

Characterization Study of Anion Exchange Resins Tulsion A-33 and Indion H-IP by Application of ^{131}I and ^{82}Br as a Tracer Isotopes Using γ -Ray Spectrometer

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Abstract The present paper deals with the application of ^{131}I and ^{82}Br radioactive tracer isotopes in characterization of strong base, nuclear grade anion exchange resin Tulsion A-33 and intermediate base, non-nuclear grade anion exchange resin Indion H-IP. The characterization study was performed by using γ -ray spectrometer having Na(I) Tl scintillation detector. The two radioactive tracer isotopes were used to trace iodide and bromide ion-isotopic exchange reactions taking place between the ion exchange resins and external ionic solution of different concentrations varying from 0.001M to 0.004 M, in the temperature range of 30.0 $^{\circ}\text{C}$ to 45.0 $^{\circ}\text{C}$. The characterization of the two resins was made by comparing the values of specific reaction rate (min^{-1}), percentage of ions exchanged, and distribution coefficient (K_d) values. It was observed that under identical experimental conditions, above values were calculated higher for Tulsion A-33 as compared to Indion H-IP resins, indicating superior performance of Tulsion A-33. It is expected that the radioactive tracer technique as applied in the present investigation will find important role not only in characterization of various ion exchange materials, but also in optimization of various industrial process parameters so as to bring about the efficient performance of selected resin.

Keywords Tulsion A-33, Indion H-IP, Tracer Isotopes, ^{131}I , ^{82}Br , Radioactive Isotopes, Reaction Kinetics, Ion-Isotopic Exchange Reactions, Reaction Rate, Instrumental Technique, γ - Ray Spectrometer, Na(I)Tl Scintillation Detector

1. Introduction

Radioisotopes as a tracer are used as a research tools to study and elucidate reaction mechanisms, structure determination/confirmation and isotope exchange reactions. The efficiency of any chemical operation as well as kinetics can easily be evaluated using radiotracers due to the sensitivity of measurement of radiation. In tracer chemistry the radioisotope is used to follow the behaviour of an element in a chemical reaction. The interest may be in the properties of the element itself, or of a compound, radical or a group of which it forms a part. This is by far the widest use of radioisotopes at present. Although radioisotopes have been applied widely over last many years to solve industrial related technical problems, research and development of the technology continues unabated[1-3].

The fundamental principle in radiochemical investigations is that the chemical properties of a radioisotope of an element are almost the same as those of the other stable/radioactive isotopes of the element. When radioisotope is present in a

chemical form identical to that of the bulk of the element in a chemical process, then any reaction the element undergoes can be directly traced by monitoring the radioisotope.

Radiochemical work involves two main steps first is the sampling of chemical species to be studied and second is quantitative determination of the radiation emitted by the radioisotope in the sample[4]. In radiotracer study, a short lived radioisotope in a physico-chemical form similar to that of the process material is used to trace the material under study. The radioisotopes in suitable physical and chemical forms are introduced in systems under study. By monitoring the radioactivity both continuously or after sampling (depending on the nature of study), the movement, adsorption, retention etc. of the tracer and in turn, of the bulk matter under investigation, can be followed. The tracer concentration recorded at various locations also helps to draw information about the dynamic behavior of the system under study. The radioisotopes preferred for such studies are gamma emitters having half-life compatible with the duration of studies. The strength of radioactivity used varies depending on the nature of application. Radiotracer methodology is described extensively in the literature[5-12]. Applications of radiotracers in chemical research cover the studies of reaction mechanism, kinetics, exchange processes and analytical applications such as radiometric titrations, solubility product estimation, isotope

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dilution analysis and autoradiography. Radioisotope tracers offer several advantages such as high detection sensitivity, capability of in-situ detection, limited memory effects and physico-chemical compatibility with the material under study. The radioisotopes have proved as a tool to study many problems in chemical, biological and medicinal fields. Radiotracers have helped in identification of leaks in buried pipelines and dams. Process parameters such as mixing efficiency, residence time, flow rate, material inventory and silt movement in harbours are studied using radioisotopes[4]. The efficiency of several devices in a wastewater treatment plant (primary and secondary clarifiers, aeration tank) is investigated by means of radiotracers[13].

Considering the above wide use of radioactive isotopes in various industrial and technical applications, in the present investigation, they are applied to assess the performance of industrial grade anion exchange resins Tulsion A-33 and Indion H-IP under different operational parameters like temperature and ionic concentrations. It is expected that the tracer technique used here can also be used for characterization of other organic ion exchange resins which are widely synthesized for their specific technical applications[14-16]. The present technique can also be extended further to standardize the operational parameters so as to bring about the most efficient performance of those resins in their specific industrial applications.

2. Experimental

2.1. Conditioning of Ion Exchange Resins

Ion exchange resin Tulsion A-33 is a nuclear grade strong base anion exchange resin in hydroxide form (by Thermax India Ltd., Pune), while Indion H-IP is an industrial grade isoporous, intermediate base anion exchange resin in chloride form (by Ion Exchange India Ltd., Mumbai). Details regarding the properties of the resins used are given in Table 1. These resins were converted separately in to iodide / bromide form by treatment with 10 % KI / KBr solution in a conditioning

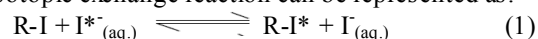
column which is adjusted at the flow rate as 1 mL / min. The resins were then washed with double distilled water, until the washings were free from iodide/bromide ions as tested by AgNO_3 solution. These resins in bromide and iodide form were then dried separately over P_2O_5 in desiccators at room temperature.

2.2. Radioactive Tracer Isotopes

The radioisotope ^{131}I and ^{82}Br used in the present experimental work was obtained from Board of Radiation and Isotope Technology (BRIT), Mumbai. Details regarding the isotopes used in the present experimental work are given in Table 2.

2.3. Study on Kinetics of Iodide Ion-isotopic Exchange Reaction

In a stoppered bottle 250 mL (V) of 0.001 M iodide ion solution was labeled with diluted ^{131}I radioactive solution using a micro syringe, such that 1.0 mL of labeled solution has a radioactivity of around 15,000 cpm (counts per minute) when measured with γ -ray spectrometer having NaI (TI) scintillation detector. Since only about 50–100 μL of the radioactive iodide ion solution was required for labeling the solution, its concentration will remain unchanged, which was further confirmed by potentiometer titration against AgNO_3 solution. The above labeled solution of known initial activity (A_i) was kept in a thermostat adjusted to 30.0°C . The swelled and conditioned dry ion exchange resins in iodide form weighing exactly 1.000 g (m) were transferred quickly into this labeled solution which was vigorously stirred by using mechanical stirrer and the activity in cpm of 1.0 mL of solution was measured. The solution was transferred back to the same bottle containing labeled solution after measuring activity. The iodide ion-isotopic exchange reaction can be represented as:



Here R-I represents ion exchange resin in iodide form; $\text{I}^{*}_{(\text{aq})}$ represents aqueous iodide ion solution labeled with ^{131}I radiotracer isotope.

Table 1. Properties of ion exchange resins

Ion exchange resin	Matrix	Functional Group	Particle Size (mm)	Moisture content (%)	Operating pH	Maximum operating Temperature ($^\circ\text{C}$)	Total exchange capacity (meq./mL)
Tulsion A-33	Polystyrene Copolymer	-N+R3	0.3-1.2	60	0-14	60	1.0
Indion H-IP	Polystyrene Copolymer	-N+R2 -N+R3	0.3-1.2	51	0-7	80	1.1

Table 2. Properties of ^{131}I and ^{82}Br tracer isotopes[17]

Isotopes	Half-life	Radioactivity / mCi	γ - energy / MeV	Chemical form	Physical form
^{131}I	8.04 d	5	0.36	Iodide*	Aqueous
^{82}Br	36 h	5	0.55	Bromide**	Aqueous

* Sodium iodide in dilute sodium sulphite

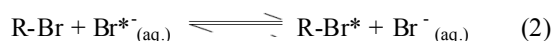
** Ammonium bromide in dilute ammonium hydroxide

The activity of solution was measured at a fixed interval of every 2.0 min. The final activity (Af) of the solution was also measured after 3h which was sufficient time to attain the equilibrium[18-32]. The activity measured at various time intervals was corrected for background counts.

Similar experiments were carried out by equilibrating separately 1.000 g of ion exchange resin in iodide form with labeled iodide ion solution of four different concentrations ranging up to 0.004 M at a constant temperature of 30.0°C. The same experimental sets were repeated for higher temperatures up to 45.0°C.

2.4 Study on kinetics of Bromide Ion-isotopic Exchange Reaction

The experiment was also performed to study the kinetics of bromide ion- isotopic exchange reaction by equilibrating 1.000 g of ion exchange resin in bromide form with labeled bromide ion solution in the same concentration and temperature range as above. The labeling of bromide ion solution was done by using ^{82}Br as a radioactive tracer isotope for which the same procedure as explained above was followed. The bromide ion-isotopic exchange reaction can be represented as:



Here R-Br represents ion exchange resin in bromide form; $\text{Br}^{*}_{(\text{aq.})}$ represents aqueous bromide ion solution labeled with ^{82}Br radiotracer isotope.

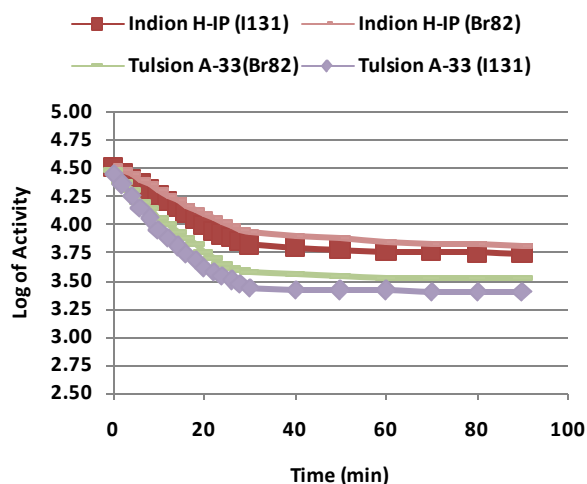


Figure 1. Kinetics of Ion-Isotopic Exchange Reactions Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Temperature = 35.0°C

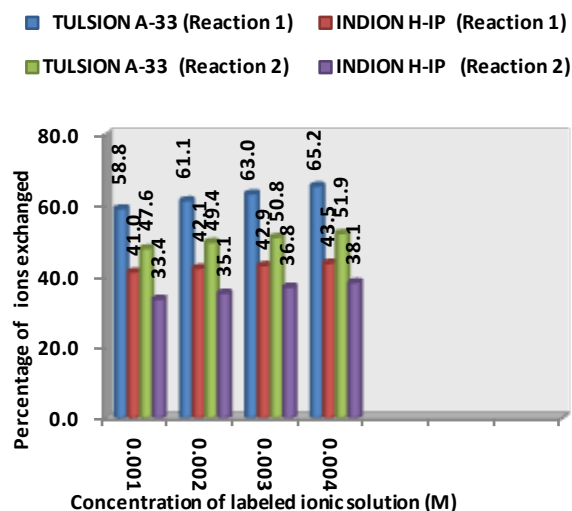


Figure 2. Variation in Percentage Ions Exchanged with Concentration of Labeled Ionic Solution Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0°C

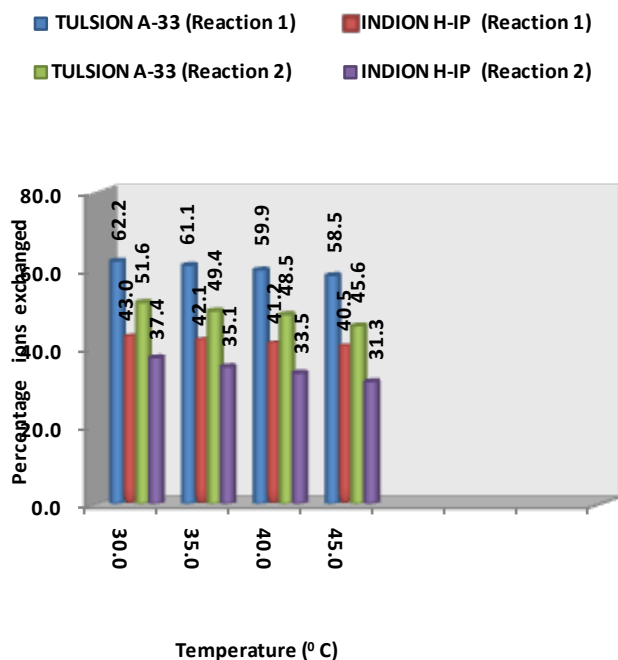


Figure 3. Variation in Percentage Ions Exchanged with Temperature of Labeled Ionic Solution. Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002 M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol

Table 3. Concentration effect on Ion-Isotopic Exchange Reactions

Concentration of ionic solution (M)	Amount of ions in 200 mL solution (mmol)	REACTION -1								REACTION -2							
		TULSION A-33				INDION H-IP				TULSION A-33				INDION H-IP			
		Specific reaction rate of rapid process min^{-1}	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K_d	Specific reaction rate of rapid process min^{-1}	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K_d	Specific reaction rate of rapid process min^{-1}	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K_d	Specific reaction rate of rapid process min^{-1}	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K_d
0.001	0.250	0.215	0.147	0.032	9.0	0.083	0.103	0.009	4.9	0.176	0.119	0.021	7.8	0.080	0.084	0.007	2.1
0.002	0.500	0.226	0.306	0.069	10.0	0.092	0.211	0.019	5.2	0.179	0.247	0.044	8.7	0.088	0.176	0.015	2.5
0.003	0.750	0.235	0.473	0.111	11.1	0.105	0.322	0.034	5.6	0.183	0.381	0.070	9.1	0.097	0.276	0.027	2.9
0.004	1.000	0.251	0.652	0.164	11.8	0.114	0.435	0.050	6.0	0.190	0.519	0.099	9.9	0.106	0.381	0.040	3.3

Amount of ion exchange resin = 1.000 g Volume of labeled ionic solution = 250 mL, Temperature = 35.0°C

Table 4. Temperature effect on Ion-Isotopic Exchange Reactions

Temperature $^\circ\text{C}$	REACTION -1								REACTION -2							
	TULSION A-33				INDION H-IP				TULSION A-33				INDION H-IP			
	Specific reaction rate of rapid process min^{-1}	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K_d	Specific reaction rate of rapid process min^{-1}	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K_d	Specific reaction rate of rapid process min^{-1}	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K_d	Specific reaction rate of rapid process min^{-1}	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K_d
30.0	0.238	0.311	0.074	10.7	0.104	0.215	0.022	6.0	0.183	0.258	0.047	9.3	0.100	0.187	0.019	3.6
35.0	0.226	0.306	0.069	10.0	0.092	0.211	0.019	5.2	0.179	0.247	0.044	8.7	0.088	0.176	0.015	2.5
40.0	0.217	0.300	0.065	9.5	0.081	0.206	0.017	4.5	0.174	0.243	0.042	8.0	0.080	0.168	0.013	2.1
45.0	0.210	0.293	0.061	9.1	0.071	0.203	0.014	4.0	0.167	0.228	0.038	7.4	0.072	0.157	0.011	1.7

Amount of ion exchange resin = 1.000 g Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol

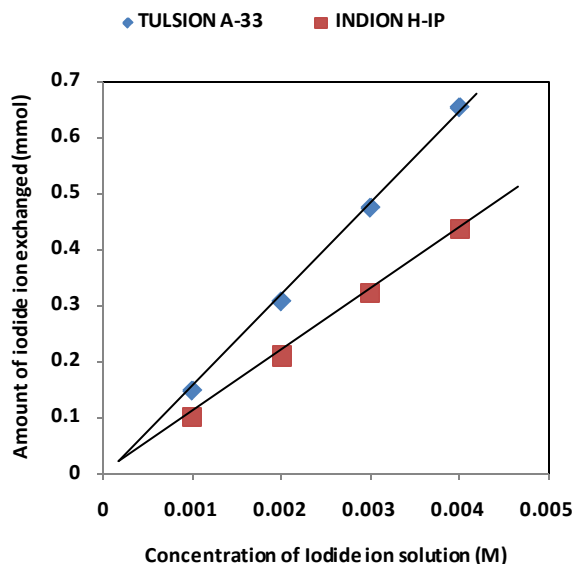


Figure 4. Correlation between concentrations of iodide ion solution and amount of iodide ion exchanged Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0°C Correlation coefficient (r) for Indion H-IP = 0.9999 Correlation coefficient (r) for Tulsion A-33 = 0.9996

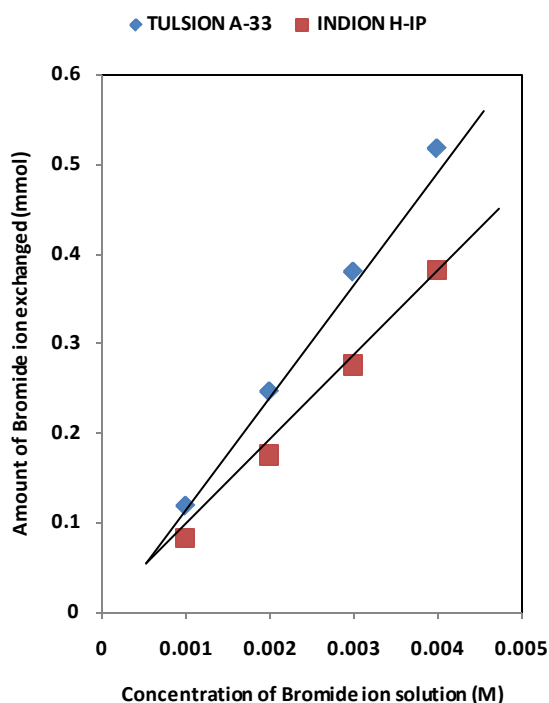


Figure 5. Correlation between concentrations of bromide ion solution and amount of bromide ion exchanged Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0°C Correlation coefficient (r) for Indion H-IP = 0.9996 Correlation coefficient (r) for Tulsion A-33 = 0.9999

3.1. Comparative Study of Ion-isotopic Exchange Reactions

In the present investigation it was observed that due to the rapid ion-isotopic exchange reaction taking place, the activity of solution decreases rapidly initially, then due to the slow exchange the activity of the solution decreases slowly and finally remains nearly constant. Preliminary studies show that the above exchange reactions are of first order[33, 34]. Therefore logarithm of activity when plotted against time gives a composite curve in which the activity initially decreases sharply and thereafter very slowly giving nearly straight line (Figure 1), evidently rapid and slow ion-isotopic exchange reactions were occurring simultaneously[18-32]. Now the straight line was extrapolated back to zero time. The extrapolated portion represents the contribution of slow process to the total activity which now includes rapid process also. The activity due to slow process was subtracted from the total activity at various time intervals. The difference gives the activity due to rapid process only. From the activity exchanged due to rapid process at various time intervals, the specific reaction rates (k) of rapid ion-isotopic exchange reaction were calculated. The amount of iodide / bromide ions exchanged (mmol) on the resin were obtained from the initial and final activity of solution and the amount of exchangeable ions in 250 mL of solution. From the amount of ions exchanged on the resin (mmol) and the specific reaction rates (min⁻¹), the initial rate of ion exchanged (mmol/min) was calculated.

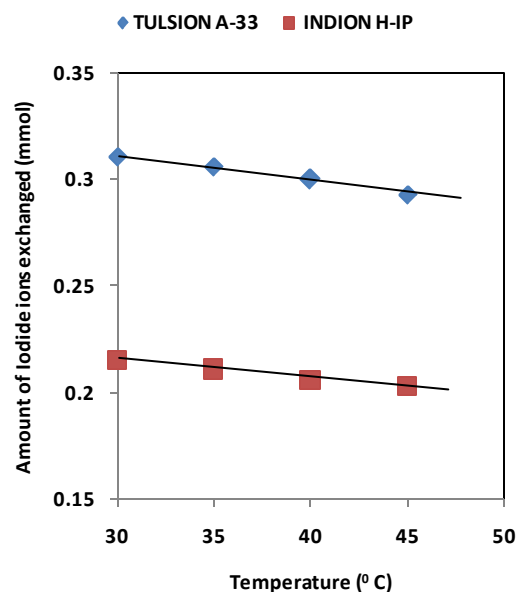


Figure 6. Correlation between Temperatures of exchanging medium and amount of iodide ion exchanged Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol Correlation coefficient (r) for Indion H-IP = -0.9959 Correlation coefficient (r) for Tulsion A-33 = -0.9972

3. Results and Discussion

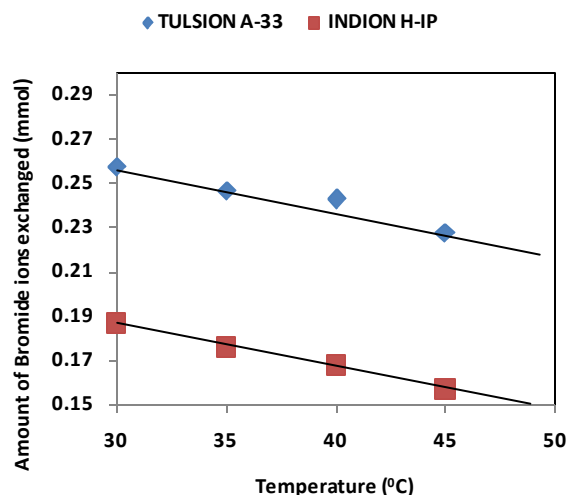


Figure 7. Correlation between Temperatures of exchanging medium and amount of bromide ion exchanged Amount of ion exchange resin = 1.000 g Concentration of labeled exchangeable ionic solution = 0.002 M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol Correlation coefficient (r) for Indion H-IP = -0.9981 Correlation coefficient (r) for Tulsion A-33 = -0.9779

Because of larger solvated size of bromide ions as compared to that of iodide ions, it was observed that the exchange of bromide ions occurs at the slower rate than that of iodide ions [35]. Hence under identical experimental conditions, the values of specific reaction rate (min^{-1}), amount of ion exchanged (mmol) and initial rate of ion exchange (mmol/min) are calculated to be lower for bromide ion-isotopic exchange reaction than that for iodide ion-isotopic exchange reaction as summarized in Tables 3 and 4. For both bromide and iodide ion-isotopic exchange reactions, under identical experimental conditions, the values of specific reaction rate increases with increase in concentration of ionic solution from 0.001M to 0.004M (Table 3). However, with rise in temperature from 30.0°C to 45.0°C, the specific reaction rate was observed to decrease (Table 4). Thus in case of Tulsion A-33 at 35.0°C when the ionic concentration increases from 0.001M to 0.004M, the specific reaction rate values for iodide ion-isotopic exchange increases from 0.215 to 0.251 min^{-1} , while for bromide ion-isotopic exchange the values increases from 0.176 to 0.190 min^{-1} . Similarly in case of Indion H-IP, under identical experimental conditions, the values for iodide ion-isotopic exchange increases from 0.083 to 0.114 min^{-1} , while for bromide ion-isotopic exchange the values increases from 0.080 to 0.106 min^{-1} . However when concentration of ionic solution is kept constant at 0.002 M and temperature is raised from 30.0°C to 45.0°C, in case of Tulsion A-33 the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.238 to 0.210 min^{-1} , while for bromide ion-isotopic exchange the values decreases from 0.183 to 0.167 min^{-1} . Similarly in case of Indion H-IP, under identical experimental conditions, the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.104 to 0.071 min^{-1} , while for bromide ion-isotopic

exchange the values decreases from 0.100 to 0.072 min^{-1} . From the results, it appears that iodide ions exchange at the faster rate as compared to that of bromide ions which was related to the extent of solvation (Tables 3 and 4).

From the knowledge of A_i , A_f , volume of the exchangeable ionic solution (V) and mass of ion exchange resin (m), the K_d value was calculated by the equation

$$K_d = [(A_i - A_f) / A_f] \times V / m \quad (3)$$

Heumann et al.[36] in the study of chloride distribution coefficient on strongly basic anion exchange resin observed that the selectivity coefficient between halide ions increased at higher electrolyte concentrations. Adachi et al.[37] observed that the swelling pressure of the resin decreased at higher solute concentrations resulting in larger K_d values. The temperature dependence of K_d values on cation exchange resin was studied by Shuji et al.[38]; were they observed that the values of K_d increased with fall in temperature. The present experimental results also indicates that the K_d values for bromide and iodide ions increases with increase in ionic concentration of the external solution, however with rise in temperature the K_d values were found to decrease. Thus in case of Tulsion A-33 at 35.0°C when the ionic concentration increases from 0.001M to 0.004M, the log K_d values for iodide ions increases from 9.0 to 11.8, while for bromide ions the values increases from 7.8 to 9.9. Similarly in case of Indion H-IP, under identical experimental conditions, the log K_d values for iodide ions increases from 4.9 to 6.0, while for bromide ions the values increases from 2.1 to 3.3. However when concentration of ionic solution is kept constant at 0.002 M and temperature is raised from 30.0°C to 45.0°C, in case of Tulsion A-33 the log K_d values for iodide ions decreases from 10.7 to 9.1, while for bromide ions the values decreases from 9.3 to 7.4. Similarly in case of Indion H-IP, under identical experimental conditions, the log K_d values for iodide ions decreases from 6.0 to 4.0, while for bromide ions the values decreases from 3.6 to 1.7. It was also observed that the K_d values for iodide ion-isotopic exchange reaction were calculated to be higher than that for bromide ion-isotopic exchange reaction (Tables 3 and 4).

3.2. Comparative Study of Anion Exchange Resins

From the Table 3 and 4, it is observed that for iodide ion-isotopic exchange reaction by using Tulsion A-33 resin, the values of specific reaction rate (min^{-1}), amount of iodide ion exchanged (mmol), initial rate of iodide ion exchange (mmol/min) and log K_d were 0.226, 0.306, 0.069 and 10.0 respectively, which was higher than 0.092, 0.211, 0.019 and 5.2 respectively as that obtained by using Indion H-IP resins under identical experimental conditions of 35.0°C, 1.000 g of ion exchange resins and 0.002 M labeled iodide ion solution. The identical trend was observed for the two resins during bromide ion-isotopic exchange reaction.

From Table 3, it is observed that using Tulsion A-33 resins, at a constant temperature of 35.0°C, as the concentration of labeled iodide ion solution increases 0.001 M to 0.004 M, the

percentage of iodide ions exchanged increases from 58.8 % to 65.2 %. While using Indion H-IP resins under identical experimental conditions the percentage of iodide ions exchanged increases from 41.0 % to 43.5 %. Similarly in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged increases from 47.6 % to 51.9 % using Tulsion A-33 resin, while for Indion H-IP resin it increases from 33.4 % to 38.1 %. The effect of ionic concentration on percentage of ions exchanged is graphically represented in Figure 2.

From Table 4, it is observed that using Tulsion A-33 resins, for 0.002 M labeled iodide ion solution, as the temperature increases 30.0 °C to 45.0 °C, the percentage of iodide ions exchanged decreases from 62.2 % to 58.5 %. While using Indion H-IP resins under identical experimental conditions the percentage of iodide ions exchanged decreases from 43.0 % to 40.5 %. Similarly in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged decreases from 51.6 % to 45.6 % using Tulsion A-33 resin, while for Indion H-IP resin it decreases from 37.4 % to 31.3 %. The effect of temperature on percentage of ions exchanged is graphically represented in Figure 3.

The overall results indicate that under identical experimental conditions, as compared to Indion H-IP resins, Tulsion A-33 resins shows higher percentage of ions exchanged. Thus Tulsion A-33 resins show superior performance than Indion H-IP resins under identical operational parameters.

3.3. Statistical Correlations

The results of present investigation show a strong positive linear co-relationship between amount of ions exchanged and concentration of ionic solution (Figures 4, 5). In case of iodide ion-isotopic exchange reaction, the values of correlation coefficient (r) were calculated as 0.9996 and 0.9999 for Tulsion A-33 and Indion H-IP resins respectively, while for bromide ion-isotopic exchange reaction, the respective values of r was calculated as 0.9999 and 0.9996.

There also exist a strong negative co-relationship between amount of ions exchanged and temperature of exchanging medium (Figures 6, 7). In case of iodide ion-isotopic exchange reactions the values of r calculated for Tulsion A-33 and Indion H-IP resins were -0.9972 and -0.9959 respectively. Similarly in case of bromide ion-isotopic exchange reactions the r values calculated were -0.9779 and -0.9981 respectively for both the resins.

4. Conclusions

The present experimental work is an excellent application of radiotracers using γ - Ray spectrometer having Na(I)TI scintillation detector. The instrumental technique used in the investigation will be further applied to standardize the operational process parameters so as to improve the performance of selected ion exchange resins. The present instrumental technique using radioactive tracer isotopes can also be applied further for characterization of different

nuclear as well as non-nuclear grade ion exchange resins.

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