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Assessment of the exchange parameters of Amberlite IR-45 (OH) resin from its elution with saturated Na₂SO₄

Muhammad B. Ibrahim¹ and Abdullahi Mustapha²

¹Department of Pure and Industrial Chemistry, Bayero University, P. M. B. 3011, Kano, Nigeria
E-mail: bashirmbi2@yahoo.ca

²Department of Chemistry, Kano State University of Science and Technology, Wudil

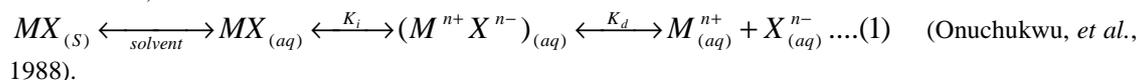
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ABSTRACT: Simple quantitative technique has been developed for assessing the exchange parameters of Amberlite IR – 45 (OH) using column chromatographic techniques from the displacement of exchangeable hydroxyl ions of the resin by sulphate ions. The results obtained showed retention time of 33min, retention volume of 56.5cm³, void volume of 18cm³, and exchange capacity of 41mmole/g and exchange site density of 2.4682 x 10²² sites /g.

Key Words: Resin; Solubility; Ion exchange; Column chromatography; Elution; Amberlite.

Introduction

The term solubility simply refers to the amount of solute needed to form a saturated solution in a given amount of solvent at a given temperature (James, 1990). Upon a critical look of the dissolution process of the solute, a number of dynamic equilibria are involved, which for a generalized electrolyte MX can be outlined thus;



In ion exchange chromatography (IEC), the free exchangeable ion of the synthetic exchanger (resin) is exchanged with the dissociated ions of the mobile phase in a reversible manner. For a resin having OH⁻ as the exchangeable ion and a stationary polymeric backbone **P**, on elution with a mobile phase having anion **Xⁿ⁻**, the following exchange takes place:



Further estimation of the amount of nOH⁻_(aq) serves as a measure of the amount of Xⁿ⁻_(aq) exchanged by the resin and by extension to the concentration of Xⁿ⁻_(aq) dissolved in the mobile phase. In this study

¹ Author for Correspondence

exchange parameters like; exchange capacity, exchange (active) site density, retention time (t_R), retention volume, (V_R) and void volume (V_m), were assessed for the resin under investigation.

The exchange capacity of the resin, which is dependent on the total number of the ion-active groups per unit weight of the resin expressed in milliequivalents per gram of dry resin or milliequivalents per cm^3 of wet resin as reported by Fritz *et al.*, (1979), and by Rendle *et al.*, (1974), is expressed by the relation:

$$\text{Exchange Capacity} = \frac{M_{\text{titrant}} \times V_{\text{titrant}}}{\text{Weight of Adsorbent}} \dots\dots\dots(3)$$

where M_{titrant} and V_{titrant} are the molarity and volume of the titrant, respectively.

On the other hand, the exchange (active) site density, assuming all the exchangeable sites of the resin are available to the ions in the mobile phase for exchange, is defined by the relation:

$$\text{Exchange (Active) Site Density} = \text{Exchange Capacity} \times L g^{-1} \dots\dots\dots(4)$$

where L is Avogadro's number.

Robert (1983) defined the retention time (t_R) as the time required for the mobile phase to sweep a sample component off the stationary phase, and the retention volume (V_R) as the volume of the mobile phase needed to elute a sample component from the column, and is related to the retention time by:

$$V_R = F_o t_R \dots\dots\dots (5) \text{ with } F_o \text{ representing the flow rate of the mobile phase expressed in units of volume per time.}$$

The void volume is expressed either from,

$$V_m = F_o t_m \dots\dots\dots(6) \text{ where } t_m \text{ is the time for average mobile phase molecule to flow from one end of the stationary phase to the other, or using the relationship adopted by McCormick } et al., (1980) \text{ as:}$$

$$V_m^{\text{max}} = \frac{M_{\text{CCl}_4} - M_{\text{meOH}}}{\rho_{\text{CCl}_4} - \rho_{\text{meOH}}} \dots\dots\dots(7)$$

where M and ρ are the masses and densities of the column equilibrated with CCl_4 and methanol, respectively.

Materials and Methods

Prior to usage, the resin was regenerated by washing severally with 2M NaOH and HCl alternately and was allowed to stand in 2M NaOH solution for 30mins (DES, 2001) and was finally washed with distilled water until the washings were neutral to phenolphthalein (Wilson, 1968). The resin was air dried, and in an oven at 60°C for 1 hour (Sigma, 1991) and stored in container for use as required.

Procedure

a. Elution

A 10g weight of the regenerated resin was packed as slurry in a sintered chromatographic column (1 x 25cm in dimension). This was achieved by preparing the slurry in 30 cm^3 distilled water in a 100 cm^3 beaker (Ibrahim, 2004).

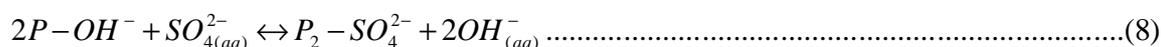
The column was eluted with a saturated solution of Na_2SO_4 at a flow rate of 2 cm^3/min .

b. Titration

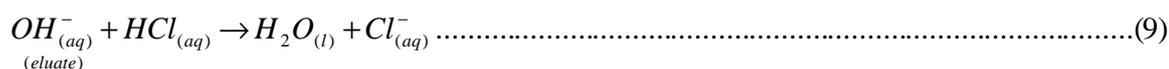
Combined eluate fractions collected after every 3min were titrated using micro burettes against 0.1M HCl to phenolphthalein end point. Both elution and titration steps were repeated several times and average of the results are recorded as in the Table.

Results and Discussion

The expected exchange during the elution process between the stationary phase, Amberlite IR – 45(OH), and the mobile aqueous Na₂SO₄ phase (from equation (2)), can be expressed as:



Thus, the quantitative amount of SO₄²⁻ ions adsorbed by the resin can be estimated by titrating the eluate (2OH⁻) with an acid.



From the mole ratio of equation (9) above, 0.15cm³ of 0.1M HCl consumed during the titration is equivalent to 1.5 x 10⁻⁵ moles of OH⁻ ions exchanged. Therefore, a total of 4.1cm³ of 0.1M HCl consumed represents an equivalence of 41.0 x 10⁻⁵ moles of OH⁻ ions exchanged, which by extension from equation (8) is equivalent to 20.5 x 10⁻⁵ moles or 19.68mg of SO_{4(aq)}²⁻ adsorbed by the resin. This value compares well with the range of values reported by Tjaart *et al.* (1983) for separating gallium of 1.5mg to 20mg using 3.0g to 10g of AG50W-X4 resin.

The void volume of the exchanger (equation (6)) was deduced from the fact that t_m in the equation corresponds to the total time taken before OH⁻ is detected in the eluate which is 9min. Thus, with a flow rate of 2cm³/min, V_m equals 18cm³. The retention time from the table is equivalent to 33min, whereas an **average** retention volume (V_R) from equation (5) is equivalent to 56.5cm³. The use of the word **average** in referring to the retention volume V_R of the resin became necessary as after elution the flow rate decreased due to swelling of the resin on prolonged usage.

The exchange capacity and exchange site density were computed from equations (3) and (4) to be 41mmole/g and 2.4682 x 10²² sites/g, respectively.

Conclusion

Comparing the values of the exchange capacity and exchange site density obtained in this study with those reported by Ibrahim and Ekanem, 2001 depicts a difference in the order of about 10³ sites/g of the synthetic Amberlite exchanger in relation to those of modified (activated) charcoal and maize cob peat. This shows that large amount of the exchangeable ions in solution can easily be removed by the resin.

Table: Average volume of titrant consumed and moles of OH⁻ ions exchanged by the resin.

Time(min)	Volume of titrant(ml)	Moles of OH ⁻ (x 10 ⁻⁵)
3	0.00	0.00
6	0.00	0.00
9	0.00	0.00
12	0.15	1.50
15	0.50	5.00
18	0.85	8.50
21	1.15	11.50
24	0.70	7.00
27	0.35	3.50
30	0.20	2.00
33	0.20	2.00
Total	4.10ml	41.0 x 10⁻⁵ moles

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