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ISOLATION OF ALEURITIC ACID WITH THE TREATMENT OF DIFFERENT PERCENTAGES OF SODIUM HYDROXIDE SOLUTION FROM LOCAL VARIETIES OF LAC

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Abstract

Aleuritic (9, 10, 16-trihydroxy palmitic acid) a unique acid containing three hydroxyl groups of which two are attached to adjacent carbon atoms, isolated from local varieties of seed lac, shellac and de-waxed shellac respectively with the treatment of different percentages (10%, 15%. 20%, 25%, 30%) of sodium hydroxide solution. From the experiment, it was observed that at optimum condition, the use of 20% sodium hydroxide solution permitted the best isolation of aleuritic acid. The isolated aleuritic acid was confirmed by Fourier Transform Infrared Spectroscopy (FTIR).

Key words: Seed lac, Shellac, De-waxed shellac, Aleuritic acid, Saponification, Comparative study.

Introduction

Seed lac, Shellac and De-waxed shellac are an important source of aleuritic acid. They contain about 30% aleuritic acid which is present in the respective lac in the form of their lactides or lactones (Harris, *et al.*, 1928). Aleuritic acid is the first crystalline compound isolated from lac. It is a white crystalline solid melting at 101.5 °C which is an attractive material for the preparation of microcylic musk -like lactones and ketones (Bose, *et al.,* 1963).

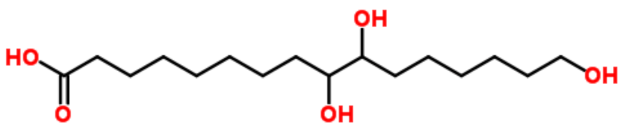

Fig. 1: Aleuritic acid (9, 10, 16-trihydroxy, palmitic acid).

It is also a valuable starting material for the preparation of transparent water clear adhesive, plasticisers and resins in combination with other chemicals (Sen, *et al.*1948). Aleuritic acids are useful in compounding up to 60% with cellulose ethers and esters for the preparation of lacquers, plastics and fibres (Sartort, 1945). Aleuritic acid is soluble in hot water and in alcohol, acetone and ammonia in the cold (Tschirch, *et al.,* 1999). It is also dissolved on warming in ethyl acetate, benzene, toluene, chloroform and potassium hydroxide but not in ether and carbon disulphide (Bhattacharya, 1935). Aleuritic acid condensed with equimolecular portion of phthalic anhydride and glycerol at 180-190°C gives a clear, flexible transparent product called glass-clear cement (Sen, *et al.,*1959). Glass clear cement shows very good adhesive property specially in glass to glass bonding.

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Several studies were carried out by Gidvani (1944), Puntumbekar (1956), and Hossain *et al.*(1987) for the isolation of aleuritic acid from de-waxed shellac, hard resin portion of lac and from seed lac respectively. But none of the methods were found to be suitable for the isolation of aleuritic acid. So, the present investigation was carried out with a view to finding out a suitable method for the isolation of aleuritic acid from the local varieties of lac.

Materials and Methods

Seed lac and shellac were collected from local lac industry of Baliapukur, Rajshahi and de-waxed shellac was collected from natural products Research Division, BCSIR, Laboratories, Rajshahi for conducting the isolation of aleuritic acid. Sodium hydroxide, sulphuric acid and methyl alcohol were purchased from a local market and used without further purification. Aleuritic acid was isolated from seed lac, shellac and de-waxed shellac in percentage of alkali are essentially same, only a representative method has been described here.

Seed lac from local varieties 'Baisaki' and 'katki' was used; Each variety was dissolved in 100 ml different concentrations (10%, 15%, 20%, 25% and 30%) of Sodium hydroxide solution. 100 g of seed lac was added to the solution with slow and constant stirring and boiled until the lac saponifies. After saponification, the mixture was allowed to stand at room temperature for 24 hours while the crystal of sodium aleuritate was separated out. It was then diluted with 100 ml of 25% Sodium hydroxide solution to facilitate the filtration. The mixture was then filtered through Buckner funnel using sand as a filtering aid. The residue was collected and washed with 25% NaCl solution to remove alkali from the solution. The residual crude sodium aleuritate was then dissolved in 100 ml of boiling water and suction filtered under hot condition. The filtrate was then acidified with 20-30 ml of 10% H_2SO_4. Here the aleuritic acid was left undissolved while all other acid went into solution (Bose *et al.,* 1963). Then it was filtered through a filter paper. The waxy residue on crystallization from aqueous alcohol yielded aleuritic acid m. p. 101 - 103° C (Harris et al., 1922) at methyl ester being 70 - 72° C (Shamsuzzaman et al., 1963). The precipitated aleuritic acid was collected and purified and nearly 25% aleuritic acid was yielded. Aleuritic acid was also isolated from shellac and de-waxed shellac following the same method. IR spectra of aleuritic acid were recorded on a Simadzu, Model-8900, FTIR spectrophotometer as KBr pellets.

Results and Discussion

Aleuritic acid was isolated from seedlac, shellac and de-waxed shellac and its yield of percentages are shown in Table-I, 2 and 3 respectively. The differences between the yield of aleuritic acid obtained from the varieties 'baisaki' and 'katki' is negligible.

In the first set of experiments, maximum yield of aleuritic acid from seed lac was optimized. It was observed that with the concentration of 10% of sodium hydroxide solution the yield of aleuritic acid was 15.8%, whereas 15% sodium hydroxide yielded 18.5% aleuritic acid. However, 20% sodium hydroxide yielded highest 25.5% aleuritic acid. But when the percentage of sodium hydroxide solution was further increased to 25% the yield of aleuritic acid was decreased to 20.3% and In 30% sodium hydroxide solution only 18.5% aleuritic acid was produced. It has been, however, concluded that the yield of aleuritic acid was depended on the percentage variation of sodium hydroxide solution. Respective data has been presented in Table 1 and figure 1.

In another set of experiment, 20% sodium hydroxide has yielded maximum aleuritic acid (27.2%) from both varieties of seed. Respective data has been shown in Table 2 and figure 3.

In de-waxed shellac, 20% sodium hydroxide again proved best for maximum yield of aleuritic acid. From the Table - 3, it was also found that the maximum yield of aleuritic acid (28.7%) was obtained from de-waxed shellac variety corresponding the same sodium hydroxide solution (20%).

Among the three different sources, highest yield of aleuritic acid obtained from de-waxed shellac (28.7%) whereas minimum yield was from seed lac (25.5%). Intermediate result from shellac variety depending on the percentage variation of sodium hydroxide solution.

Infrared spectra

The IR spectra provide valuable information regarding the nature of functional group attached to the aleuritic acid molecule. The appearance of a broad strong band in the IR spectra of the molecule in 3402cm^{-1} is assigned to the O-H stretching vibration of hydroxyl group of the molecule. The spectrum of the molecule shows –C=O bands in the region (1708-1651) cm^{-1}, which is assigned for the carbonyl group of the carboxylic group of the molecule. The C–O stretch appears in the region (1311-1130) cm^{-1}, and the O–H bend is in the region 1406 cm^{-1} and 1057 cm^{-1}. The FTIR spectrum of the aleuritic acid shows the band related to aliphatic C-H stretching in the range (2924-2856) cm^{-1}. The characteristics FTIR bands of the isolated aleuritic acid are shown in Table 4.

Table 1. Yield of aleuritic acid from seed lac

No. of Expt.	Percentage of sodium hydroxide (%)	Yield of aleuritic acid from seed lac (%)
1	10	15.8
2	15	18.5
3	20	25.5
4	25	20.3
5	30	18.3

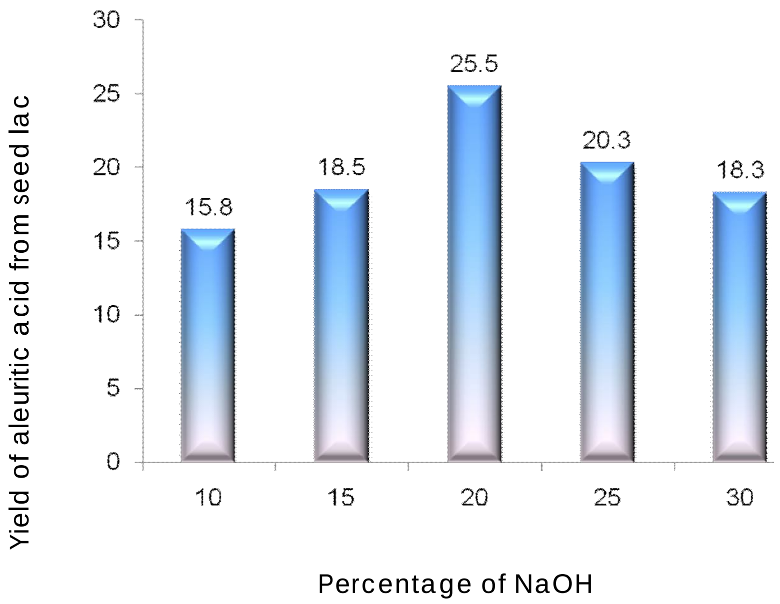

Fig. 1: The effect of different percentage of NaOH solution on the yield of aleuritic acid from seed lac

Table 2. Yield of aleuritic acid from shellac (%)

No. of Expt.	Percentage of sodium hydroxide (%)	Yield of aleuritic acid from shellac (%)
1	10	16.2
2	15	18.5
3	20	27.5
4	25	25.2
5	30	22.3

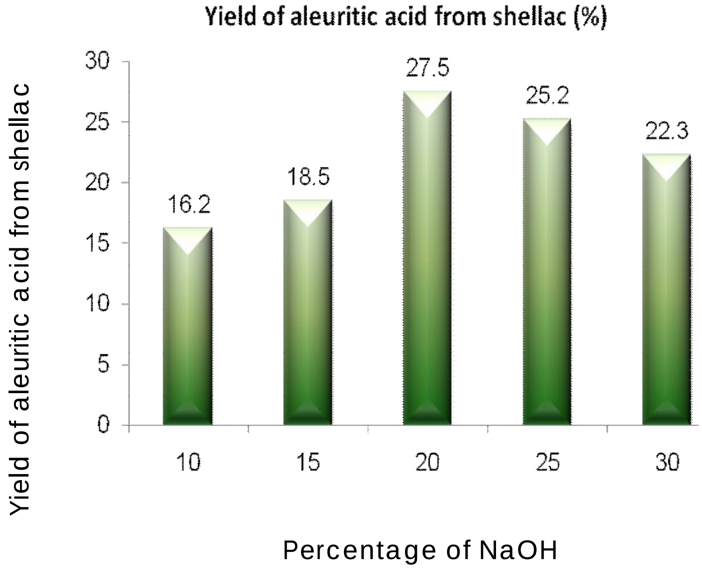

Fig. 2: The effect of different percentage of NaOH solution on the yield of aleuritic acid from shellac

Table 3. Yield of aleuritic acid from de-waxed shellac (%)

No. of Expt.	Percentage of sodium hydroxide (%)	Yield of aleuritic acid from de-waxed lac (%)
1	10	18.2
2	15	20.5
3	20	28.7
4	25	25.2
5	30	22.4

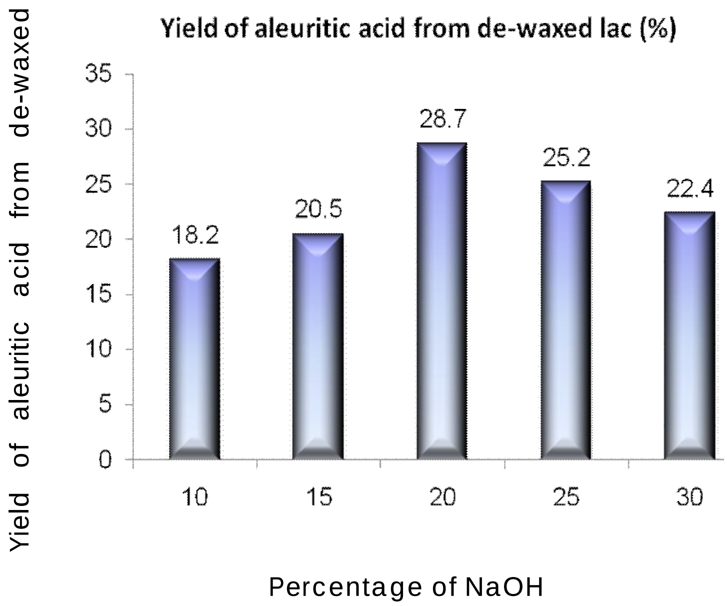

Fig. 3: The effect of different percentage of NaOH solution on the yield of aleuritic acid from de-waxed shellac

Table 4. Characteristic FTIR bands of the compound Aleuritic acid

Sl. no.	Peak cm-1	Intensity
1.	669.32	53.7235
2.	1057.03	50.1864
3.	1130.32	55.9355
4.	1311.64	43.7644
5.	1406.15	45.4159
6.	1651.12	42.7105
7.	1708.99	41.8397
8.	2349.38	43.7622
9.	2856.67	27.8553
10.	2924.18	21.6551
11.	3402.54	17.2908

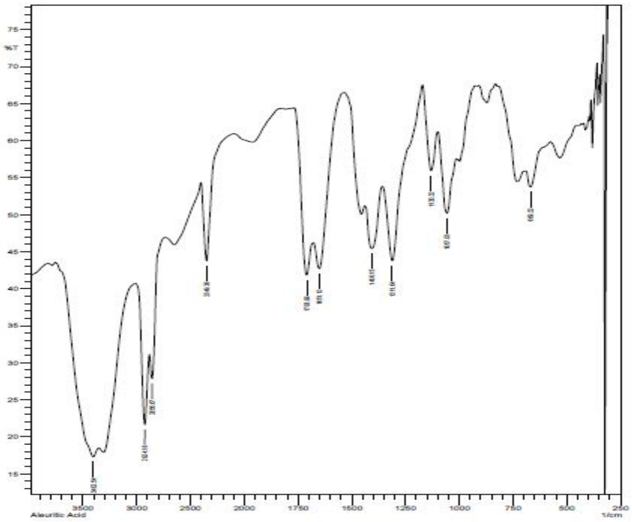

Fig. 4: FTIR spectrum of aleuritic acid

Conclusion

Aleuritic acid which were isolated from the local varieties of seed Lac, shellac and de-waxed shellac respectively with the treatment of different percentages (10%, 15%. 20%, 25%, 30%) of sodium hydroxide solutions has a continuous growing demand in the fields of perfumery and pharmaceuticals due to it being an excellent starting material for the synthesis of civetone, ambrettolide, isoambrettolide etc, which has the musk like odour.

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