

Styrene Divinylbenzene-Based Strong Acid Ferromagnetic Cation Exchanger

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Synopsis

A strong acid ferromagnetic cation-exchange resin based on styrene divinylbenzene polymer has been prepared and characterized.

INTRODUCTION

The synthesis of a ferromagnetic cation-exchange resin based on styrene and divinylbenzene polymer has been reported in the literature.^{1,2} However, details of the preparation of such a resin using flue dust as a magnetic particle have not so far been reported. This paper describes the synthesis and characterization of a strong acid ferromagnetic cation exchanger.

EXPERIMENTAL

Preparation of Styrene Divinylbenzene Copolymer by Incorporating Flue Dust Containing Iron Silicate

Styrene and divinylbenzene were washed with 4% sodium hydroxide solution to remove all the inhibitors present. Flue dust was also treated with concentrated hydrochloric acid to remove the acid-soluble material.

Polyvinyl alcohol (dispersing agent, 2 g) was dissolved in 300 ml of boiled distilled water. This solution was transferred to a 500-ml round-bottomed three-neck flask, fitted with a stirrer, a thermometer, and a condenser. To this was added a mixture of 58.17-ml styrene, 21.83-ml (55%) divinylbenzene, 0.866-g benzoyl peroxide, 53.0-ml *n*-amylalcohol ($F_m = 0.6$), and 1.0-g flue dust. The reaction mixture was heated at 85°C with constant stirring for about 3 hr until

TABLE I
Capacity and Magnetic Susceptibility of the Samples^a

Salt-Splitting Capacity (meq/g absolutely dry resin)	Total Capacity (meq/g absolutely dry resin)	Magnetic Susceptibility of Polymer (cgs/g)	Magnetic Suscepti- bility of Resin (cgs/g)
1.85	1.95	13.9×10^{-6}	$8.3-8.7 \times 10^{-6}$

^a Sulfonation temperature, 55°C; polymer acid ratio, 1:12 (w/w); $F_m = 0.6$.

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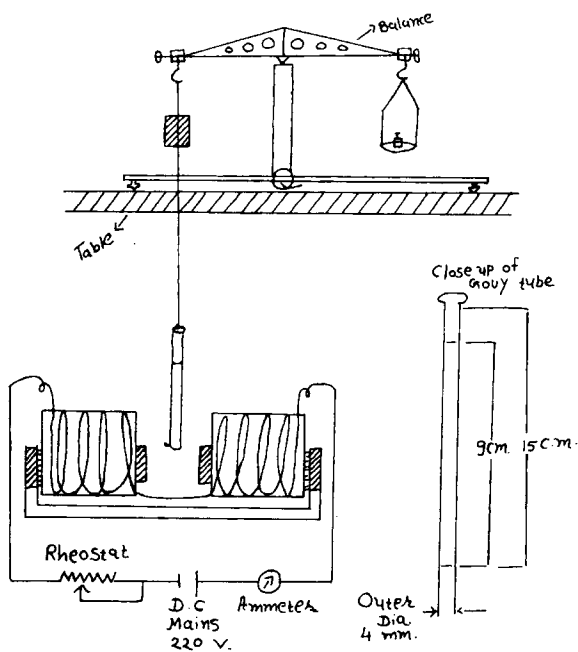


Fig. 1. Schematic drawing of the magnetic balance.

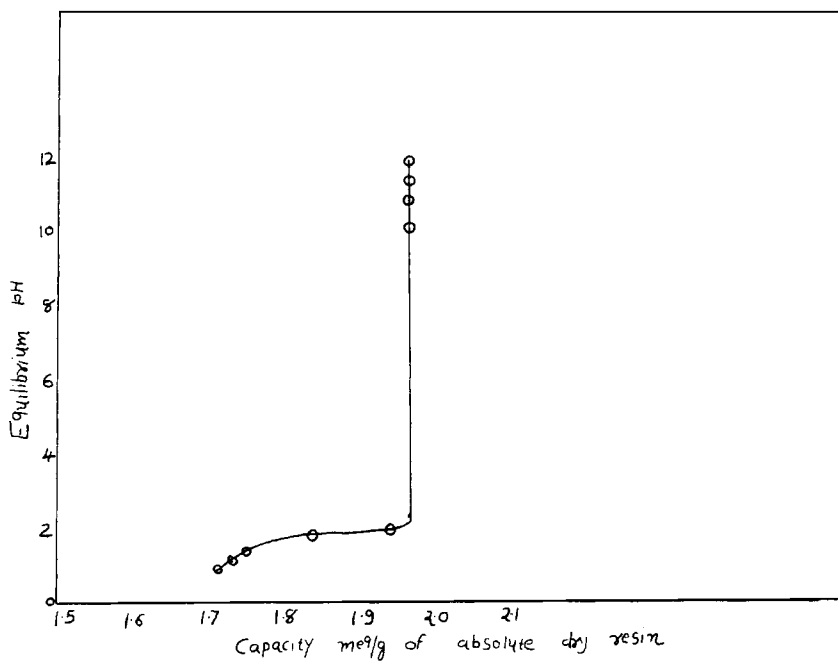


Fig. 2. pH titration curve of the ferromagnetic resin. Conditions: sulfonation temperature, 55°C; quantity of resin, 0.5 g (air-dried); particle size, 20 + 40 BSS mesh; equilibrium period, 24 hr at 28°C; solutions: 1.0N NaCl and 0.1N NaOH in 1.0N NaCl.

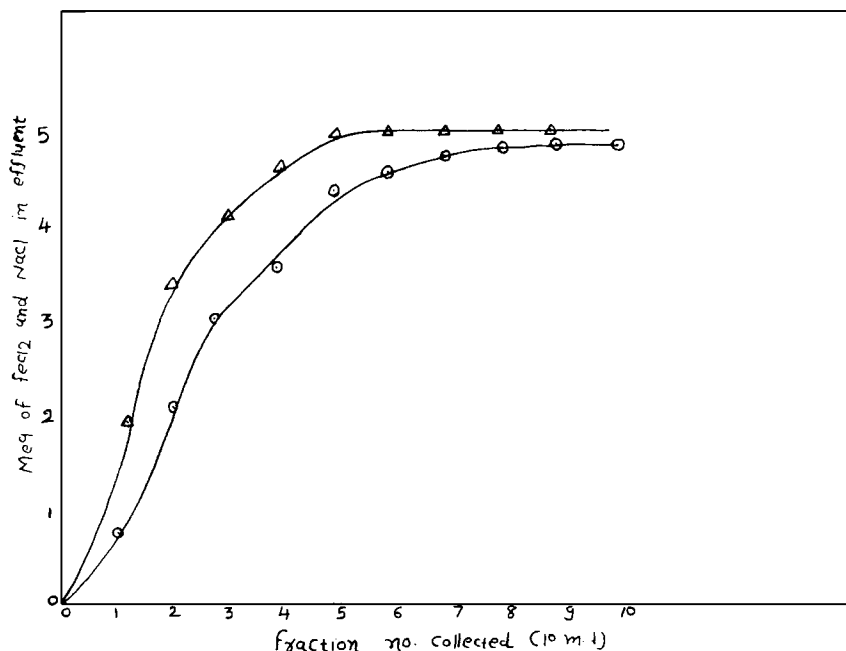


Fig. 3. Effect of separation of NaCl (Δ) and FeCl_2 (\odot) in 10-ml effluent by the ferromagnetic resin. Particle size: 20 + 40 BSS mesh; solutions: 0.5N FeCl_2 in 0.5N NaCl. Flow rate: 10 ml/hr.

the polymer was formed. This was removed from the reaction vessel, filtered, washed with distilled water, and boiled again for $\frac{1}{2}$ hr with an excess of distilled water. Then the hard mass was cooled, filtered, washed with distilled water, and dried at room temperature. The product was sieved to proper mesh size, and the sieved polymer was stored for further experimental work. Yield was 95%. The above experiment was repeated using 0.5 and 0.7 F_m .

Sulfonation of Polymer. Five grams of the above polymer were swollen by keeping it in contact with nitrobenzene for 24 hr. The swollen polymer was sulfonated for 8 hr at 100, 55, and 35°C in different experiments. The reaction mixture was then cooled, washed with different concentrations of sulfuric acid (80%, 60%, 40%, and 20%), and finally with distilled water to remove the excess of acid. The resin was filtered, dried, sieved to proper mesh size, and stored for further experimental work.

Conditioning. The sulfonated resin as prepared above was conditioned by following Kunin's standard procedure.³

Capacity and Moisture. The ion-exchange capacity and moisture content of the resin were determined by Kunin's method.³

pH Titration Curve. The pH titration study was carried out by the standard procedure.³

Magnetic Susceptibility. Magnetic susceptibility of polymer and resin was determined by the Gouy method.⁴ The complete apparatus for the measurement of magnetic susceptibility of the polymer and resin was set up as shown in Figure 1.

Ferrous Ion Uptake Studies. The following two systems were studied by columnwise operation at a constant flow rate:

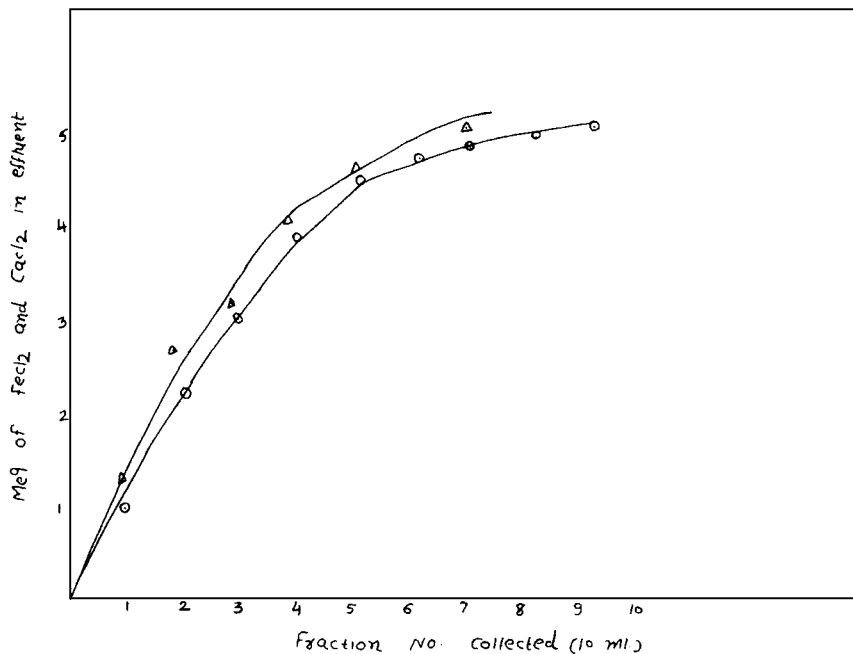
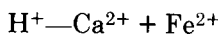
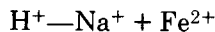


Fig. 4. Effect of separation of CaCl_2 (Δ) and FeCl_2 (\odot) in 10-ml effluent by the ferromagnetic resin. Particle size and flow rate same as in Fig. 3. Solution used: 0.5N CaCl_2 in 0.5N FeCl_2 .



RESULTS AND DISCUSSION

From the results summarized in Table I, the value obtained for the magnetic susceptibility of the polymer (13.9×10^{-6}) agreed with the theoretical value within experimental error.

The pH titration curve presented in Figure 2 shows that at 1.95, the exchange capacity of the resin is independent of pH. The nature of the curve is similar to that of an ordinary strong acid cation exchanger like Zeo Karb 225. This indicates the magnetic susceptibility of the resin has no effect on its pH titration curve, which depends on the exchanging groups present in the resin (Ref. 5, pp. 81-82).

Figures 3 and 4 indicate that when the ferromagnetic resin was used to separate a mixture of sodium chloride and calcium chloride with ferrous chloride, the separation was more significant with sodium chloride (Fig. 3). This can be explained by the fact that at ordinary temperature the extent of exchange increases with the increasing valency of the exchanging ion (Ref. 3, p. 32).

It was also observed experimentally that less contact time of the resin with the mixture of calcium chloride and ferrous chloride does not help in better separation. This may be due to the low value of the magnetic susceptibility of the resin.

References

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