S1. No. 236

Research and Industry
Vol. 24, December 1979, pp. 235-237

Cation Exchange Resin from Shellac

A RAHMAN, P R BHATTACHARYA & B B KHANNA
Indian Lac Research Institute, Namkum, Ranchi

Received 17 December 1979; accepted 25 December 1979

The preparation and characteristics of a cation exchange resin developed from sulphonated lac, resorcinol and paraformaldehyde are described. The resin has good ion exchange capacity, thermal stability and chemical resistance.

Cation exchange resins are used widely in the treatment of water for use in chemical and pharmaceutical industries, in metallurgy as well as in chemical analysis work. These resins have a polymeric base and contain sulphonic, carboxyl or hydroxyl groups. Shellac, a natural resin, is composed of a number of aliphatic and hydro-aromatic hydroxy acids, which are present as lactones, lactides, inter-esters and ethers[1,2]. It has been suggested that an average molecule of lac consists of an equivalent of a little more than one free carboxyl, three ester and five hydroxyl groups, apart from one unsaturated linkage and an aldehydic group, partly free and partly combined. The presence of hydroxyl and carboxyl groups in shellac makes it suitable for the preparation of cation exchange resin. An attempt in this direction was first made by Dhar[3]. However, the ion exchange capacity of the resin prepared was low. The present paper presents the results of a study relating to the preparation of a cation exchange resin from shellac having high capacity. The properties of the resin have also been investigated.

Experimental procedure

Materials—Dewaxed lac (DL) was obtained from M/s Angelo Bros., Calcutta. The chemicals used were of pure quality.

Sulphonation of lac—Two techniques for sulphonation of lac, one worked out in the laboratory of the Shellac Export Promotion Council (SEPC), Calcutta[4] and the other at the Central Leather Research Institute (CLRI), Madras[5], were made use of. The same are briefly described below.

Method 1 (*SEPC method*)—Concentrated sulphuric acid (80 g) was added gradually to a solution of dewaxed lac (100 g) in rectified spirit (250 ml) at room temperature. The precipitated lac was filtered, washed free from acid with distilled water and dried. The yield was 95-97%.

Method 2 (*CLRI method*)—Dewaxed lac (100 g) was dissolved in rectified spirit (100 ml) and to the solution, concentrated sulphuric acid (89 g) was added gradually, the rate of addition being adjusted to keep the temperature of the system at 70-80°C. The addition took 30-45 min. The temperature was maintained at this level for another 2 hr. Next day, the product was salted out with sodium chloride solution (30%). It was washed with distilled water till free from sulphuric acid and neutralized to *p*H 7 with sodium hydroxide solution (30%). The product was thereafter dried. The yield was 55-60%.

Preparation of cation exchange resin—Shellac or sulphonated lac (22 g) was dissolved in sodium hydroxide solution (4%, 100 ml) and boiled with paraformaldehyde (1.5 g) for 1 hr. Thereafter resorcinol (11 g) was added and boiling continued for another 1 hr. The solution was cooled to 50°C and another lot of paraformaldehyde (1.5 g) was added with stirring. The mass was boiled again and cooled slowly; two more batches of paraformaldehyde (1.5 g) were added with stirring at intervals of 1 hr, when it formed a brown gel within a few minutes. The gel was broken and further hardened in an oven at 100-105°C for 4 hr. The resin was washed to remove colouring matter, first with water and subsequently with spirit and sodium carbonate solution (4%), equilibrated with hydrochloric acid (4%) and dried. A black, fine mesh resin of irregular shape was obtained which was leached with 0.1 *N* hydrochloric acid for 24 hr. The resin was washed free of acid and air dried at room temperature, when an H-form resin was obtained. The yield was 60-65%. The resins were tested for their cation exchange capacity following the methods described by Shah and Bafna[6].

Table 1—Cation Exchange Capacity of Resins
[The capacity values are in m eq/g dry resin]

Resin	In the presence of NaCl at 25°C, *p*H 7	From $BaSO_4$ determination	From limiting exchange with $BaCl_2$ solution
Shellac	0.35	0.34	0.34
Sulphonated lac (Method 1)	1.51	1.50	1.52
Sulphonated lac (Method 2)	2.99	2.98	2.97

Table 2—Characteristics of Ion Exchange Resin from Sulphonated Lac

Moisture content, %	9.2
Salt splitting capacity, m eq/g	0.195
Total capacity, m eq/g	4.58
Wet absolute density, g/ml	1.228
Degree of column utilization, %	71
Thermal stability	Tolerable
Colour throw	Negligible
Rate of exchange	Slow

The data presented in Table 1 show that the ion exchange resin prepared from sulphonated lac (Method 2) gives encouraging results. The ion exchange capacity was still higher when a catalyst (ammonium chloride) was used. The resin was got evaluated at another reputed research institute. The resin was sieved and the fraction −83+30 ISS mesh size was selected by them for characterization. The characteristics of the product are given in Table 2.

The results in Table 2 indicate that the resin has good capacity, thermal stability and chemical resistance (negligible colour throw) but requires improvement in respect of column utilization (which should be 80%) and rate of exchange. Work to improve the resin in respect of these properties is in progress.

Since resorcinol is very costly, attempts were also made to substitute it with cashewnut shell liquid (CNSL) which is much cheaper and contains phenolic compounds. A number of approaches were tried, after which it was found that a gel could be obtained by dissolving sulphonated shellac in sodium hydroxide solution and adding CNSL followed by formaldehyde. The ratio 1.5 parts CNSL: 1 part lac was found to be optimum. The cation exchange capacity of the resin obtained was, however, somewhat low (2.3 m eq/g). One advantage of this approach is that the cost of the product is very low.

The results of the present study indicate that although the ion exchange resin developed from shellac requires improvement in some respects, it has shown promise for the use of shellac in the field of ion exchangers and a new field of its application may be opened up.

Acknowledgement

The authors wish to express their thanks to Dr T.P.S. Teotia, Director of the Institute for his keen interest during the course of the work.

References

1 Bose P K, Sankaranarayanan Y & Sengupta SC. *Chemistry of lac* (Indian Lac Research Institute, Ranchi) 1963.

2 Misra G S & Sengupta S C, cited in *Encyclopedia of polymer science and technology*, Vol. 12, edited by Norbert H Bikales (John Wiley & Sons., Inc., New York), 1970, 419-40.

3 Dhar A N, *J scient ind Res*, **13B** (1954) 384.

4 Shellac Export Promotion Council, Calcutta, *Supplement to Technical Notes on New Uses of Shellac* (1960).

5 Central Leather Research Institute, Madras, Private communication.

6 Shah H A & Bafna S L, *J Indian chem Soc*, **29** (1952) 189.