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Shellac-Graft Copolymers: Part III — Shellac-Vinyl Monomer Mixture-Graft Copolymers

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Graft copolymerization of shellac with three vinyl monomers at a time has been attempted with the object of obtaining compositions with improved film properties. The monomers used were: Styrene, ethyl acrylate, acrylamide, methyl methacrylate and acrylonitrile. The best performance in respect of finish, hardness, flexibility, impact resistance and resistance to the action of water and other solvents is shown by a graft copolymer prepared using ethyl acrylate-styrene-acrylamide mixture (45:10:5 per cent on the wt of shellac).

It has been reported earlier^{1,2} that shellac can be made to graft copolymerize with a single vinyl monomer or with two vinyl monomers to produce copolymers with improved film properties. The preparation

of graft copolymers containing three vinyl monomers has now been attempted. The vinyl monomers were so chosen that each one of them imparted some specific property³ to the product on polymerization. For instance

while styrene, acrylonitrile and methyl methacrylate give hardness, ethyl acrylate imparts flexibility; acrylamide, crosslinks between the polymer molecules on baking. In all the compositions studied, therefore, three types of the monomers were included and graft copolymerized with lac by the method employed earlier^{1,2}.

In analogy with the previous^{1,2} reaction mechanism, the possible steps of reactions are as follows: Shellac on passing oxygen under specified condition¹ forms the hydroperoxide, which can break down into free radicals under suitable conditions. It is then graft copolymerized with a mixture of vinyl monomers, e.g. styrene, ethyl acrylate and acrylamide. The polymers formed on baking might lead to the formation of a crosslink structure.

Experimental procedure

Shellac was first converted into its hydroperoxide by passing oxygen into its aqueous ammonia solution as described earlier^{1,2}. The hydroperoxidized lac was then treated with a monomer mixture (60 per cent on the weight of lac) and sodium sulphoxylate of formaldehyde (0.2 g.). The reaction mixture was kept at $40 \pm 1^\circ\text{C}$. for 3 hr, while nitrogen was bubbled through it continuously. Films of the resulting emulsions were prepared on glass slides and tin panels by flowing and allowing to drain in an almost vertical position. The films were examined under standard conditions.

Results

The characteristics of the films are presented in Table 1.

Finish — The films on glass and tin surfaces, both air dried and baked ($150^\circ\text{C}/30$ min.) were smooth and glossy with no smell of the free monomers. They were also perfectly tack-free.

Hardness — In respect of hardness, there was no marked improvement except for two compositions, viz. (1) ethyl acrylate-styrene-acrylamide (30:20:10 per cent), and (2) ethyl acrylate-methyl methacrylate-acryl-

Table 1 — Film properties of shellac-vinyl monomer mixture graft copolymers

Ethyl acrylate		Vinyl monomers added (% on wt of shellac)				Scratch hardness (over 1 mm. steel ball), kg.	Flexibility over 3 mm. mandrel	Falling block impact resistance test	Water resistance after*		
		Styrene	Acrylamide	Acrylonitrile	Methyl methacrylate				24 hr	48 hr	One week
—	—	—	—	—	—	Cracks	Fails	No bluish	No bluish	No bluish	
40	10	10	—	—	0.7	do	do	do	do	do	
45	10	5	—	—	0.6-0.7	Passes	Almost passes	do	do	do	
35	15	10	—	—	0.7	Almost passes	do	do	do	do	
30	20	10	—	—	1.0	Cracks	Fails	do	do	do	
45	—	5	—	—	0.7	Almost passes	Almost passes	do	do	do	
30	—	10	—	—	0.6-0.7	Passes	do	do	do	do	
20	—	20	—	—	0.5	Cracks	Fails	do	do	do	
45	—	5	—	10	0.4	do	do	do	do	do	
30	—	10	—	20	0.7	Almost passes	do	do	do	do	
20	—	20	—	20	1.0	Cracks	do	do	do	do	

*For this test the films were immersed in water at room temp. ($22-26^\circ\text{C}$.) for different periods.

amide (20:20:20 per cent), for which the scratch hardness over 1 mm. steel ball was 1.0 kg. In all other cases it was 0.7 kg. or below.

Flexibility — With regard to flexibility also, only two compositions, viz. (1) ethyl acrylate-styrene-acrylamide (45:10:5 per cent); and (2) ethyl acrylate-acrylonitrile-acrylamide (30:20:10 per cent) showed satisfactory performance.

Falling block impact resistance test — Most of the films showed failure in this test; only in a few cases, the films just passed the test.

Water resistance — All the films were water (blush) resistant for one week.

Resistance to other solvents — Baked films of all the compositions were found to be resistant to spirit, toluene, benzene, acetone and methyl ethyl ketone for more than 24 hr,

but the films peeled off in dilute alkali and dilute soap solutions.

Conclusion

The results of the present study indicate that the graft copolymers of shellac with the vinyl monomers, ethyl acrylate, styrene and acrylamide added at 45, 10 and 5 per cent concentration levels on the weight of lac, possess satisfactory film properties when styrene is used as the film hardener. When acrylonitrile and methyl methacrylate are used as the film hardeners, the former gives a performance comparable to that of styrene, but methyl methacrylate does not give satisfactory results.

References

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