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Dewaxing of Lac in Aqueous Medium

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A simple method for dewaxing lac in aqueous medium is described. The method consists in adding lac wax (10% on the weight of lac) to the lac solution, heating it to boiling and then allowing it to cool till most of the wax present in lac deposits in hard and compact form at the top and can be easily removed. The optimum conditions for separation are : Temperature of dissolution, 90±5°C; amount of lac wax to be added, 10% on the weight of lac; temperature of cooling, 10±2°C; period of cooling, 1 hr; concentration of solution for separation, 10%. Kieselguhr (5% on the wt of lac) and rayon have been found to be efficient filter aid and filtering medium respectively for the filtration of aqueous lac solution containing traces of wax. The method is applicable to all types of lac and takes one-fourth the time needed in the conventional methods.

SHELLAC contains a natural wax (4-5%) which acts as a plasticizer for the resin. Solutions of shellac are cloudy due to the presence of this wax, as it is insoluble in the common lac solvents and has to be eliminated to obtain clear varnishes and bright films.

There is at present increasing demand for dewaxed lac, as practically all new uses developed for lac are based on it. The conventional processes[1-5] for the production of dewaxed lac consist in dissolving seedlac in spirit, in which lac resin alone is soluble but not the wax, refrigerating, filtering off the solution and reclaiming the lac by distilling off the solvent. In view of acute shortages of spirit in India, it is necessary to switch over to dewaxing in aqueous media.

In aqueous media[6-8], the separation of wax is usually carried out by filtering an alkaline solution of lac through a cloth bag. This is a time-consuming process. Therefore, a study aimed at reducing the filtration time was undertaken.

Experimental procedure

Lac solution was prepared by extracting 100 g seedlac with 400 ml water containing 10 g anhydrous sodium carbonate at 90±5°C for about 30 min. The solution was strained hot to remove insolubles through a 100 mesh brass sieve and the residue washed with 100 ml hot water. The volume was made to 500 ml, the extract heated to boiling to melt the wax, and the solution allowed to stand undisturbed for 3–4 hr. Most of the wax rose to the surface and formed a cake at the top. A small quantity, however, remained suspended in the upper layer of the solution, which formed about 15% of the solution. The lower 85% of the solution (wax content about 1%), which was comparatively clear, was run off and filtered through a drill cloth bag unless mentioned otherwise. Dewaxed lac was reclaimed from the filtered solution by lowering its temperature below 20°C, precipitating it with dil. sulphuric acid and subsequently washing it free of acid.

The determination of wax was carried out according to the method prescribed in IS : 16–1956 (ref. 9).

Results and discussion

Various temperatures and alkalies were tried for the dissolution of lac to examine the mode of dispersion of wax. It was observed that at lower temperatures, the period of dissolution was higher and the dispersion of wax in the solution after cooling was heterogeneous and scattered throughout. At higher temperatures, the solution could be prepared conveniently

Table 1 — Effect of temperature of dissolution on dispersion of wax

Temp. at which solution was prepared °C	Period required for preparing solution hr	Period required for filtration through cloth bag hr	Dispersion of wax in solution after cooling overnight
28±2 (room temp.)	7	24	Scattered throughout
50±2	3	20	do
60±2	2	15	do
80±2	1	4.5	Scattered, but more wax in the upper layer
90±2	0.5	3.5	Wax mostly in the uppermost layer

Table 2—Effect of addition of lac wax on the rate of filtration

(Conc. of lac solution, 20% wt/vol.)

Wax added on the wt of lac %	Time taken for filtration hr
Nil	3.5
2.5	2.5
5.0	2.4
7.5	2.25
10.0	2.0
15.0	2.0

and the collection of wax in the solution after cooling was mostly confined to the uppermost layer (Table 1). The filtration was found to be quicker in the latter case. The optimum temperature for dissolution was found to be 90±5°C.

It was also observed that in a solution containing sodium carbonate solution, the wax collected was in more compact form and the filtration faster than in solution containing sodium bicarbonate.

In another approach tried, an outside non-reactive substance (heavier or lighter than the lac solution) was added and the solution boiled. It was expected that this substance might take the wax in the solution with it and would either settle down or come up to the surface after cooling. For this purpose, barytes (sp. gr. 4.3-4.7) was tried.

Lac solution was boiled with different types of barytes (up to 50% on the lac content) and cooled, when barytes settled down at the bottom leaving almost a clear solution above it. The clear solution was decanted in each case (75% of the total volume) and filtered through cloth bag. The filtration was found to be quite fast, but the filtrate in each case after overnight settlement showed the presence of barytes passing into the filtrate.

The use of barytes was, therefore, discontinued. Instead, lighter substances, such as paraffin wax, lac wax, etc. were tried and their effect examined. When paraffin wax was added to lac solution, and the solution boiled and cooled, it was observed that it did not mix well with the lac wax present in the solution and so did not improve the deposition of lac wax in the form of a cake for removal. On the contrary, lac wax itself, if added from outside, was found to help in the coagulation of lac wax present in the solution. Different amounts of lac wax were added to lac solution and the solution boiled for 2–3 min and then cooled. It was found that additional lac wax mixed intimately with the wax in the solution and formed a hard and firm cake at the surface, leaving almost a clear solution below. The clear solution (almost 85% of the total volume) was drained out and filtered through drill cloth. The rate of filtration was faster with this solution than with the usual lac solution. The optimum amount of lac wax to be added was found to be 10% on the weight of lac present in the solution (Table 2).

Next, the effect of temperature of cooling was studied, keeping the duration of cooling constant. Lac solution with 10% additional wax was heated to boiling, cooled at different temperatures for 4 hr and 85% of the total volume of the solution (mostly clear) was drained out and filtered. Best results were obtained at 10±2°C.

To find out the optimum cooling period, the solution was cooled for different durations and the corresponding rate of filtration noted. It was found that 1 hr cooling of the solution at 10±2°C was sufficient to get maximum wax separation and a satisfactory rate of filtration.

The effect of diluting the solution on the rate of filtration for obtaining the same amount of lac content in the filtrate was also studied. The optimum concentration of the solution for rapid filtration was found to be 10%.

To further enhance the rate of filtration, various filter aids, such as kieselguhr, paper pulp and *ghungi* (lac refuse, powdered to 40 mesh) were tried. Kieselguhr (5% on the wt of lac) was found to be the best agent in this respect.

The effect of various filtering media, such as rayon, terylene, turkish towel, nylon and the usual drill cloth, on the rate of filtration and on the wax content of the filtrate was also studied. It was found that rayon gave the lowest wax content in the resultant product and a reasonably fast rate of filtration.

It was found that the upper layer of the aqueous lac solution (15%) left over could be worked up with the subsequent batch.

The method developed is applicable to all types of lac, and takes one-fourth the time needed in the conventional methods.

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