

Comparative Study of Strongly Basic Anion Exchange Resins Tulsion A-30 and Indion-820 by Application of ^{131}I and ^{82}Br as a Tracer Isotopes

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Abstract In the present investigation radioactive tracer isotopes ^{131}I and ^{82}Br were used to study the kinetics of iodide and bromide ion–isotopic exchange reaction. The kinetics study was performed to study the effect of ionic concentration and temperature on the specific reaction rate calculated in min^{-1} . From the initial ionic concentration and knowing the value of specific reaction rate, the values of amount of ions exchanged in mmol and percentage ion exchange were calculated. It was also observed that for both the resins, there exists strong positive co-relationship between amount of ions exchanged and concentration of ionic solution, while a strong negative co-relationship was observed between amount of ions exchanged and temperature of exchanging medium. The values of specific reaction rate, amount and percentage of ions exchanged so obtained for the ion-isotopic exchange reactions using strong base, nuclear grade anion exchange resin Tulsion A-30 and non-nuclear grade anion exchange resin Indion-820 were compared. It was observed that the above values calculated were higher for Tulsion A-30 as compared to that for Indion-820, indicating superior performance of Tulsion A-30 under identical experimental conditions. The similar study on application of radioactive tracer isotopes can be extended further for performance assessment of different nuclear as well as non-nuclear grade ion exchange resins.

Keywords Tulsion A-30, Indion-820, Nuclear Grade Ion Exchanged Resin, Anion Exchange Resins, ^{131}I , ^{82}Br , Tracer Isotopes, Reaction Kinetics, Ion-Isotopic Exchange Reactions, Reaction Rate, Distribution Coefficient

1. Introduction

Although radioisotopes have been applied to the solution of problems in industry for over 50 years, research and development of the technology continues unabated [1, 2]. The economic benefits that may be derived from the use of this technology are great, a fact that is recognized by the governments of developing countries. There are two main reasons for the continuing interest. Firstly, it is industry driven. Because of their unique properties, radioactive isotopes can be used to obtain information about plants and processes that cannot be obtained in any other way. Often, the information is obtained with the plant on-stream and without disrupting the process in any way. This can lead to substantial economic benefits, from shutdown avoidance to process optimization. Secondly, the methodology is derived from many fields of science and technology including radioisotope production, radiation detection, data acquisition, treatment and analysis, and mathematical modelling [3-9]. Important characteristics of radioisotope for use as tracer are

half life, type and energy of radiation and availability. Short lived isotopes (a few hours to a few days of half life) are preferred as they have high specific activity and the system becomes non-radioactive very soon. If larger amounts of radioactivity are needed for use, then it is preferred to have short lived isotopes. Also it is ideal to have a gamma emitting isotope as gamma ray measurement is the simplest and also external monitoring of the process is practicable.

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 [10]. 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,

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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 behaviour 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. 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 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[10]. The efficiency of several devices in a wastewater treatment plant (primary and secondary clarifiers, aeration tank) is investigated by means of radiotracers[11].

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-30 and Indion-820 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 [12-14]. 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-30 is a nuclear grade strong base anion exchange resin in hydroxide form (by Thermax India Ltd., Pune), while Indion-820 is a macroporous strong 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.

Table 1. Properties of ion exchange resins

Ion exchange resin	Matrix	Functional Group	Particle Size (mm)	Moisture content (%)	Operating pH	Maximum operating Temperature (°C)	Total exchange Capacity (meq./mL)
Tulsion A-30	Polystyrene Copolymer	Quaternary Ammonium Type-I	0.3-1.2	60	0-14	60	1.0
Indion-820	Polystyrene Copolymer	Quaternary Ammonium Type-II	0.3-1.2	50	0-14	40	1.1

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.

Table 2. Properties of ^{131}I and ^{82}Br tracer isotopes[15]

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.

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; $I^*_{(aq.)}$ represents aqueous iodide ion solution labeled with ^{131}I radiotracer isotope.

The activity of solution was measured at a fixed interval of every 2.0 min. The final activity (A_f) of the solution was also measured after 3h which was sufficient time to attain the equilibrium [16-30]. 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; $Br^*_{(aq.)}$ represents aqueous bromide ion solution labeled with ^{82}Br radiotracer isotope.

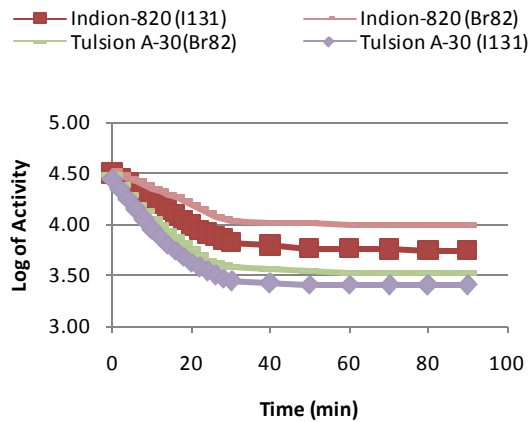


Figure 1. Kinetics of Ion-Isotopic Exchange Reactions Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C

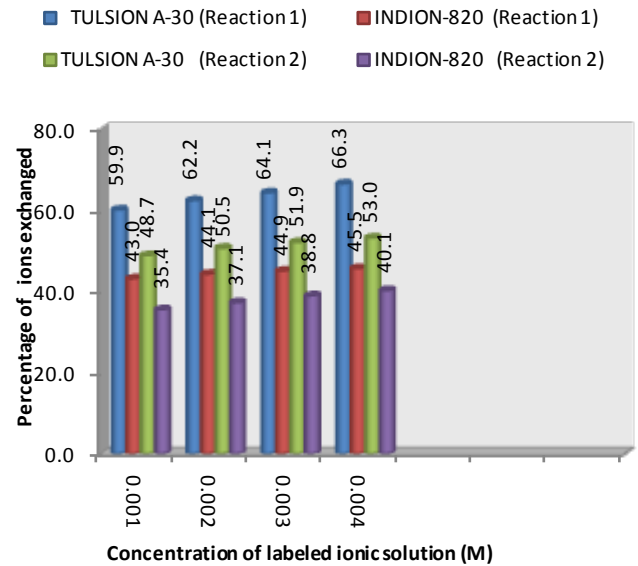


Figure 2. Variation in Percentage Ions Exchanged with Concentration of Labeled Ionic Solution Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C

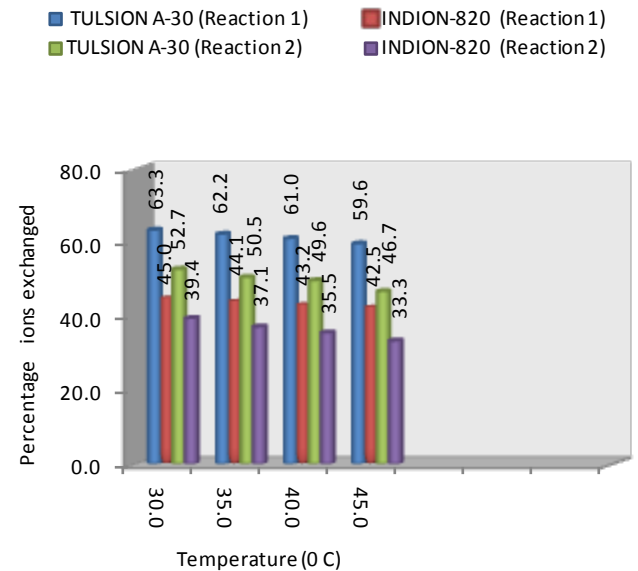


Figure 3. Variation in Percentage Ions Exchanged with Temperature of Labeled Ionic Solution Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002 M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500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

Table 3. Concentration effect on Ion-Isotopic Exchange Reactions Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C

Concentration of ionic solution (M)	REACTION -1								REACTION -2							
	TULSION A-30				INDION-820				TULSION A-30				INDION-820			
	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 exchanged (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	0.222	0.150	0.033	9.5	0.100	0.108	0.011	5.9	0.184	0.122	0.022	8.0	0.088	0.089	0.008	2.4
0.002	0.234	0.311	0.073	10.6	0.112	0.221	0.025	6.2	0.190	0.253	0.048	9.1	0.095	0.186	0.018	3.0
0.003	0.249	0.481	0.120	11.7	0.121	0.337	0.041	6.9	0.208	0.389	0.081	9.6	0.103	0.291	0.030	3.4
0.004	0.265	0.663	0.176	12.3	0.132	0.455	0.060	7.3	0.220	0.530	0.117	10.3	0.115	0.401	0.046	3.9

Table 4. Temperature effect on 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, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol

Temperature °C	REACTION -1				REACTION -2											
	TULSION A-30		INDION-820		TULSION A-30		INDION-820									
	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				
30.0	0.246	0.317	0.078	11.1	0.120	0.225	0.027	7.0	0.206	0.264	0.054	9.8	0.107	0.197	0.021	4.0
35.0	0.234	0.311	0.073	10.6	0.112	0.221	0.025	6.2	0.190	0.253	0.048	9.1	0.095	0.186	0.018	3.0
40.0	0.225	0.305	0.069	10.1	0.099	0.216	0.021	5.6	0.183	0.248	0.045	8.6	0.087	0.178	0.015	2.5
45.0	0.218	0.298	0.065	9.6	0.090	0.213	0.019	5.2	0.178	0.234	0.042	7.8	0.080	0.167	0.013	2.0

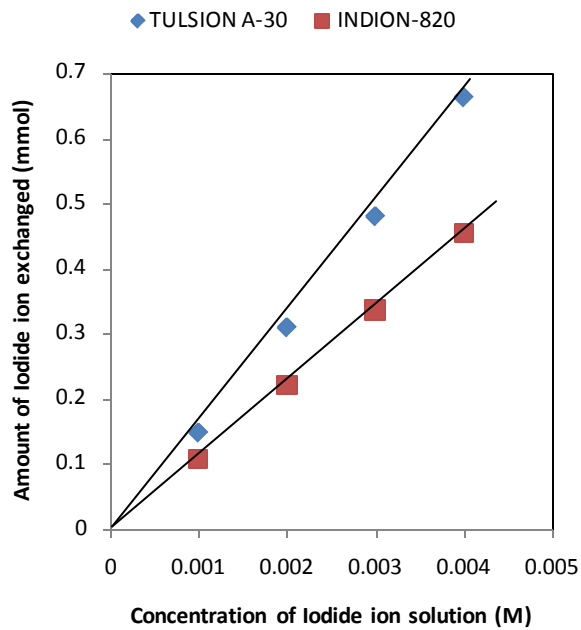


Figure 4. Correlation between concentrations of iodide ion solution and amount of iodide ion exchanged Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C Correlation coefficient (r) for Indion-820 = 1.0000 Correlation coefficient (r) for Tulsion A-30 = 0.9996

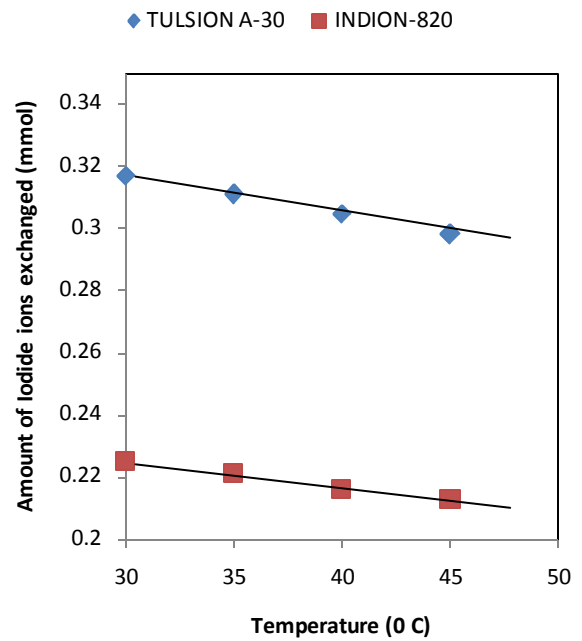


Figure 6. Correlation between Temperatures of exchanging medium and amount of iodide ion exchanged Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol Correlation coefficient (r) for Indion-820 = -0.9959 Correlation coefficient (r) for Tulsion A-30 = -0.9992

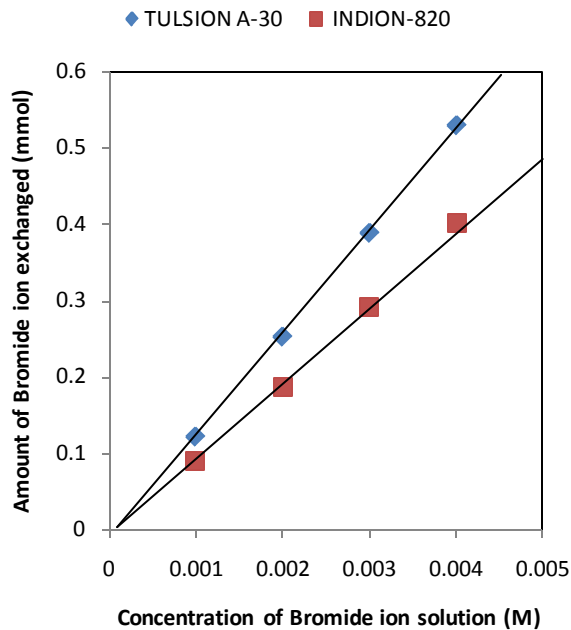


Figure 5. Correlation between concentrations of bromide ion solution and amount of bromide ion exchanged Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C Correlation coefficient (r) for Indion-820 = 0.9996 Correlation coefficient (r) for Tulsion A-30 = 0.9999

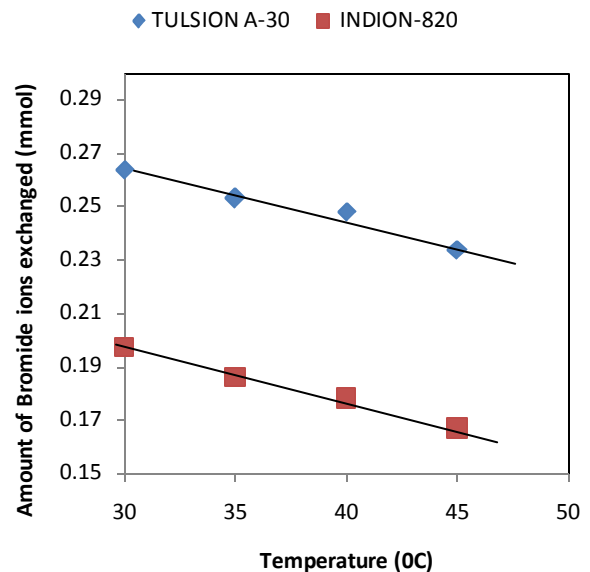


Figure 7. Correlation between Temperatures of exchanging medium and amount of bromide ion exchanged Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002 M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol Correlation coefficient (r) for Indion-820 = -0.9981

3. Results and Discussion

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 [31, 32]. 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 [16-30]. 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.

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 [33]. 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-30 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 0.222 to 0.265 min^{-1} , while for bromide ion-isotopic exchange the values increases from 0.184 to 0.220 min^{-1} . Similarly in case of Indion-820, under identical experimental conditions, the values for iodide ion-isotopic exchange increases from 0.100 to 0.132 min^{-1} , while for bromide ion-isotopic exchange the values increases from 0.088 to 0.115 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-30 the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.246 to 0.218 min^{-1} , while for bromide ion-isotopic exchange the values decreases from 0.206 to 0.178 min^{-1} . Similarly in case of Indion-820, under identical experimental conditions, the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.120 to 0.090 min^{-1} , while for bromide ion-isotopic exchange the values decreases from 0.107 to 0.080 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.* [34] 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.* [35] 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.* [36]; 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-30 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 9.5 to 12.3, while for bromide ions the values increases from 8.0 to 10.3. Similarly in case of Indion-820, under identical experimental conditions, the log K_d values for iodide ions increases from 5.9 to 7.3, while for bromide ions the values increases from 2.4 to 3.9. 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-30 the log K_d values for iodide ions decreases from 11.1 to 9.6, while for bromide ions the values decreases from 9.8 to 7.8. Similarly in case of Indion-820, under identical experimental conditions, the log K_d values for iodide ions decreases from 7.0 to 5.2, while for bromide ions the values decreases from 4.0 to 2.0. It was also observed that the K_d values for iodide ion-isotopic reaction were calculated to be higher than that for bromide ion-isotopic 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-30 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 0.234, 0.311, 0.073 and 10.6 respectively, which was higher than 0.112, 0.221, 0.025 and 6.2 respectively as that obtained by using Indion-820 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-30 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 59.9 % to 66.3 %. While using Indion-820 resins under identical experimental conditions the percentage of iodide ions exchanged increases from 43.0 % to 45.5 %. Similarly in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged increases from 48.7 % to 53.0 % using Tulsion A-30 resin, while for Indion-820 resin it increases from 35.4 % to 40.1 %. 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-30 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 63.3 % to 59.6 %. While using Indion-820 resins under identical experimental conditions the percentage of iodide ions exchanged decreases from 45.0 % to 42.5 %. Similarly in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged decreases from 52.7 % to 46.7 % using Tulsion A-30 resin, while for Indion-820 resin it decreases from 39.4 % to 33.3 %. 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-820 resins, Tulsion A-30 resins shows higher percentage of ions exchanged. Thus Tulsion A-30 resins show superior performance than Indion-820 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 1.0000 for Tulsion A-30 and Indion-820 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-30 and Indion-820 resins were -0.9992 and -0.9959 respectively. Similarly in case of bromide ion-isotopic exchange reactions the r values calculated were -0.9854 and -0.9981 respectively for both the resins.

4. Conclusions

The experimental work carried out in the present investigation will help to standardize the operational process parameters so as to improve the performance of selected ion exchange resins. The radioactive tracer technique used here can also be applied for characterization of different nuclear as well as non-nuclear grade ion exchange resins.

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