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# Utilization of Byproducts of Lac Industry: Part II—Manufacture of Shellac from Refuse Lacs

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**A simple method is reported for the reclamation of lac resin from refuse lacs. The refuse lac is subjected to an extraction treatment with a hot (85-90°C) aqueous solution of 7 parts of sodium carbonate and 3 parts of sodium sulphite per 100 parts of lac and the extract obtained is treated with 2 parts of sodium hydrosulphite. Lac is precipitated out of the solution obtained using dilute sulphuric acid. The reclaimed lac on sheeting has good colour and storage stability, and hue comparable to that of ordinary shellac.**

**I**N an earlier communication<sup>1</sup>, a method for the production of good quality bleached lac from *molamma* was reported. In this paper, a method for preparing shellac from refuse lacs, such as *molamma* and *kunhi* is described.

The presence of a large proportion of insoluble impurities in fine condition in refuse lacs makes the recovery of resin by the conventional hot filtration method of shellac manufacture difficult. The resin can be reclaimed only by solvent extraction. This process requires, in addition to the solvent (rectified spirit), costly equipment normally not available with the average shellac manufacturer. Lac resin, because of its acidic nature, is easily soluble in aqueous alkalies and alkaline salts, and as such a method based on aqueous extraction would be more acceptable, particularly to the small scale manufacturer, as it would not involve the use of expensive equipment or solvents.

Previous investigators<sup>2</sup> have observed that extraction of lac with alkali and its regeneration with inorganic or organic acids results in a resin characterized by: (i) High colour index; (ii) poor keeping quality; (iii) poor flow; and (iv) reduced polymerization time.

Various alkalies have been tried for the extraction of lac resin. Similarly different acids have been tried for its precipitation from solution<sup>2,3</sup>, but the recovered lac was always darker and of a hue altogether different from that of conventional shellac acceptable to the trade. Alkali extraction has, therefore, not been commercially adopted, except in the manufacture of bleached lac.

The colour of shellac is mainly due to the presence of the yellow dye, erythrolaccin (1,2,5,7-tetrahydroxy-4-methyl anthraquinone)<sup>4,5</sup>. Like other hydroxyanthraquinones, erythrolaccin also reacts with alkalies giving a highly coloured solution. Since the original colour of the resin is not regained when lac is reprecipitated, it appears that the change in colour of erythrolaccin is irreversible under these conditions. Regaining of the original colour may be possible if the chemical treatment is not very drastic.

## Experimental procedure

Keeping the above considerations in view, a few preliminary experiments were carried out with *Rangeeni* seedlac. It was extracted with solutions of various alkalies and

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alkaline salts, such as ammonia, caustic soda, sodium carbonate, sodium sulphite, sodium sulphide, sodium silicate, borax, etc. Lac was then precipitated from the alkaline extracts with 5% sulphuric acid solution. Lac obtained in all the cases, except from sodium sulphite extract, was very dark. The one obtained with sodium sulphite though yellow in colour, was soft. In normal practice, sodium carbonate is used for dissolution, but it gives a dark coloured product. It was considered worthwhile trying a mixture of sodium sulphite and sodium carbonate. From experiments using these two chemicals in different proportions, a mixture of 7 parts of sodium carbonate and 3 parts of sodium sulphite per 100 parts of the resin in seedlac was found to give a solid precipitated lac whose hue, though very similar to that of conventional shellac, did not exactly match with that of shellac prepared from the same seedlac by the country (*bhatta*) method. Unlike the other cases, the colour index could also be determined without any difficulty and was 20-21 as against 16 for the *bhatta* shellac.

To further improve the hue and minimize the darkening, other reagents, such as oxalic, tartaric, citric and boric acids and sodium hydrosulphite were tried. With oxalic and citric acids there was only slight improvement, while with tartaric and boric acids there was no improvement at all. Sodium hydrosulphite proved very effective. In conjunction with sodium carbonate and sodium sulphite it produced a shellac with colour index and hue comparable to those of the control lac. In the presence of hydrosulphite, the optimum amounts of carbonate and sulphite were 7 and 3 parts per 100 parts of the resin in seedlac. The optimum amount of hydrosulphite, however, varied with the bleach index; it was 1% on the weight of the resin in the case of seedlac of bleach index 100 or lower, and 2% in the case of samples with bleach index above 100. On increasing the proportion of hydrosulphite beyond 2%, the product obtained was soft.

After standardizing the method with seedlac, it was extended to *molamma* and *kunhi*

and was found equally satisfactory. Since the bleach index of the resin from refuse lacs, whether extracted at 50°C or 85-90°C, is always greater than 100, the amount of sodium hydrosulphite necessary in all cases was 2% on the weight of lac present.

For the preparation of good quality bleached lac from *molamma*, best results are obtained if the alkali extraction is carried out at a lower temperature (50°C) for a longer time (4 hr)<sup>1</sup>. For the reclamation of lac from the refuse lacs, the final product is similar whether the extraction is carried out at 50°C for 4 hr or at 85-90°C for 30 min. Extraction at 85-90°C is, therefore, preferable, as it is less time consuming. When vessels made of metals other than stainless steel or enamelled iron were used for the dissolution of the resin in alkali, invariably a dark product was obtained.

*Recommended procedure*—Anhydrous sodium carbonate (7 parts) and sodium sulphite (3 parts) are dissolved in water (400 parts) in an enamelled iron or stainless steel vessel. The temperature of the solution is raised to 85-90°C and such quantity of the refuse lac or seedlac as contains 100 parts of lac resin is added gradually with occasional stirring. After complete addition, the temperature is maintained at 85-90°C and the stirring is also continued for 30 min for the completion of the extraction.

The extract is then strained through cloth (or preferably in a continuous type rotary strainer of stainless steel wire screen of 100 mesh BS or 15 mesh IS) to free it from insolubles. The filtrate is allowed to cool to 40°C in a wooden vat. Then the requisite amount of sodium hydrosulphite (1 or 2%) is added and the solution allowed to stand with occasional stirring for 15-20 min. It is then diluted with cold water to bring the lac content to 5% and the temperature to 25°C or below. The solution is then acidified under vigorous stirring with excess of 5% sulphuric acid added in the form of a fine spray. With the addition of sulphuric acid the precipitation of lac starts. After allowing sufficient time for complete neutralization, common salt

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Table 1 — Characteristics of reclaimed lac samples obtained by the country method and the present method

Raw material	Hot alcohol solubles %	Reclamation method	Yield on lac content %	Colour index	Life at 150°C min	Flow time at 125°C sec	Acid value	Hot alcohol insolubles %
<i>Molamma</i> (Indian Lac Res. Inst.)	80	Country method*	60†	30.0	34	185 for 7.5 cm	71.2	2.10
			80	32.0	22	350 for 5 cm	71.5	1.96
		Present method		33.0	20	350 for 5 cm	71.6	2.01
				32.5	22	350 for 5 cm	71.5	2.01
do	84	Country method	65†	29.0	29	265 for 10 cm	71.3	2.02
		Present method	81	29.0	29	70 for 5 cm	71.8	1.89
<i>Molamma</i> (commercial)	70	Present method	78	33.0	23	84 for 5 cm	71.1	2.00
<i>Bhole kunhi</i> (commercial)	63	Present method	78	32.0	22	82 for 5 cm	71.6	1.20
<i>Seedlac</i> ( <i>Rangeeni</i> ) (Indian Lac Res. Inst.)	96	Country method	89	16.0	46	125 for 12.5 cm	69.9	0.96
		Present method	87	18.0	28	540 for 12.5 cm	70.1	1.06
				17.5	30	545 for 12.5 cm	69.9	1.02
				17.5	29	545 for 12.5 cm	70.2	1.02
<i>Seedlac</i> ( <i>Rangeeni</i> ) (commercial)	97	Country method	90	16.5	42	75 for 12.5 cm	70.1	1.08
		Present method	88	18.0	28	310 for 12.5 cm	70.6	1.06

\*Hot filtration through cloth bag as in the production of shellac from seedlac.

†Shellac by the country method was prepared with great difficulty for comparison purpose only.

Table 2 — Storage stability of reclaimed lac samples obtained by the present method

Raw material used	Duration of storage months	Life at 150°C min	Flow time at 125°C sec	Hot alcohol insolubles %
<i>Molamma</i>	0	22	600 for 5 cm	1.85
	6	20	650 for 5 cm	1.85
	12	19	650 for 5 cm	1.85
	18	16	660 for 5 cm	1.85
<i>Seedlac</i>	0	28	600 for 12.5 cm	1.02
	6	26	650 for 12.5 cm	1.02
	12	22	705 for 12.5 cm	1.02
	18	22	720 for 12.5 cm	1.02

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(5 parts/100 parts lac) is added to hasten settling of the precipitate. The precipitated lac is then washed thoroughly with water (preferably using a rotary drum washer having drill cloth as filter) till free from sulphate.

After completion of the washing operation, the precipitated lac is suspended in water and heated to boiling, when it melts and forms a lump. The molten mass is then removed from the hot water, cooked in a steam-heated stainless steel pan to remove moisture and made into sheets by squeezing between cold rollers. Alternatively, the precipitated lac may be centrifuged in a basket type centrifuge and dried in trays. In this method, the precipitated lac is obtained in the form of a powder resembling bleached lac.

The above method has given reproducible results with batches of seedlacs and refuse lacs containing 5, 10 and 20 kg of lac resin.

### Results and discussion

The yield of reclaimed lac was nearly 80% on the lac content in the case of refuse lacs and 90% in the case of seedlacs. The characteristics of a few samples of reclaimed lac are given in Table 1. It is seen that the lacs produced by the new process have a slightly higher colour index than that of the conventional hand-made shellacs. The hue, however, is very similar. The hot alcohol insolubles and acid values are also of the same order. The life and flow are, however, poor.

Samples stored under laboratory conditions for 18 months remained soluble in alcohol (Table 2), indicating their good storage stability. Even after 18 months, there was no change in the percentage of hot alcohol insolubles, although life and flow were affected to a small extent.

The wax content of lacs reclaimed from refuse lacs and seedlacs was 4.0-4.8 and 3.8-4.1% respectively.

*Film properties of reclaimed lacs*—Reclaimed lacs obtained by the new method were found to contain 1.05-1.25% combined sulphur. The possible influence of this sulphur on the film properties of the lacs was examined. The scratch hardness and water resistance of the films of these lacs were found to be similar to those for original lacs.

Bleached lac is becoming popular in this country and it is usually marketed in powder form. Accordingly, if the reclaimed lacs instead of being converted into flakes are dried in powder form and sold as such, the process will be a little more economical. The lacs thus prepared from refuse lacs are dark coloured, but since there is demand for dark coloured lacs for certain industries there can be no difficulty regarding their disposal.

The new process needs very little capital outlay and, therefore, its commercial adoption for the reclamation of lac from refuse lacs can improve the overall economy of the Indian lac industry.

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### References

1. GHOSH, A. K. & SEN GUPTA, S. C., *Res. & Ind., New Delhi*, **12** (1967), 171.
2. GIDVANI, B. S. & KAMATH, N. R., *London Shellac Research Bureau, Tech. Paper No. 26*, 1945.
3. GHOSH, P. K. & BHOWMIK, T., *Annual Reports, Indian Lac Research Institute* (1954-55), 71; (1955-56), 60; (1956-57), 42; (1957-58), 17.
4. YATES, P., MACKAY, A. C., PANDE, L. M. & AMIN, M., *Chem Ind.*, **33** (1964), 1991.
5. BHIDE, N. S., RAMA RAO, A. V. & VENKATARAMAN, K., *Tetrahedron Lett.*, **7** (1965), 33.