

## Improvement in Lac Processing Techniques: Part II— Production of Lac Wax of Superior Quality

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An oily fraction has been found mainly responsible for the tackiness, softness and low solvent retentivity of lac wax. Methods have been developed for its removal to give waxes having properties similar to those of the costly and imported carnauba wax. Methods have also been developed for converting dark lac waxes to perfectly white ones. These modified waxes can find use in various industries as a partial or complete substitute for carnauba wax.

Lac wax, an important byproduct of the lac industry, is obtained during various stages of lac processing. The wax possesses properties<sup>1</sup> very similar to those of the costly (nearly three times as much as lac wax) and imported carnauba wax and has a wide field of applications<sup>2,3</sup>. It is slightly inferior to carnauba wax as regards its hardness, melting point and solvent retentivity<sup>4,5</sup>. The dark coloured wax recovered from wash water, *kiri* or refuse lacs is inferior even to lac wax. A study was, therefore, undertaken to modify lac wax so as to upgrade it in respect of the above-mentioned characteristics. The improvement achieved towards this end is detailed in this paper.

Sticklac contains 5-6% wax. During the various stages of refining of sticklac to seedlac and shellac, the wax gets distributed into (i) sticklac wash water, (ii) seedlac, (iii) shellac and (iv) *kiri* or refuse lacs. The wax recovered during the production of dewaxed lac in alcoholic medium and the dewaxed bleached lac recovered in aqueous medium are of better quality as regards colour and hardness. The one recovered from wash water and *kiri* or refuse lacs is of inferior quality; it is soft, slightly tacky and dark.

### Experimental procedure

Various types of wax obtained during the different stages of lac processing, as mentioned

above, were taken. For modification of the waxes, a number of methods were tried and the most promising ones repeated several times with different types of waxes and found reproducible.

The following standardized methods have been found suitable in order of preference for upgradation of waxes in respect of hardness, melting point and solvent retentivity. Method D is meant for achieving colour improvement.

*Method A: Fractionation with acetone*—Pulverised lac wax is taken in a cloth bag and extracted with boiling acetone in a soxhlet extractor either glass lined or made up of cast aluminium. The extraction is carried out till all the soluble portion is dissolved out (usually 20-25 extractions are necessary) leaving the boiling acetone insoluble mass (I) in the bag. The extract is then allowed to cool to room temperature (22°-26°); a portion separates out (II) which is filtered and washed thoroughly with fresh acetone. Acetone is then removed by distillation and an oily mass (III) is obtained.

*Method B: Fractionation with n-hexane*—Lac wax, pulverised or in small bits, is dissolved in boiling *n*-hexane and allowed to stand at room temperature (22°-26°) for 12 hr. The precipitated wax is separated by filtration and washed thoroughly with fresh *n*-hexane. Distillation of

the solvent yields the soluble fraction as an oily mass.

*Method C: Extraction with caustic soda solution*—Lac wax, preferably pulverised, is boiled under stirring with 2% of its weight of caustic soda in water as a 0.2% solution for 4-5 hr. On cooling, the major portion of wax solidifies on the surface as a hard mass. It is taken out, treated twice or thrice with boiling water under stirring to remove any adhering alkali; on cooling it results in hard wax. The portion which remains as aqueous emulsion is removed as an oily mass by treatment with dilute sulphuric acid and washing with water. The properties of different waxes and their fractions are given in Table 1.

*Method D: Improvement of colour*—(i) Pale yellow wax: Dark wax (100 g) is dispersed in

boiling water (500 ml) by efficient stirring and is then oxidised by the addition of either sodium nitrite (1.5 g) followed by conc. nitric acid (2.5 ml, sp.gr. 1.18) or finely powdered potassium permanganate (2 g) followed by 10% hydrochloric acid (5 ml). The oxidation is allowed to proceed under boiling condition for 10 min and the pale yellow wax is recovered after cooling the mixture at room temperature. In the latter case, after oxidation, treatment with a small quantity of nitric acid (0.5 ml) under boiling condition is necessary.

(ii) Light straw coloured wax: The above pale yellow wax is redispersed in boiling water and bleached at room temperature under efficient stirring with the gradual addition of sodium hypochlorite solution containing 3% available chlorine. The normal requirement is 10g of available chlorine for 100 g of wax. The reaction

Table 1—Characteristics of Various Wax Fractions Obtained by Different Methods

Source of wax	Method used	Fraction No.	Yield of fraction %	Penetration value	Acid value	Sap. value	Ester value	Melting point °C	Solvent retentivity
ILRI, during manufacture of refined bleached lac	A	—	—	4.0-4.5	8.4	68.7	60.3	78-82	28.0
		I	70	1.5-2.0	2.8	63.7	60.3	83-85	16.0
		II	26	5.0-5.5	7.7	110.1	102.4	73-74	64.0
ILRI, from sludge obtained from wash water	A	III	3	—	21.1	272.0	250.9	oily	0.6
		—	—	5.0-5.5	10.2	71.0	60.8	77-80	19.2
		I	48	1.5-2.0	5.1	83.2	78.1	83-85	17.7
Commercial wax from wash water sludge	A	II	40	5.5-6.0	13.6	111.0	97.4	72-73	64.0
		III	11	—	24.2	283.0	258.8	oil	0.5
		—	—	5.5-6.5	38.2	108.0	69.8	75-78	23.0
Commercial wax (Angelo)	A	I	25	1.5-2.0	5.2	57.6	52.4	82-83	16.0
		II	52	6.0-6.5	15.7	87.8	72.1	71-72	68.0
		III	22	—	67.8	296.0	228.2	oily	0.6
Wax sample 2	B	—	—	3.0-3.5	12.2	72.2	60.2	81-82	11.0
		I	50	1.5-2.0	4.1	64.4	60.3	84-85	13.5
		II	41	2.5-3.0	6.9	98.3	91.4	72-73	68.6
Wax sample 2	C	III	8	—	20.9	107.0	186.1	oily	0.4
		I	76	3.5-4.0	7.2	98.3	88.8	80-82	44.6
Wax sample 2	C	II	24	—	20.1	207.0	83.9	oily	1.3
		I	81	4.0-4.5	8.0	95.0	87.0	80-82	43.2
Commercial carnauba wax	—	II	17	—	24.3	108.0	83.7	oily	1.7
		—	—	—	1.0-1.5	2.9-9.7	79-95	—	78-85

is allowed to proceed for 12 hr under stirring. The wax is removed, washed thrice with boiling water; a light straw coloured wax is obtained on cooling.

(iii) White wax: The light straw coloured wax is treated under molten condition with activated carbon (10-12 g per 100 g wax) and put on a filter paper over a hot water funnel, while the colourless wax filters out slowly. For a bigger charge, a bed of activated carbon is prepared on a thickly woven cloth put over a double jacketed Buchner type funnel which is kept hot by circulating hot water (95°). The molten wax is poured on the bed and allowed to pass through under gravity. The operation takes nearly 6 hr. The filtered product solidifies to a perfectly white wax.

The characteristics of different qualities of products thus obtained are given in Table 2.

### Results and discussion

It is seen from Table 1 that the proportionate yield of fractions depends on the sources of wax. The proportion of fraction I was highest in wax 1, and fractions II and III in wax 3. Fraction I was lighter in colour, tack free, hard, with high melting point and good solvent retentivity. Though fraction II was less hard and of lower melting point, it had the highest solvent

retentivity, while fraction III was oily and very dark with extremely low solvent retentivity.

The acid and saponification values were lowest in the case of fraction I in comparison to fractions II and III. It is evident that fraction I has got all the qualities of carnauba wax but for the solvent retentivity<sup>5</sup>.

It is seen from Table 2 that there is some loss of wax at each stage of treatment. The colour gradually becomes lighter and ultimately after the carbon treatment, the wax becomes perfectly colourless. There is a slight increase in the melting point. The wax becomes harder and non-tacky which is possibly due to partial removal of the oily portion during treatment with sodium hypochlorite.

Perfectly white wax was obtained only when the various operations were conducted in the sequence: oxidation, bleaching and adsorption. Any change in the sequence did not give the expected decolorisation. Adopting the above sequence, the solid fractions (I and II) from the dark coloured waxes could be converted to perfectly white variety, retaining the other desirable properties intact.

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Table 2—Characteristics at Various Stages of Oxidation, Bleaching and Adsorption of Dark Waxes

Sl. No	Treating agent	Yield %	Colour index*	Melting point °C	Penetration value
1	Nil (control)	—	0.7	77-80	5.5
2	NaNO <sub>2</sub> + HNO <sub>3</sub>	97	0.35	78-80	3.5
3	KMnO <sub>4</sub> + HCl	97	0.41	78-80	3.5
4	KMnO <sub>4</sub> + HCl + HNO <sub>3</sub>	97	0.3	78-80	3.5
5	No 2,3 or 4 + NaOCl	95	0.1	80-82	3.5
6	No 5 + activated carbon	85	0.0	80-82	3.0

\*Determined by dissolving 1g wax in 30 ml benzene and comparing 10 ml of the solution with 10 ml 0.01N aqueous iodine solution in a Dubasque colorimeter.