goswami, 2006), epoxies (Tripathi *et al.*, 1966; Goswami and Kumar, 1988), alkyds (Chopra and Sankaranarayanan, 1967; Goswami *et al.*, 2009), vinyls (Mishra *et al.*, 1990), phenolics (Kumar and Sankaranarayanan, 1965; Goswami and Kumar, 1988; Bhatia *et al.*, 2006a; Ansari *et al.*, 2013), etc., for modification and improvement in the properties of lac. The modified products showed improved properties at the cost of other properties. Satisfactory formulation could not be achieved. Recently, chemical modification of shellac was carried out by blending shellac with epoxidised-novolac resin

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(Ansari *et al.*, 2014). Improvement in coating properties of shellac such as gloss, scratch hardness, impact resistance and alkali resistance was recorded but water and chemical resistance properties of shellac were adversely affected.

Melamine formaldehyde (MF) resin is a synthetic resin, prepared by condensation reaction of melamine and formaldehyde. It offers very good and varying properties when used along with other resins (Malshe and Sikchi, 2002; Chattopadhyay *et al.*, 2003a, 2003b). The MF resin has been tried for blending with lac by several researchers (Majee and Kumar, 1970; Lakshminarayanan *et al.*, 1974; Lakshminarayanan and Gupta, 1978). Kumar (1965) developed a varnish formulation based on shellac and MF resin. The varnish provided a quick drying glossy finish and its films were found to be heat- and water-resistant. Bhatia *et al.* (2006b) reported the effects of thermal stress on lac-MF blends at elevated temperatures and different baking times. Treatment of shellac-rosin blends with MF resin (Ansari *et al.*, 2012) overcame the drawbacks of shellac-rosin blends. Shellac-rosin blends showed resistance to water and heat on treatment with the MF resin. Gloss was further enhanced, but scratch hardness and flexibility were impaired with the treatment of the resin. Because shellac-epoxidised-novolac blends showed good scratch hardness, impact resistance and fairly good alkali resistance, the present study was aimed to improve other coating properties of shellac-epoxidised-novolac blends by treating with MF resin at ambient temperature.

## Experimental

### Materials

Novolac resin was synthesised by reaction of phenol with formaldehyde in acidic condition (Knop and Schieb, 1979). Phenol (AR grade), formaldehyde (AR grade, 37-41 per cent w/v), oxalic acid (AR grade), epichlorohydrin (AR Grade), sodium hydroxide (AR grade) of CDH, New Delhi, were used. MF resin (Biomine 1651, 60 per cent solid content) used, was purchased from M/s Hard Castle & Waud Manufacturing Co. Ltd., Mumbai, India.

Shellac was prepared in the form of button lac from kusmi lac of the Institute Research Farm. Epoxidised-novolac resin used was synthesised by epoxidation of novolac resin (Smit *et al.*, 2000; Ansari *et al.*, 2014). Epoxidation was carried out by reaction of novolac resin with epichlorohydrin in an alkaline medium. Novolac resin was dissolved in epichlorohydrin in a specific ratio (novolac/epichlorohydrin = 1:8 molar ratio), in a conical flask fitted with a reflux condenser in an inert atmosphere. In total, 20 per cent NaOH solution was added to the solution over a period of about 3 h maintaining the temperature at 100°C. The mixture was heated further for 10-15 min at 100°C and cooled down to room temperature. The whole viscous mass was transferred in a separating funnel and was extracted with toluene (1:1 v/v) by shaking for about 1 h to perform complete extraction of the epoxidised novolac resin. The NaCl crystals were removed by washing the resin with distilled water and dried. The epoxidised-novolac resin was isolated by evaporating the toluene.

### Preparation of test samples

Shellac-epoxidised-novolac blends were prepared in solution. Further, 20 per cent solutions of shellac (parent) and epoxidised-novolac resin (parent) were prepared in butanoltoluene (50:50 V/V) separately and blended together in appropriate ratios to make them 90:10, 80:20, 70:30, 60:40 and 50:50 (V/V). The blends were treated with MF resin in fixed ratio (Kumar, 1965) and allowed to stand for three days at ambient temperature for reaction to take place. Pre-cleaned and degreased tin panels and glass slides were used for developing films of the samples using the flow method. Curing of films was done at ambient temperature for seven days (Kumar, 1965). Properties of the blended products were studied using standard procedures.

### Analytical and testing methods

#### *Determination of finish and gloss*

Gloss of the blend films was measured on tin panels with the help of a gloss meter supplied by M/s Komal Scientific, at an angle of 45° according to ASTM: D 523-99. Gloss was recorded with respect to a standard black stone (57 per cent).

#### *Determination of flexibility*

Flexibility of blends' films developed on tin panels was measured as per ASTM D522, with a conical mandrel by rotation of the roller frame, tapering from 37 to 3 mm in diameter. The test was done by putting the prepared panels into mandrel, keeping the coated side upward. The bent portion of the panels was examined for any damage in the film.

#### *Determination of water resistance*

Water resistance test was carried out according to ASTM D 5402. Here the glass slides coated with sample were dipped in water for seven days. Wax sealing of the coated glass slides from all the sides was done prior to dipping in water.

#### *Determination of chemical resistance*

A chemical resistance test was performed by dipping the glass slides coated with the blend samples in the chemicals. The chemicals used were H<sub>2</sub>SO<sub>4</sub> (10 per cent), NaOH (10 per cent) and ethyl methyl ketone (EMK). The coated glass slides were sealed with wax from all the sides before dipping in the chemicals so as to expose only the coating materials to the chemicals.

#### *Determination of scratch hardness*

Scratch hardness of films of untreated and treated shellac, epoxidised-novolac resin and their blends, coated on tin panels, was measured by an automatic scratch hardness tester make of Sheen Instruments Limited, England, as per British Standards Method of test for Paints (BS3900: Part E2). Scratch resistance was measured by putting weights on 1 mm of tungsten carbide tipped needle with increments of 100 g. The machine operated at a constant speed of 3-4 cm/s.

#### *Determination of impact resistance*

The impact resistance of the cured film samples on tin panels was conducted as per BS 3900 (Part E3) by dropping a hemispherical-shaped indenting tool (diameter 7 mm) weighing 10.5 lbs from a height of 22.5 inch over the panels (in Sheen Falling Block Impact Tester). The tests were performed with the uncoated side of the panel facing the falling weight.

*Determination of contact angle measurement*

Contact angle was measured on coated metal surfaces against polar (water), moderate polar (ethylene glycol) and non-polar (dioxane) solvents, using optical contact angle measuring instrument OCAH 230 supplied by M/s Data Physics, Germany. The instrument CCD camera has a capacity of 450 images/s. The volume of the solvent used was 3  $\mu\text{l}$  and the rate of addition of solvent was 0.5  $\mu\text{l/s}$ .

**Characterisation techniques***Fourier transform infrared spectroscopy analysis*

The dried film samples of the blends were used in powder form for recording Fourier transform infrared (FT-IR) spectra with IR-Prestige 21 by Shimadzu Corporation, Japan, in DRS mode in the range of 4,000–450  $\text{cm}^{-1}$  at ambient temperature. Germanium-coated KBr pellet has been used in the study. The resolution was 4  $\text{cm}^{-1}$  and the number of scans was 20.

*Thermogravimetric analysis*

Thermogravimetric analysis (TGA) of parent shellac-epoxidised-novolac and their blends were carried out with Shimadzu TGA (model DTG-60) in the range of 30–800°C at a heating rate of 5°C/min under nitrogen atmosphere (30 ml/min). In total, 8–10 mg of powdered sample was taken for scanning.

*Scanning electron microscopic analysis*

The morphological changes in the films of the blends were studied with JEOL Scanning Electron Microscopy (SEM; model JSM 6,390 LV). The samples were coated with a thin layer of gold-platinum alloy by puttering to provide conductive surfaces.

**Results and discussion**

Blending of shellac with epoxidised-novolac resin produced homogeneous solutions. It is expected that the hydroxyl and carboxyl groups of shellac are taking part in the reaction with an oxirane group of epoxidised-novolac resin, opening the oxirane ring in a hydroxyl group, which, in turn react with alkoxy groups of MF resin, forming a more cross-linked structure (Scheme 1). Films of all the compositions obtained on glass slides and tin panels were smooth, hard and glossy with no tackiness. Treatment of shellac-epoxidised-novolac blends with MF resin improved water and chemical resistance of the blends, besides enhancing gloss further.

**Finish and gloss**

Films of shellac, epoxidised-novolac resin and their blends were found to be smooth and glossy. It is evident from Figure 1 that the gloss of blend samples increased as the concentration of epoxidised-novolac resin increased which was further enhanced on treatment with the MF resin.

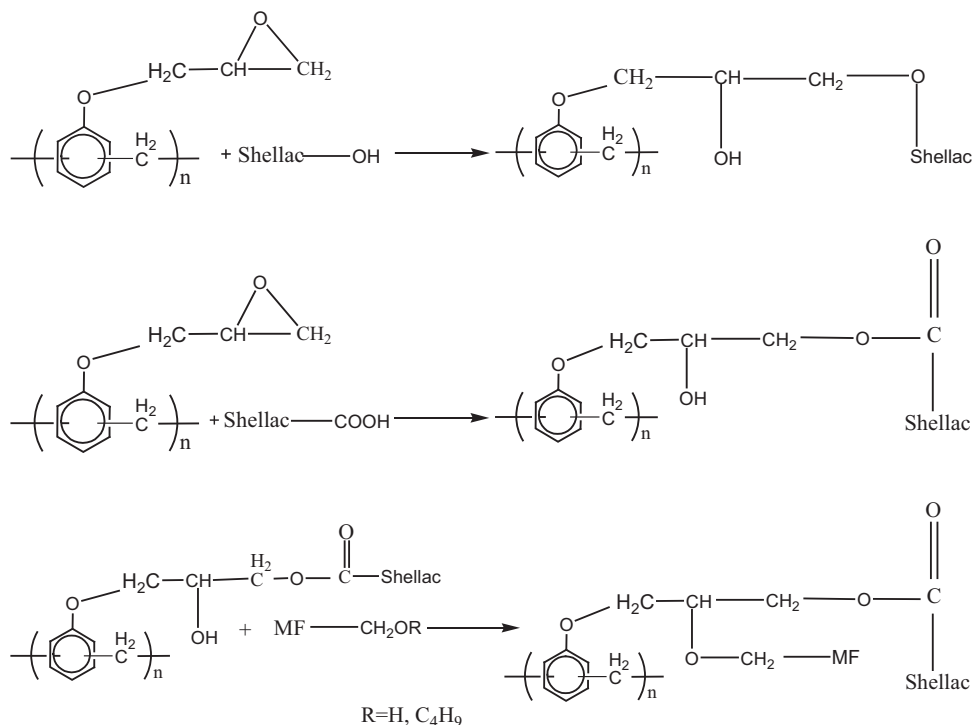
**Flexibility**

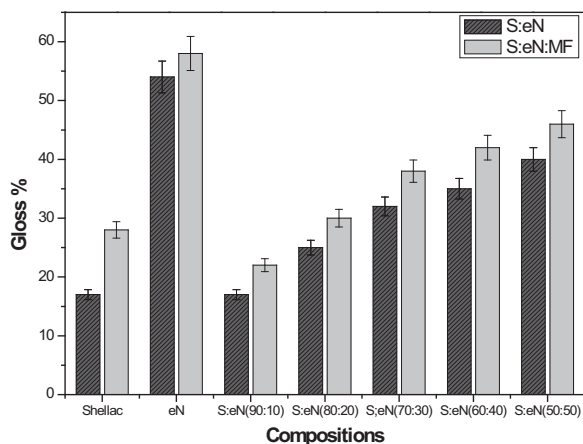
Films of parent samples and their blends showed uniform adhesion and were flexible in nature, as no cracks, peeling-off or detachment of the films was observed on bending the films in conical mandrel. Treatment with MF resin to the blends also showed flexibility as no cracks or flaking-off of the films was observed.

**Water resistance**

Treatment of shellac – epoxidised-novolac blends with MF resin improved water resistance tremendously. It was found that epoxidised-novolac resin was less resistant towards water

**Scheme 1** Reaction of shellac with epoxidised-novolac resin and reaction of blends with MF resin



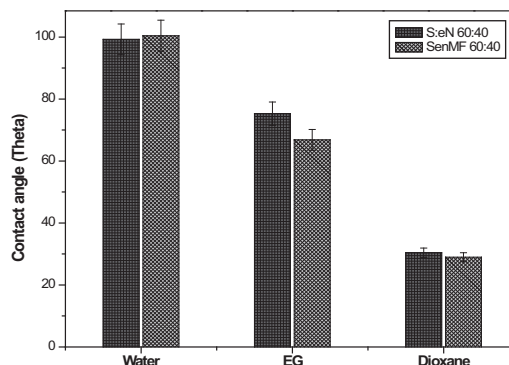
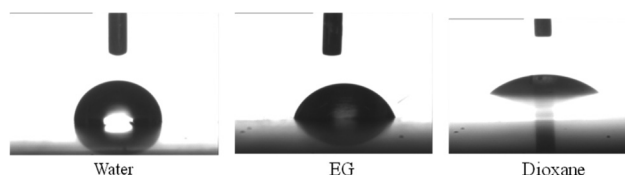
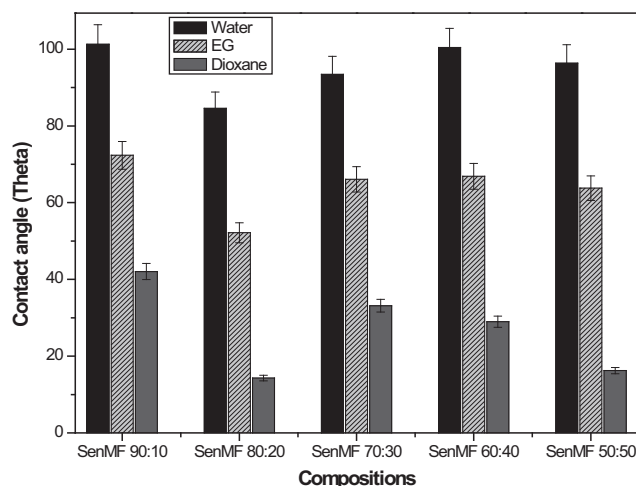
**Figure 1** Comparative gloss of untreated and MF resin-treated shellac-epoxidised-novolac blends

Notes: S = shellac; eN = epoxidised-novolac resin

and blushing occurred within 20 min. Blending with shellac decreased water resistance property, and as the concentration of epoxidised-novolac resin was increased, water resistance of blends decreased. Treatment of the blends with MF resin resulted in significant improvement, as no effect of water was noticed up to seven days on the films of the blends (Table I). It is believed that as shellac is blended with epoxidised novolac resin, hydroxyl and carboxyl groups of shellac reacted with oxirane of epoxidised novolac resin resulted in generation of free hydroxyl groups which make the resultant product more prone to water attack (decreasing the water resistance). The free hydroxyl groups generated reaction with alkoxy groups of MF resin, forming a more cross-linked structure and leading to significant improvement in water resistance of the blends (Scheme 1).

#### Chemical resistance

Chemical resistance of the untreated and treated blends with MF resin was studied in EMK,  $H_2SO_4$  (10 per cent) and NaOH (10 per cent). No marked effect of EMK and  $H_2SO_4$  on the films of untreated and MF resin-treated blends was observed for seven days. Films of untreated blends shrunk and were washed away in NaOH (10 per cent) solution. Resistance to alkali increased as the concentration of epoxidised-novolac resin increased. The blends treated with MF resin showed further increase in alkali resistance of the blends (Table I). It

**Figure 2** Contact angle of untreated and MF resin-treated shellac-epoxidised-novolac resin (60:40) blend against water, ethylene glycol and dioxane on tin panels**Figure 3** Contact angle images of shellac-epoxidised-novolac (60:40) blend treated with the MF resin against water, ethylene glycol (EG) and dioxane on tin panels**Figure 4** Contact angle of MF resin-treated shellac-epoxidised-novolac blends against water, ethylene glycol and dioxane on tin panels**Table I** Water and chemical resistance of the untreated and treated shellac-epoxidised-novolac blends with MF resin

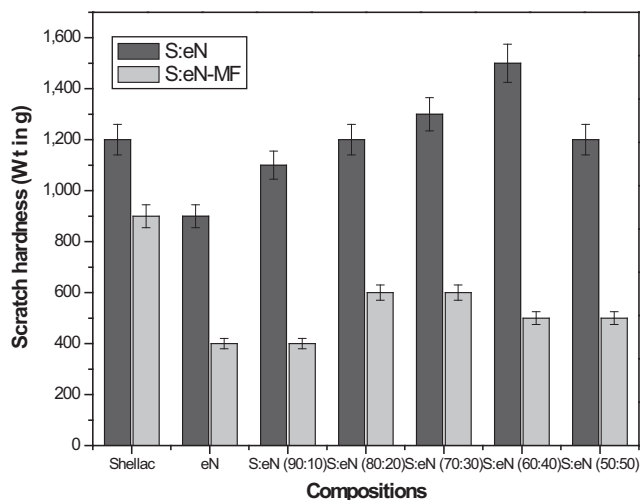
Shellac-epoxidised-novolac blends	No MF resin		MF resin	
	Water resistance	NaOH (10%)	Water resistance	NaOH (10%)
Shellac	26 h	10 min	> 7 days	25 min
Epoxidised-novolac resin	15 min	3 h	> 7 days	5 h
S:eN (90:10)	18 h	10 min	> 7 days	30 min
S:eN (80:20)	12 h	20 min	> 7 days	1.5 h
S:eN (70:30)	6 h	35 min	> 7 days	3 h
S:eN (60:40)	4 h	40 min	> 7 days	3 h
S:eN (50:50)	1 h	50 min	> 7 days	4 h

is thought that the oxirane group of epoxidised novolac resin (which is itself more alkali resistant) reacts with the carboxyl groups of shellac which is the reactive site in shellac for alkali. Treatment of blends with MF resin (an amino resin) makes it into more cross-linked structure, leading to further increase in alkali resistance.

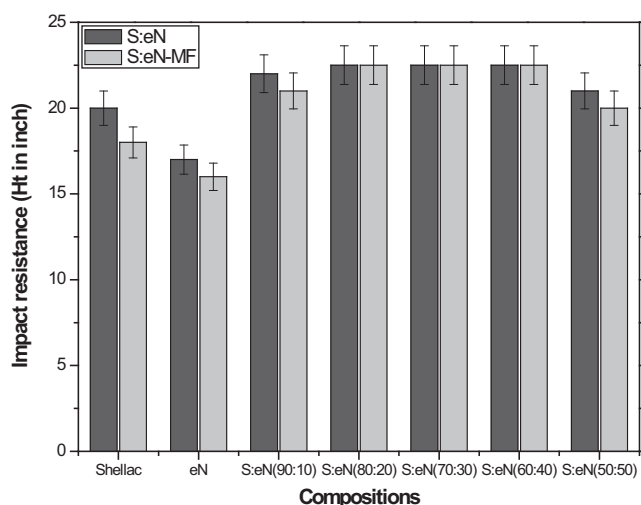
### Contact angle

Contact angle of shellac against water decreased on blending with epoxidised-novolac resin and decreased further as the concentration of epoxidised-novolac resin was increased. While, the contact angle increased on blending with epoxidised-novolac resin with respect to ethylene glycol and dioxane which further increased with the increasing concentration of epoxidised novolac resin, meaning it was difficult to wet the surface. On treatment with MF resin, the contact angle of the blends against water increased, while it decreased against ethylene glycol and dioxane (Figure 2). The contact angle of the blends decreased

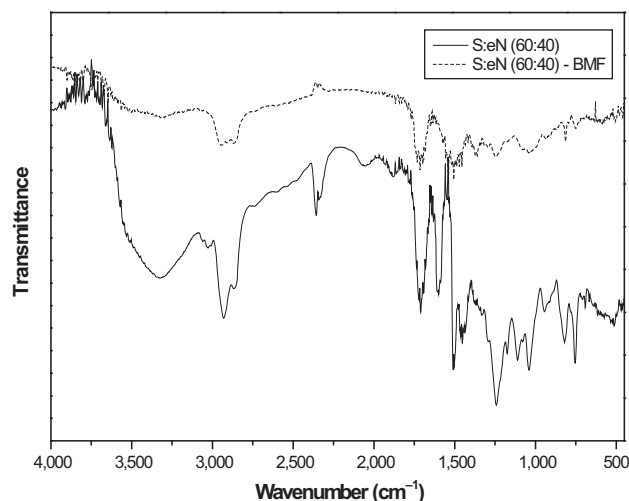
**Figure 5** Scratch hardness of untreated and MF resin-treated shellac–epoxidised-novolac blends



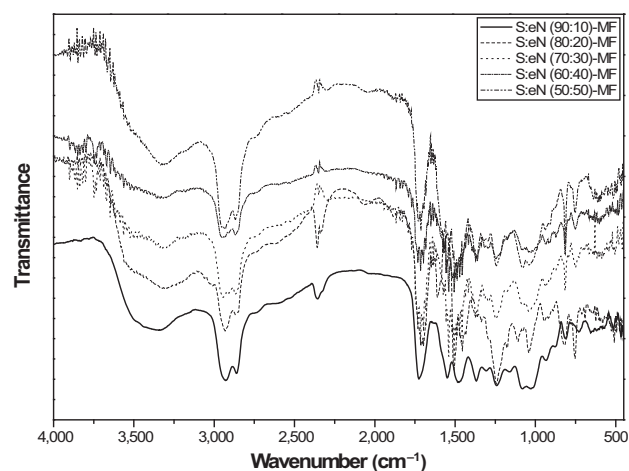
**Figure 6** Impact resistance of untreated and MF resin-treated shellac–epoxidised-novolac blends



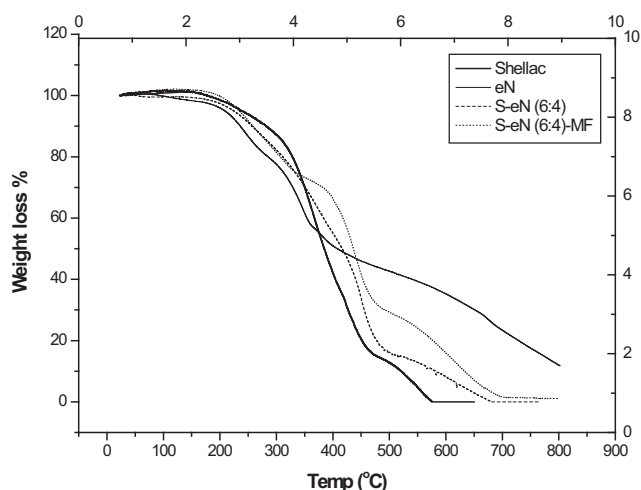
**Figure 7** FT-IR spectra of untreated and MF resin-treated shellac–epoxidised-novolac blends



**Figure 8** FT-IR spectra of shellac–epoxidised-novolac blends treated with MF resin



**Figure 9** TGA curves of films of shellac, epoxidised-novolac and shellac–epoxidised-novolac (60:40) blend and shellac–epoxidised-novolac (60:40) blend treated with MF resin





with the increasing concentration of epoxidised-novolac resin. The contact angle images of shellac-epoxidised-novolac blend (60:40) treated with MF resin with respect to water, ethylene glycol and dioxane on tin panels have been shown in the Figure 3. Comparing the contact angle of the compositions with respect to water, ethylene glycol and dioxane, it was found that the compositions were more resistant to polar solvents than non-polar ones, suggesting that the compositions shifted to hydrophobic (lipophilic) nature on treatment with the MF resin (Figure 4).

### Scratch hardness

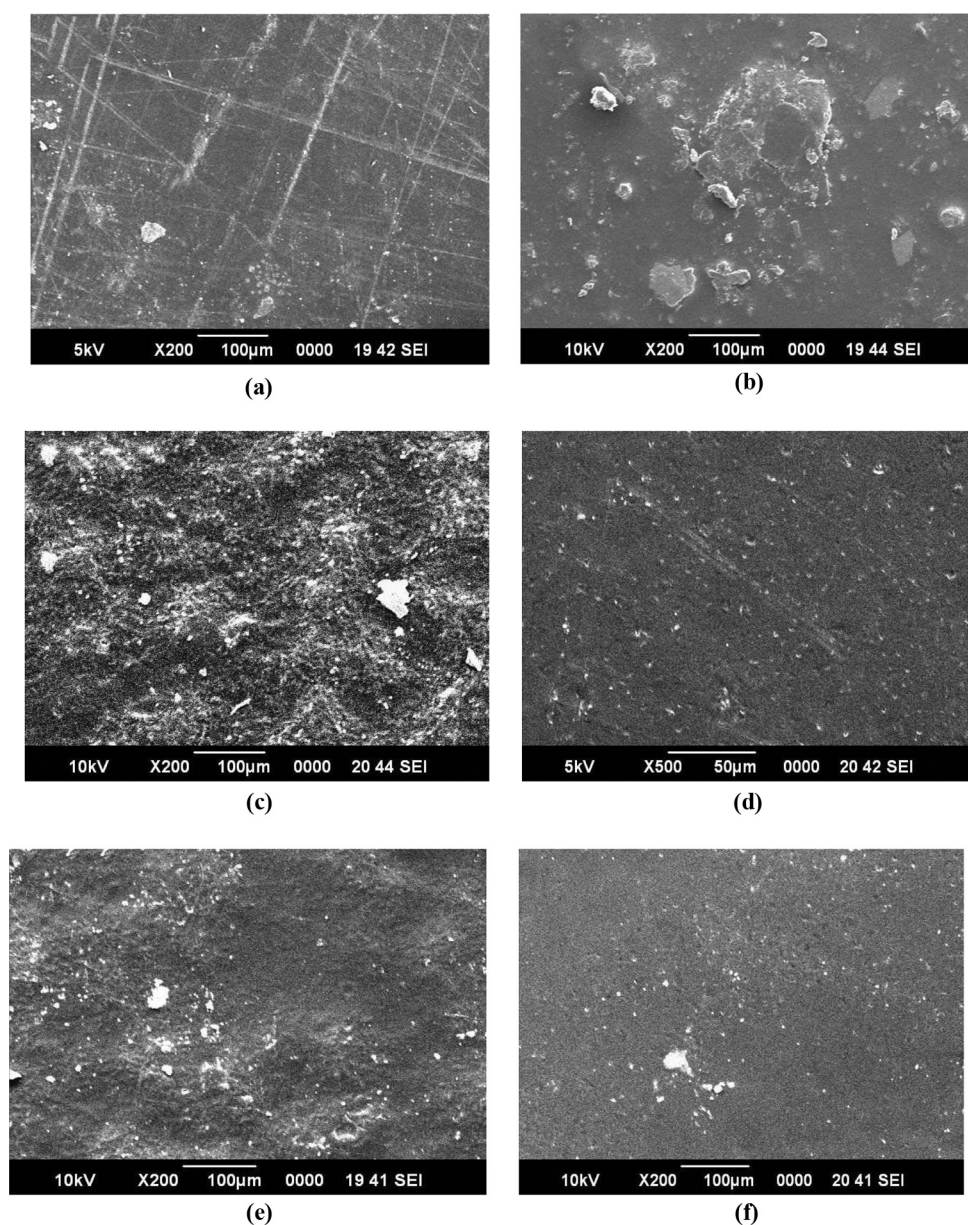
Scratch hardness increased with the increasing concentration of epoxidised novolac resin, and the highest scratch resistance

value was recorded for shellac-epoxidised-novolac resin (60:40) blend. Treatment of the blends with the MF resin decreased scratch resistance considerably; this might be due to the brittle nature of the MF resin (Figure 5).

### Impact resistance

Impact resistance increased on blending of shellac with epoxidised-novolac resin. Treatment with MF resin decreased impact resistance of the blends slightly, indicating decrease in adhesion of films. However, films of the blends (80:20, 70:30 and 60:40) showed resistance and adhered to the tin surfaces on impact of the block on the films of the blends from a height of 22.5 inch (Figure 6).

**Figure 10** Images of films of shellac (a), epoxidised-novolac (b) shellac-epoxidised-novolac (70:30) blend (c), shellac-epoxidised-novolac (70:30) treated with MF resin (d), shellac-epoxidised-novolac (60:40) blends (e) and shellac-epoxidised-novolac (60:40) treated with MF resin (f)



### FT-IR spectroscopy

Shellac – epoxidised-novolac blends treated with the MF resin were analysed with FT-IR spectroscopy. FT-IR spectra of untreated and MF resin-treated shellac–epoxidised-novolac blend (70:30) is shown in Figure 7. It is evident from the figure that with treatment of the blend with the MF resin, the intensity of hydroxyl, carboxyl, -CH moiety and epoxy decreased. Intensity of the hydroxyl group ( $3,323\text{ cm}^{-1}$ ) decreased may be due to reaction of hydroxyl groups of MF resin with the hydroxyl groups generated in reaction of shellac with epoxidised-novolac resin (Scheme 1). Intensity and sharpness of carbonyl group peak (appeared at  $1,708\text{ cm}^{-1}$ ) reduced and intensity of aromatic nuclei ( $2,931\text{ cm}^{-1}$ ) and oxirane group ( $1,250\text{ cm}^{-1}$ ) decreased after treatment with the MF resin, indicating the interaction with the resin. Peaks of hydroxyl groups broadened and flattened on increasing the concentration of epoxidised-novolac resin, while the intensity of carbonyl group, -CH moiety and epoxy group increased as the concentration of epoxidised-novolac resin increased (Figure 8).

### Thermogravimetric analysis

Films of shellac, epoxidised-novolac and their untreated and treated shellac–epoxidised-novolac blends were analysed for thermal stability and degradation. The TGA thermograms obtained for samples of shellac, epoxidised-novolac (eN) and their untreated and treated blends (6:4) with the MF resin are shown in the Figure 9. Degradation of shellac was found to occur in two steps, and complete degradation took place at  $575^{\circ}\text{C}$  only. Degradation of epoxidised-novolac resin occurred in four steps, and total degradation was 88 per cent up to  $800^{\circ}\text{C}$ , requiring higher temperature for complete degradation. It may be because of the presence of NaCl crystals in the epoxidised-novolac resin, which is formed during preparation of the resin. While, the shellac–epoxidised-novolac blend (60:40) sample degraded in two steps and 100 per cent degradation occurred at  $681^{\circ}\text{C}$ . Degradation of the shellac–epoxidised-novolac blend (60:40) treated with the MF resin occurred in three steps and 98.43 per cent degradation took place at  $700^{\circ}\text{C}$  and remained constant thereafter up to  $800^{\circ}\text{C}$ , may be because of the presence of some traces of NaCl crystals or some metal oxides present in the MF resin. It was observed that treatment of shellac–epoxidised-novolac blend with the MF resin increased the thermal stability of the blend.

### Scanning electron microscopy

SEM images of films of shellac–epoxidised-novolac and their untreated and treated blends on tin panels are shown in the Figure 10. The images showed that the films of parent shellac and epoxidised novolac resin and untreated blend have more pore structures, uneven surfaces, sagging of the films and voids, whereas film of shellac–epoxidised-novolac blend treated with the MF resin showed more of compact structures and uniform and smooth surfaces. This resulted in an increase in resistance of films towards water and chemicals as the film showed more cross-linked, smooth and even surface leaving no pores for water and chemicals to penetrate the surface.

### Conclusions

Gloss of shellac–epoxidised-novolac blends increased on treatment with the MF resin, whereas scratch hardness and impact resistance were adversely affected; the properties decreased with the treatment of the MF resin in the blends, inducing the brittleness in the blends. Resistance of the blends towards water and alkali improved with the treatment of the MF resin. The contact angle of the blends against water increased on treatment with the MF resin, showing lesser interaction of water with the films of the blends. Treatment with MF resin increased hydrophobicity of the blend. Based on the overall performance of the blends, the composition of shellac–epoxidised-novolac (60:40) blend treated with the MF resin was found to be the best and can be used as a coating material for metal surfaces.

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