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SHELLAC PLASTICS

PART I. COMPOSITIONS CONTAINING UREA

BY

S. RANGANATHAN

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Introduction

Shellac compositions were introduced as long ago as 1870 for making moulded articles like buttons, knobs etc. but the discovery of its suitability for the manufacture of gramophone records towards the end of the 19th century marks the next great forward stride. Subsequently shellac found large applications in the moulding of electrical goods. However, during the last 25 years the use of phenol-formaldehyde resins for moulded goods has rapidly increased since they are stronger and more heat resistant than shellac mouldings and also because of the comparatively faster rate of output possible. Many patents have been taken out in the past to improve the heat resistance of shellac mouldings (1). It is well-known that shellac becomes progressively infusible on continued heating. Ranganathan and Aldis (2) studied the effect of temperature, pressure and various chemicals on the rate of hardening of shellac. They found that of a large number of chemicals investigated, urea was the most powerful accelerator of heat-hardening and that pressure retards hardening. Preliminary trials on the use of urea in shellac moulding compositions showed that it improves the heat resistance of the moulded specimens, more so, if the composition is heated at 95°C for a short time before moulding. (3). The results obtained were, however, merely qualitative. Gardner and co-workers (4, 5) prepared shellac compositions by mixing shellac, fillers, and various quantities of phthalic anhydride, dicyandiamide etc., between hot rollers until the stock was more or less cured. The blank sheets were coarsely ground and dried (preferably in a vacuum oven), and moulded specimens were tested according to the A.S.T.M. specifications.

In the present paper the mixing of shellac was effected in the presence of solvents for shellac or by running finely ground ingredients through a ball mill. The object of this paper is to describe experiments carried out with a view to improving the properties of shellac mouldings by the use of urea.

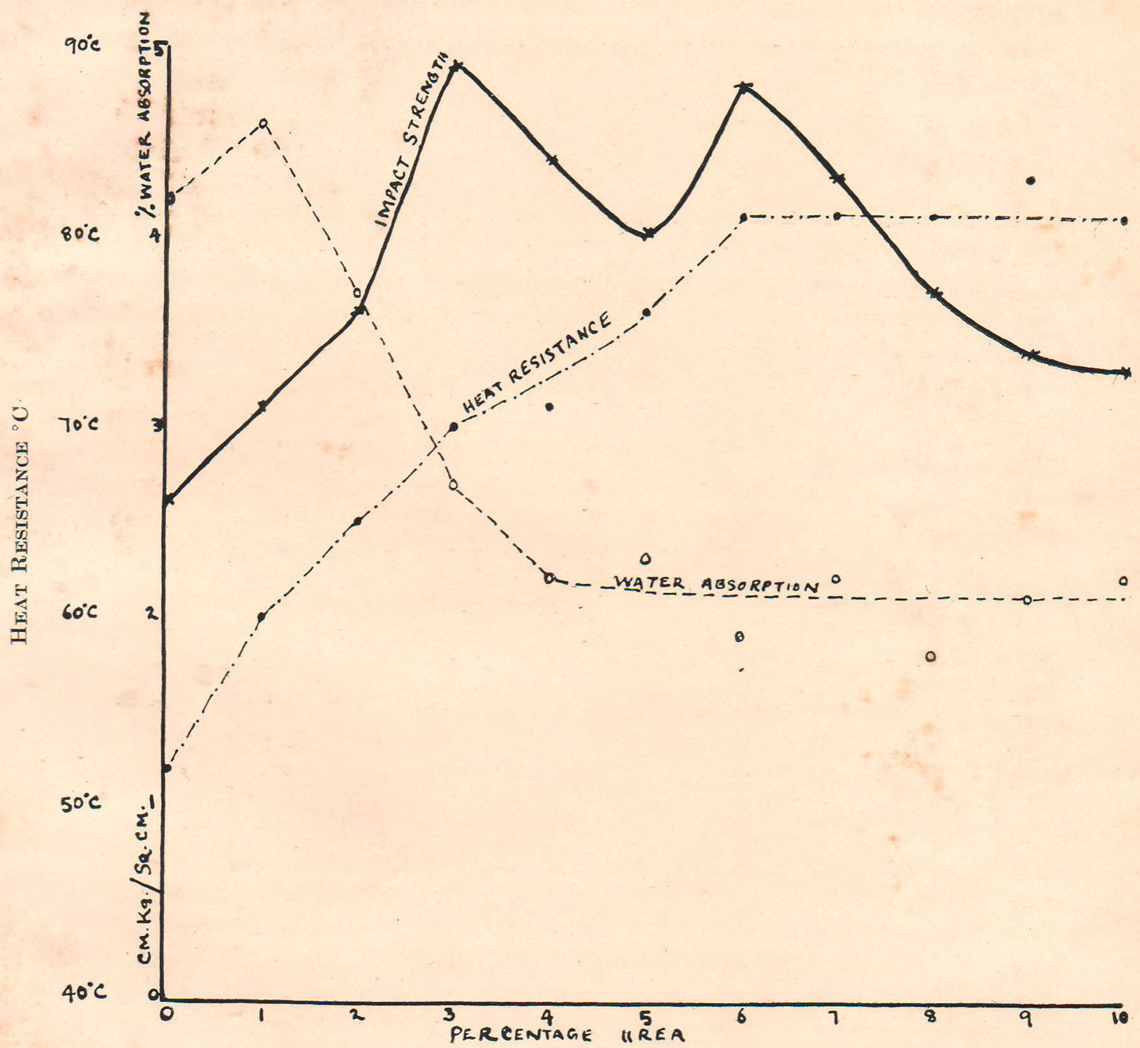
Proportion of urea

Pure Kusum shellac was dissolved in rectified spirit, 0-10% urea on the weight of shellac and a quantity of 60-mesh wood flour, equal to that of shellac used, were added and mixed to form a thick dough-like mass, which was first air-dried and then powdered to 60 mesh and dried in a vacuum oven at 55°C for 2 hours. Test specimens 12×1·5×1 cm. were prepared by using externally heated hand moulds in a Carver laboratory press and tested according to the German V.D.E. specifications. The results are tabulated below and also graphically represented in Fig. 1.

TABLE I.

Serial No.	Urea % on wt. of shellac.	Impact strength cm. kg/sq. cm.	Heat stability Marten's grade °C.	Vicat needle test °C.	Water absorption %	
					1 day.	7 days.
39	Nil	2 6	52	69	4·2	12·0
29	1	3·1	60	78	4·6	12·2
30	2.	3·6	65	87	3·7	11·4
31	3	4·9	70	96	2·7	8·6
32	4	4·4	71	108	2·2	7·1
33	5	4·0	76	115	2·3	7·3
34	6	4·8	81	121	1·9	6·0
35	7	4·3	81	127	2·2	7·1
36	8	3·7	81	129	1·8	6·5
37	9	3·4	83	132	2·1	7·7
38	10	3·3	81	130	2·2	8·0

It would be apparent from Table I and Fig. 1 that there is a definite improvement with 3% urea on the weight of shellac and by adding more than 6% there is practically no advantage as regards heat resistance and water absorption and the impact strength is definitely lowered.



HEAT RESISTANCE °C
90°C
80°C
70°C
60°C
50°C
40°C
% WATER ABSORPTION
CM. KG./SQ. CM. I
IMPACT STRENGTH
HEAT RESISTANCE
WATER ABSORPTION
PERCENTAGE UREA

FIG. 1

Fillers

The properties of moulded specimens depend on the nature and amount of fillers used. Wood flour is the usual filler employed in phenolic moulding powders for general purposes and Table II indicates the variation in properties of shellac compositions due to different proportions of 60-mesh pine-wood flour. In all cases shellac was dissolved in rectified spirit, 6% urea on the weight of shellac and varying amounts of wood flour were mixed and the powders were vacuum dried. The results are shown in Table II.

TABLE II.

Serial No.	Ratio of lac to wood flour.	Impact strength Cm. Kg/Cm².	Heat resistance Marten's Grade °C.	Vicat needle test °C.	Water absorption %.	
					1 day.	7 days.
20	25:75	4·8	81	134	3·1	10·0
21	30:70	5·3	81	126	2·4	7·9
22	35:65	5·4	79	120	1·8	6·4
23	40:60	5·5	77	107	1·6	5·3
24	45:55	5·6	75	110	1·4	5·1
25	50:50	5·0	70	111	1·2	3·9
26	55:45	5·1	68	103	1·2	3·9
27	60:40	4·8	68	97	1·0	2·9

With increasing proportions of shellac there is a diminution in heat resistance and water absorption but the impact strength attains a maximum at a ratio of 45 parts lac to 55 parts wood flour. This is explained by the fact that the maximum strength of a composition would occur when the binding medium is just sufficient to cover every interspace between the filler particles and neither so excessive as to form a weak resinous bridge between them nor insufficient to cover all particles. Further it was found that compositions with less than 35% lac yield specimens which do not possess a smooth surface.

Generally, shellac compositions contain mineral fillers like barytes, kaolin and asbestos and in Table III are given results showing the effect of the nature of filler on mouldings with and without the addition of urea.

TABLE III.

Serial No.	Composition.		Impact strength cm. kg/cm².	Heat resistance Marten's grade °C.	Vicat Needle test °C.	Water absorption %.	
						1 day.	7 days.
41	Lac ... Kaolin ... Barytes ... Bone black ...	30 32·5 32·5 5	2·6	56	75	0·43	2·1
42	Lac ... Micro-asbestos Bone black ...	30 65 5	2·6	58	74	0·11	0·35
77	Lac ... Wood flour ... Bone black ...	30 65 5	4·0	58	85	5·5	17·1
46	Lac ... Kaolin ... Barytes ... Bone black ... Urea ...	30 32·5 32·5 5 1·8	3·0	78	112	0·26	0·82
43	Lac ... Micro-asbestos Bone black ... Urea ...	30 65 5 1·8	2·6	81	105	0·08	0·31
116	Lac ... Wood flour ... Bone black ... Urea ...	30 65 5 1·8	5·2	89	130	3·4	11·9

The effect of urea in improving the heat resistance and mechanical strength of mouldings is greatest in the case of wood flour filler. Asbestos composition is the least affected by water immersion and wood flour-filled mouldings, the most. Fibrous fillers give mouldings of greater impact strength. Further work is on hand to improve the mechanical and thermal properties of mineral filled shellac compositions.

Mixing

It is not possible to mix fillers, shellac, and as much as 6% urea between hot rollers since the composition would quickly polymerise and lose all fluidity. In adopting the wet mixing procedure either alcohol or aqueous ammonia may be used for dissolving the shellac and the results obtained by the two methods, using pre-heated powders for moulding, are shown in Table IV.

TABLE IV.

Serial No.	Composition and mixing medium.		Impact strength Cm. Kg./Cm².	Heat resistance Marten's grade (°C)	Vicat Needle test (°C)	Water absorption %.	
						1 day.	7 days.
12	Lac 50 Wood flour 50 Urea 5	Ammonia ...	2·9	85	136	3·0	15·0
13	Lac 50 Wood flour 50	Ammonia ...	2·3	73	120	8·2	24·5
14	Lac 50 Wood flour 50	Rectified spirit ...	1·9	52	69	4·8	12·7
15	Lac 50 Wood flour 50 Urea 5	Rectified spirit ...	3·9	82	129	2·2	6·9

It would appear that the use of ammonia increases the impact strength and heat resistance but lowers the water-resistance of shellac mouldings. However, a comparison of the two mixings with urea shows that the alcohol-mixed composition is better than the other. The high water absorption due to the use of ammonia is a serious drawback in the adoption of this method.

Pre-heating

In the moulding of gramophone records, the stock composition is pre-heated for 4 to 5 minutes till it assumes a plastic consistency before being charged into the dies, so as to minimise the amount of heat taken up from the press platens. But if the pre-heating is too prolonged, the powder loses its plasticity. Urea treated shellac moulding powders fuse at much higher temperatures than ordinary shellac compositions and pre-heating at 90-95° for 15-20 minutes decreases the "flow" of the powder considerably but the resulting moulded specimens have a greater heat resistance. The following table illustrates the differences due to pre-heating.

TABLE V.

Serial No.	Composition and condition of powder.		Impact strength Cm. Kg./Cm²	Heat resistance Marten's grade °C	Vicat Needle test °C.	Water absorption%.	
						1 day.	7 days.
112	Lac 50 gm Wood flour 50 ,,	Vac-dry ...	3·4	52	62	5·1	14·2
		Pre-heated ...	3·6	60	76	5·0	13·9
113	Lac 50 gm Wood flour 50 ,, Urea 3 ,,	Vac-dry ...	5·4	78	102	2·0	7·7
		Pre-heated ...	5·5	84	123	2·2	7·8

Plasticity of the Moulding powder

Moulding powders soften and flow easily when pressed in hot moulds. The degree of fluidity depends on the melting point of the resin used and the nature and amount of filler used, for any predetermined temperature and hydraulic pressure. Mineral filled shellac compositions with a shellac : filler ratio of 25 : 75 moulds perfectly at 120°C and ¾ ton pressure but a wood flour composition with 50 : 50 ratio requires 120°C and 1½ tons pressure, or 150°C and 1 ton pressure. In every case where urea has been added, the plasticity of the powder is considerably reduced but good mouldings are possible at 160-170°C and 2 tons pressure, using hand moulds heated outside the press and pressing between cold platens.

Gloss of the moulded specimens

Shellac mouldings are bright and glossy when removed from the moulds but there is a slight falling off in surface appearance on keeping. Experimental observations have brought out the following points :

(*a*) Mineral fillers give more permanent gloss than fibrous fillers.

(*b*) The more fibrous the wood flour used, the poorer is the gloss retention. Figs. 2 and 3 are micro-photographs of the surfaces of mouldings with 60 mesh wood flour from pinewood and 60 mesh saw-dust from ebony wood. The less glossy specimens show up more fibrous patches.

(*c*) Mouldings of urea-treated shellac compositions do not suffer as much loss in gloss as those from compositions without urea.

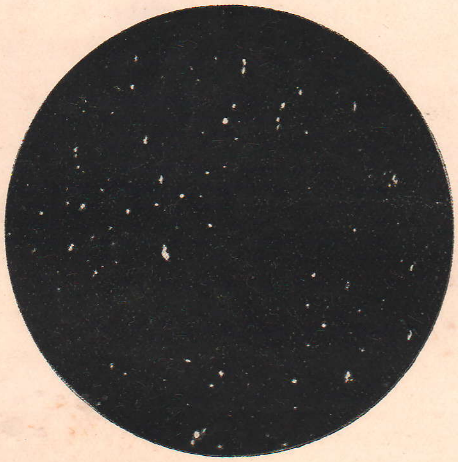


Fig. 2
Micro-photograph of
moulded surface using
saw-dust filler.

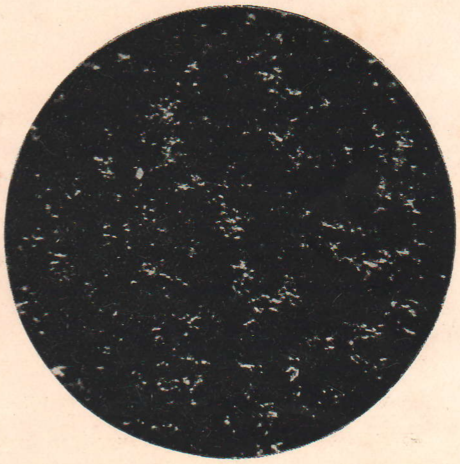


Fig. 3
Micro-photograph of
moulded surface using
wood-flour filler.

(*d*) Exposure to moisture decreases the gloss of wood flour filled mouldings. Retention of gloss depends on the softening point of the binder, and the texture, amount and hygroscopicity of the filler.

The initial surface finish of the mouldings depends on the ratio of lac to filler, the apparent density and texture of the filler and the temperature and pressure used in the moulding operation. It is possible to arrive at the optimum conditions for each type and ratio of filler.

Further work

Further work is in progress on the use of modified shellacs e.g. after removing the soft resin portion in part or completely, sulphur-treated lac, and lac combined with amines, aldehydes, polycarboxy acids, and phenols, the objective of such experiments being the discovery of a thermohardening type of shellac composition or a thermo-plastic modification with a higher softening temperature.

Summary

Urea is one of the best accelerators for increasing the heat resistance of shellac mouldings. Equal parts of shellac and wood flour with 6% urea on the weight of shellac intimately mixed together in the presence of alcohol and carefully dried, yield moulded articles of improved mechanical stength, heat resistance, and water resistance. Pre-heating of the powder for about 20 minutes at 90-95°C improves the properties of the mouldings. Addition of urea decreases the plasticity of the moulding powder but there is sufficient 'flow' at 160°C and 1-2 tons pressure for producing ordinary mouldings. For special high fluidity powders, the proportion of shellac should be increased slightly.

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