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STUDIES ON MODIFIED SHELLAC

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Investigations have been made for the utilization of indigenous shellac in paint industry after its modification. The product obtained by fusion of equal parts of modified shellac and epoxy resin at 150 °C for 20 min gave the best surface coating properties. The fused mass is soluble in methyl ethyl ketone and the baked film obtained from it has been found to be superior. The film after baking at 190°C for 10 min shows good bond, flexibility, appreciably high scratch value and good water and alkali resistance.

Much work has been done on synthetic resins, which are superior to shellac. It is, therefore, losing its importance day by day. Shellac can be improved by esterification but even the modified shellac is inferior to synthetic resins.

The solubility of shellac in alcohol is the governing factor in determining the utility of lac. The indigenous shellac is of poor quality, has high acid value and insoluble matter. Modification of lac with epoxy resin has been reported.[1] The object of the studies was to see the possibilities of utilization of indigenous shellac in paint industry. The acid value and insoluble matter were lowered by modification and it was further improved in conjunction with epoxy resin. The hydroxyl and carboxyl groups present in the modified shellac react with epoxide groups of the epoxy resin and there is much improvement in the quality of film.

Materials Used

Lac of East Pakistan origin, m.p. (Durrans) 83–84°C, insoluble matter in cold alcohol 7.3%, acid value 123.2, and iodine value 48. Bisphenol A commercial. Epichlorohydrin commercial. Epoxy resin, m.p. (Durrans) 68°C, epoxide equivalent[2] 494, mol wt 890. Linseed oil fatty acids prepared from raw linseed oil. acid value 199.5.

Preparation of Starting Materials

Modified Shellac.—Modified shellac[3] of low acid value can be prepared by decreasing the amount of shellac and increasing that of linseed oil fatty acids while keeping the ratio of glycerine constant. In view of the utilization of major portion of indigenous shellac, shellac and linseed oil fatty acids in the ratio of 7 to 3 were used. Modified shellacs of different acid values were tried. The shellac of acid value 80 was most suitable.

Modified Shellac of Acid Value 80.—Thirty g linseed oil fatty acids and 0.5 g *p*-toluenesulphonic acid were heated to 130°C and then 10 g glycerine was added slowly with stirring. The reaction mixture was then heated to 140°C for 15 min. 70 g powdered shellac was then added, the temperature maintained at 140°C and the reaction allowed to proceed for 1 hr. The modified shellac so obtained is completely soluble in methyl ethyl ketone (MEK).

Epoxy Resin.—A mixture of 114 g of bisphenol and 37.5 g NaOH (10% aq solution) was heated to 45°C with stirring and then 72.5 g epichlorohydrin was introduced with rapid stirring. The temperature was raised to 95°C and maintained for 1½ hr. Heating was then stopped and the resin was washed with hot water several times. It was then dried.

Experimental

Epoxy resin and modified shellac were separately dissolved in solvent (methyl ethyl ketone) mixed well and then fused.

Effect of Heat Treatment.—When modified shellac and epoxy resin are heated together, chemical reaction takes place and we get a product which is soluble in solvents. On further heating the reaction proceeds further and the product becomes insoluble. Heating was done in two stages called fusion and baking. The mixture was first fused at 150° ± 5°C for 10, 15 and 20 min. The panels were then baked at 150, 170 and 190°C (±5°C) for 20, 15 and 10 min respectively.

Results

Varnish Films

Each composition of epoxy and modified shellac in the ratios of 15:85, 25:75 and 50:50 were separately fused for 20, 15 and 10 min at 150°C and applied on tin panels. Ease of application, appearance and finish were found satisfactory.

Baked Films

(1) All the panels pass the bend test.

(2) Scratch value

Epoxy: mod. shellac	Fusion time (min)	Scratch value (Baking temp/time) 150°C/ 20 min	170°C/ 15 min	190°C/ 10 min
15 : 85	10	700	800	850
	15	800	850	850
	20	900	1100	1200
25 : 75	10	1050	1150	1200
	15	1100	1250	1350
	20	1100	1200	1500
50 : 50	10	1400	1450	1450
	15	1400	1500	1600
	20	1400	1450	1600

(3) *Resistance to Water (72-hr immersion).*—The panels prepared from 15:85, 25:75 and 50:50 epoxy shellac, fused for 10, 15 and 20 min and baked at 150 and 170°C showed gradual improvement in the behaviour of the films from complete disintegration to only discoloration. All the panels prepared as above but baked at 190°C remained unaffected. It was only in the case of 50:50 epoxy, modified shellac, fused for 20 min that no undesirable effect was observed for the panels baked at 150 and 170°C.

(4) *Resistance to* 5% Na_2CO_3 *(72-hr immersion).*—The behavior shown by the baked panels was similar to that of the previous test.

(5) *Resistance to* 5% NaOH *(48-hr immersion).*—All the panels of 15:85 and 25:75 epoxy, modified shellac baked at 150, 170 and 190°C showed poor resistance. But the panels of 50:50 epoxy modified shellac fused for 20 min and baked at 150, 170 and 190°C showed good resistance and no harmful was effect observed.

Modified Shellac of Acid Value 100.

Modified shellac and epoxy in ratio 3 to 1, fused at 150 for 20 min, thinned and panels baked at 150°C, 170°C and 190°C

Varnish Films. Ease of application is satisfactory. Finish is not full gloss and brush marks are visible. The film is not smooth.

Baked films. The scratch value of the panels remains on the lower side i.e. 700, 750 and 800. Resistance to water, 5% sodium carbonate solution and 5% sodium hydroxide solution was also found unsatisfactory.

Modified Shellac of Acid Value 80.

Varnish Films of the modified shellac only after thinning with MEK.

Ease of application, finish and appearance were satisfactory.

Baked Films. The panels baked at 150°/20 min 170°C/15 min and 190°C/10 min were tested for scratch value which remains on the lower side i.e. 500, 500 and 550 respectively. Resistance to water, 5% Na_2CO_3 and 5% NaOH solution were also found unsatisfactory.

Discussion

Modified shellac of acid value 80 gives the best results. The poor quality shellac used in the studies has iodine value of 48. It is adulterated with rosin to the extent of above 16.5% and this is the maximum impurity that can be allowed to achieve best results.

There is much improvement in film properties of modified shellac alone. Flexibiliy, hardness and resistance to water and alkali increases.

A small addition of 15% epoxy resin shows improvement. Scratch value of 1200 is obtained. Film is resistant to water and 5% sodium carbonate

Optimum fusion temperature was found to be 150°C for 20 min and optimum baking temperature was 190°C for 10 min.

Film produced by 50 parts modified shellac and 50 parts epoxy resin show very good results. Scratch value of 1600 was obtained as compared to 550 of modified shellac. The film is resistant to 5% sodium hydroxide solution and no effect was noted after 48 hr immersion.

References

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