

Determination of kinetic and thermodynamic properties of Radio Tracer Exchange on Indion GS-300 and Indion GS-400 by Using Radio Analytical Technique

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Received 27.3.2013; Accepted 30.4.2014; Published 14.7.2014

ABSTRACT

The present paper deals with characterization of two organic based anion exchange resins Indion GS-300 and Indion GS-400. The characterization was based on the kinetic data of iodide and bromide ion-isotopic exchange reactions for the two resins. It was observed that during iodide ion-isotopic exchange reactions, under identical experimental conditions of 35.0°C, 1.000 g of ion exchange resins and 0.002 M labeled iodide ion solution, the values of specific reaction rate (min^{-1}), amount of iodide ion exchanged (mmol), initial rate of iodide ion exchange (mmol/min) and $\log K_a$ were 0.324, 0.381, 0.124 and 19.7 respectively for Indion GS-300, which was higher than 0.206, 0.274, 0.057 and 17.7 respectively for Indion GS-400 resins. Similar trend was observed for the two resins during bromide ion-isotopic exchange reactions. The results of the study indicate existence of strong positive linear co-relationship between amount of ions exchanged and concentration of ionic solution; while a strong negative co-relationship exists between amount of ions exchanged and temperature of exchanging medium. The results indicate superior performance of Indion GS-300 over Indion GS-400 resins under identical experimental conditions. It is expected here that the present radio analytical technique can be extended further to evaluate the performance of various industrial grade ion exchange resins.

Keywords: nuclear grade resins; organic ion exchange resins; characterization; Indion GS-300; Indion GS-400; tracer isotopes; radio analytical technique; reaction kinetics; ion-isotopic exchange; ^{131}I ; ^{82}Br .

INTRODUCTION

Development of new organic ion exchange materials for specific industrial and technological applications is a biggest challenge to present day researchers. The main advantages of synthetic organic ion exchange resins are their high capacity, wide applicability, wide versatility and low cost relative to some synthetic inorganic media. Organic ion exchange resins are used in a number of chemical decontamination or cleaning processes and in nuclear industries for removal of radionuclide [1-5].

Extensive work was done by previous researchers to synthesis various organic ion exchange resins for specific applications and their characterization [6, 7]. Efforts are also made to develop new organic ion exchangers for their specific applications in nuclear industries are continuing and various aspects of ion exchange technologies have been continuously studied to improve the efficiency and economy of their application in various technological applications [8, 9]. However, since the selection of the appropriate ion-exchange material depends on the needs of the system, it is expected that

the data obtained from the actual experimental trials will prove to be more helpful.

Hence there is a need to evaluate systematically the performance of such organic ion exchange resins under various operational conditions. Therefore in the present investigation, attempts are being made to test the performance of Indion GS-300 (nuclear grade) and Indion GS-400 (non-nuclear grade) ion exchange resins under different experimental conditions like temperature and concentration of ionic species present in the external exchanging medium.

2. EXPERIMENTAL

2.1 Conditioning of ion exchange resins Indion GS-300 (nuclear grade) and Indion GS-400 (non-nuclear grade) are strong base anion exchange resins in hydroxide form supplied 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 ($^{\circ}\text{C}$)	Total Exchange capacity (meq/mL)
Indion GS-300 (Type 1)	Crosslinked Polystyrene	$-\text{N}^+(\text{CH}_3)_3$	0.3-1.2	55	0-14	60	1.0
Indion GS-400 (Type 2)	Crosslinked Polystyrene	$-\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{OH}$	0.3-1.2	40	0-14	40	1.2

Table 2. Properties of ^{131}I and ^{82}Br tracer isotopes [10]

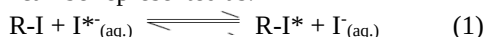
Isotopes	Half-life	Radioactivity (mCi)	γ - energy (MeV)	Chemical form	Physical form
^{131}I	8.04 d	5	0.36	Iodide*	Aqueous
^{82}Br	36 h	5	0.55	Bromide**	Aqueous

* 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; $\text{I}^*_{(\text{aq.})}$ 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)

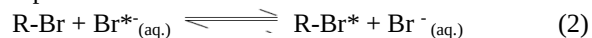
2.2 Radioactive Tracer Isotopes The radioisotope ^{131}I and ^{82}Br used in the present experimental work was obtained from Board of Radiation and Isotope Technology (BRIT), Mumbai, India. Details regarding the isotopes used in the present experimental work are given in Table 2.

of the solution was also measured after 3h which was sufficient time to attain the equilibrium [11-16]. 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.

Table 3. Concentration effect on Ion-Isotopic Exchange Reactions

Concentration of ionic solution (M)	Amount of ions in 250 mL solution (mmol)	REACTION -1								REACTION -2							
		Indion GS-300				Indion GS-400				Indion GS-300				Indion GS-400			
		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	0.318	0.188	0.060	19.0	0.196	0.131	0.026	16.9	0.249	0.160	0.040	14.1	0.150	0.119	0.018	12.0
0.002	0.500	0.324	0.381	0.124	19.7	0.206	0.274	0.057	17.7	0.257	0.327	0.084	14.7	0.159	0.246	0.039	12.4
0.003	0.750	0.335	0.585	0.196	20.4	0.218	0.439	0.096	18.3	0.266	0.503	0.134	15.4	0.170	0.382	0.065	13.5
0.004	1.000	0.347	0.800	0.278	20.9	0.231	0.611	0.141	19.1	0.277	0.688	0.191	16.0	0.180	0.515	0.093	13.9

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 °C	REACTION -1								REACTION -2							
	Indion GS-300				Indion GS-400				Indion GS-300				Indion GS-400			
	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	0.333	0.389	0.129	20.6	0.215	0.288	0.062	18.4	0.264	0.333	0.088	15.1	0.169	0.254	0.043	12.9
35.0	0.324	0.381	0.124	19.7	0.206	0.274	0.057	17.7	0.257	0.327	0.084	14.7	0.159	0.246	0.039	12.4
40.0	0.321	0.379	0.122	18.9	0.199	0.264	0.052	17.1	0.253	0.324	0.082	14.0	0.154	0.242	0.037	11.9
45.0	0.319	0.377	0.120	18.4	0.195	0.263	0.051	16.5	0.250	0.322	0.081	13.5	0.151	0.240	0.036	11.5
Energy of Activation (J.mol ⁻¹)	2224.5				5258.6				2878.1				5946.5			
Enthalpy of activation (J.mol ⁻¹)	-357.0				2677.1				296.6				3365.0			

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

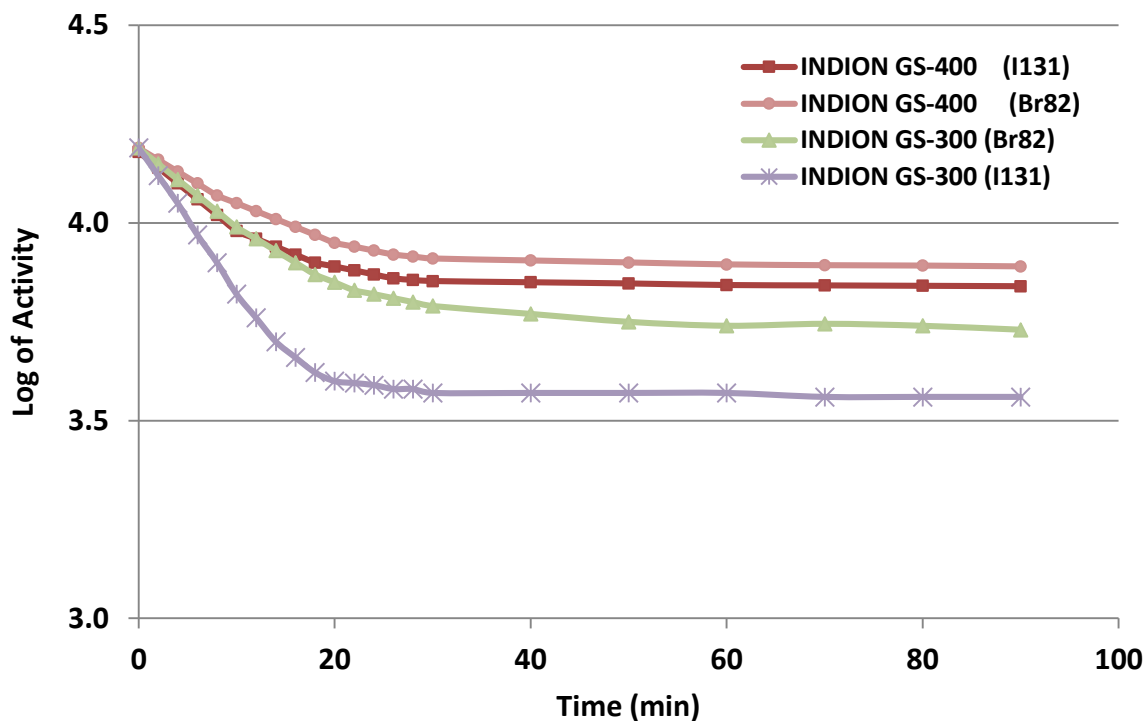


Figure 1: Kinetics of Ion-Isotopic Exchange Reactions (Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C)

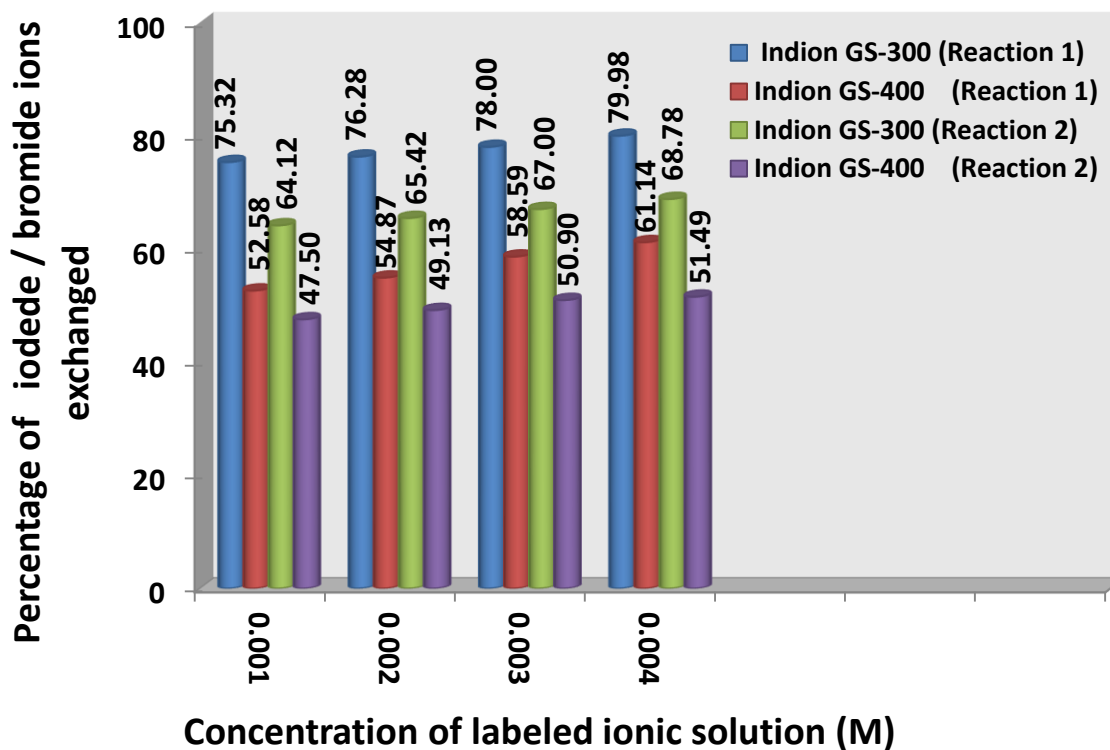


Figure 2: Variation in Percentage Ions Exchanged with Concentration of Labeled Ionic Solution (Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C)

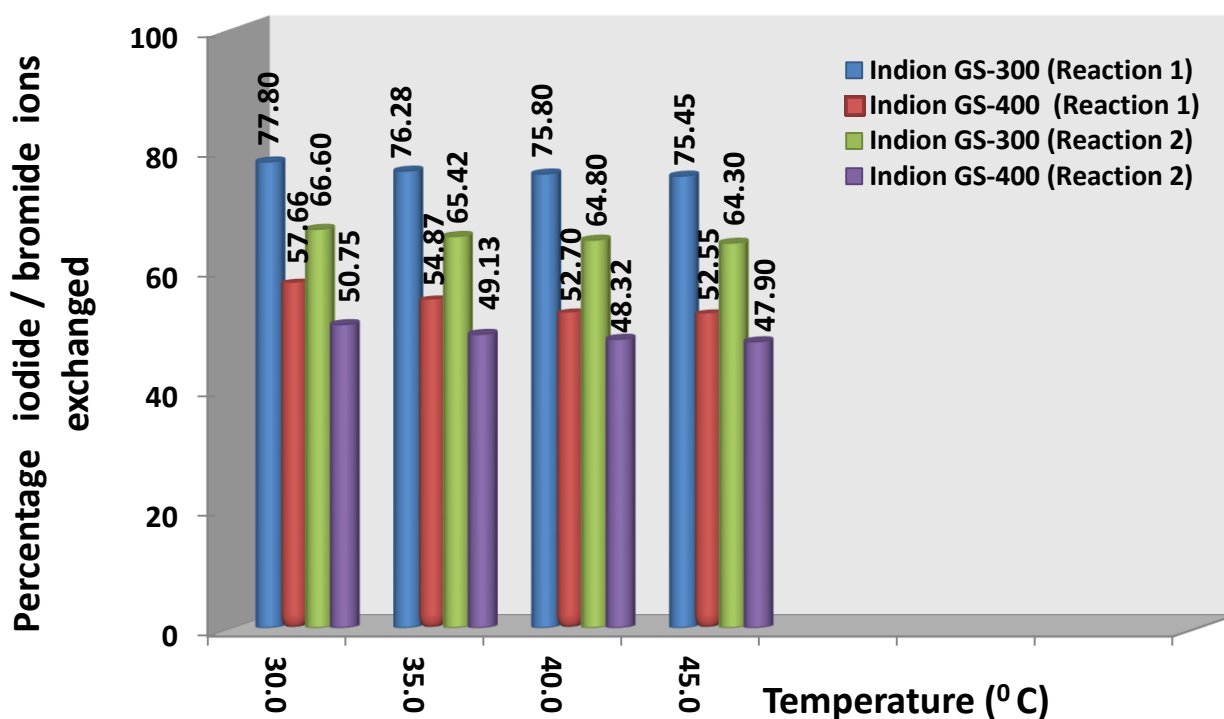


Figure 3: Variation in Percentage Ions Exchanged with Temperature of Labeled Ionic Solution (Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002 M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500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

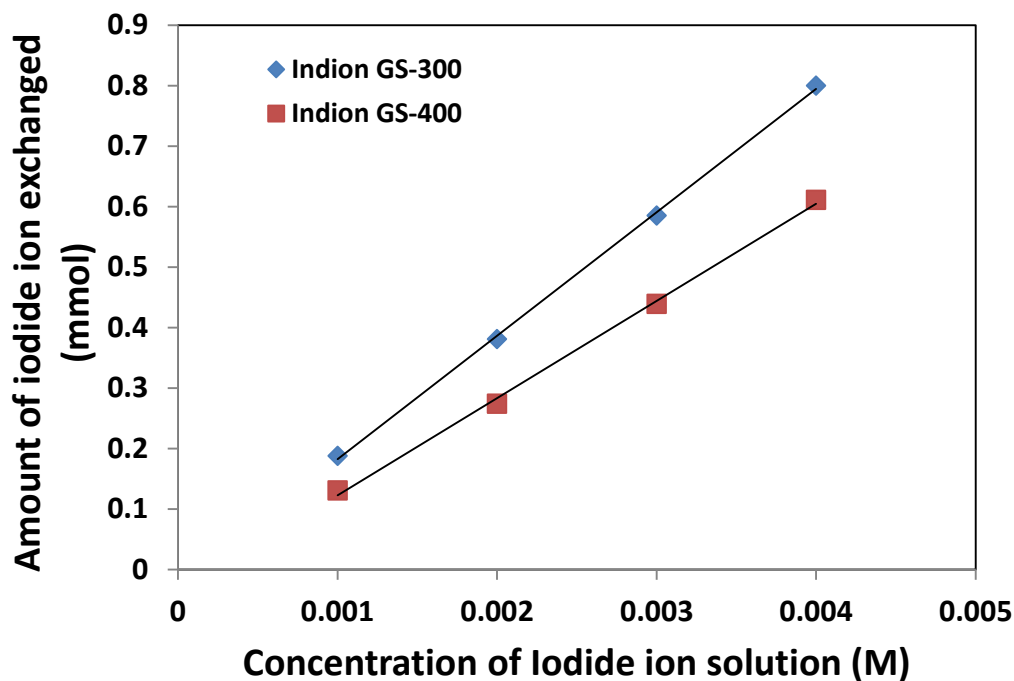


Figure 4: Correlation between concentration of iodide ion solution and amount of iodide ion exchanged (Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C, Correlation coefficient (r) for Indion GS-400 = 0.9991, Correlation coefficient (r) for Indion GS-300 = 0.9997)

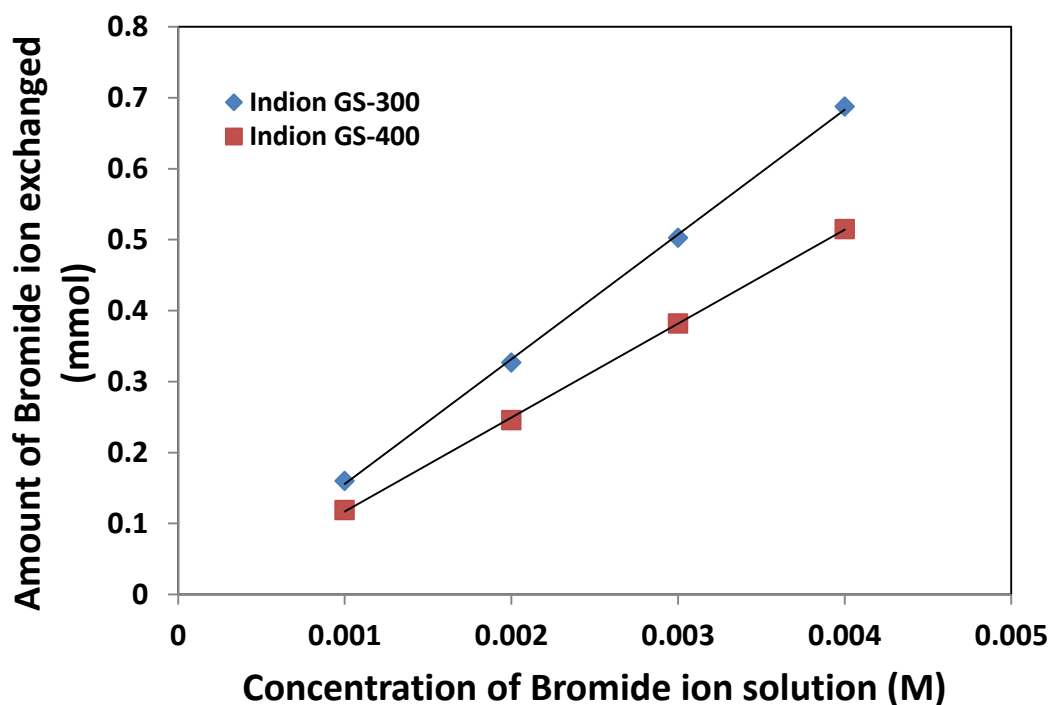


Figure 5: Correlation between concentration of bromide ion solution and amount of bromide ion exchanged (Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 35.0 °C, Correlation coefficient (r) for Indion GS-400 = 0.9999, Correlation coefficient (r) for Indion GS-300 = 0.9997)

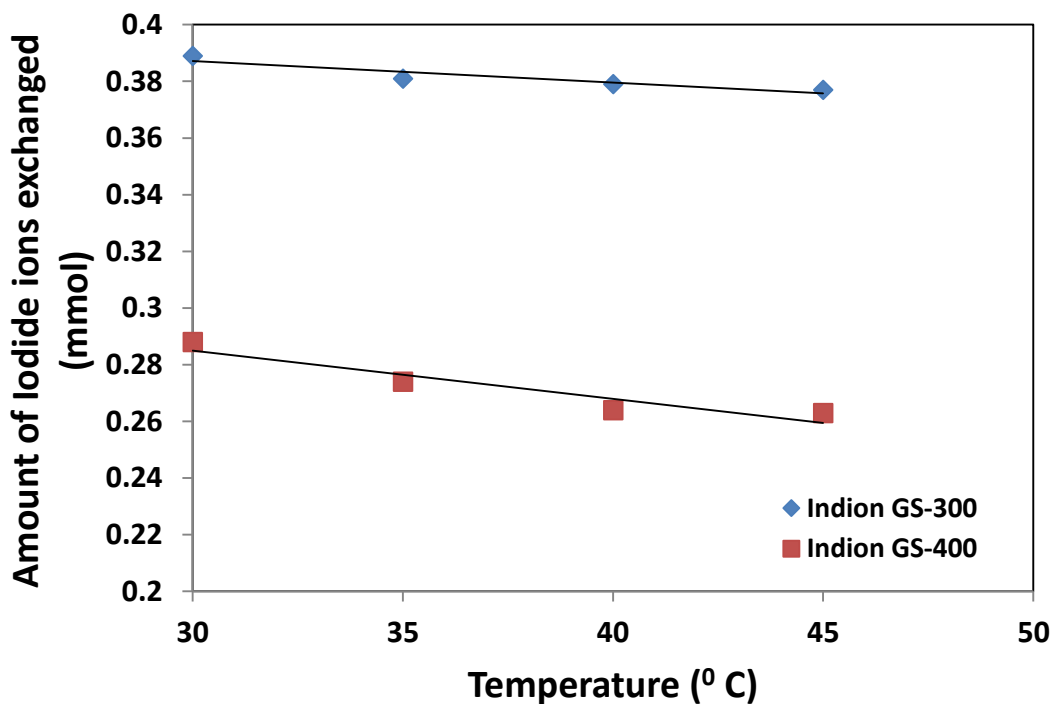


Figure 6: Correlation between Temperature of exchanging medium and amount of iodide ion exchanged (Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol, Correlation coefficient (r) for Indion GS-400 = -0.9447, Correlation coefficient (r) for Indion GS-300 = -0.9327)

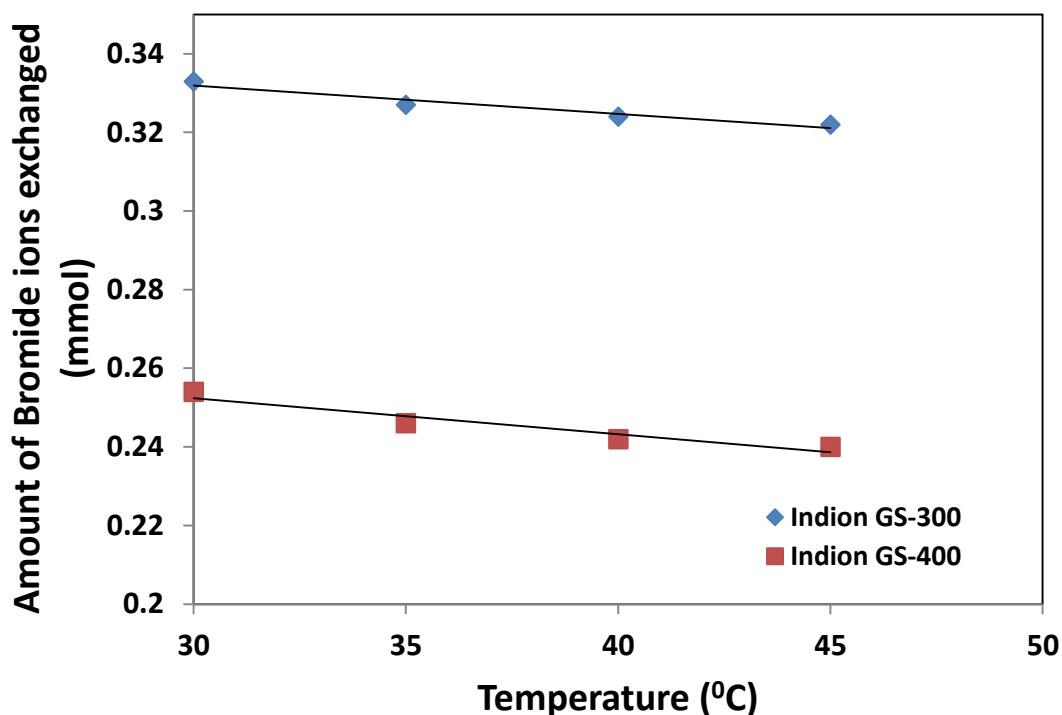


Figure 7: Correlation between Temperature of exchanging medium and amount of bromide ion exchanged (Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.002 M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.500 mmol, Correlation coefficient (r) for Indion GS-400 = -0.9592, Correlation coefficient (r) for Indion GS-300 = -0.9691)

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 [11-16]. 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[11-16].

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. 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 Indion GS-300 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 0.318 to 0.347 min⁻¹, while for bromide ion-isotopic exchange the values increases from 0.249 to 0.277 min⁻¹.

Similarly, in case of Indion GS-400, under identical experimental conditions, the values of specific reaction rate for iodide ion-isotopic exchange increases from 0.196 to 0.231 min⁻¹, while for bromide ion-isotopic exchange the values increases from 0.150 to 0.180 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 Indion GS-300 the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.333 to 0.319 min⁻¹, while for bromide ion-isotopic exchange the values decreases from 0.264 to 0.250 min⁻¹.

Similarly, in case of Indion GS-400, under identical experimental conditions, the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.215 to 0.195 min⁻¹, while for bromide ion-isotopic exchange the values decreases from 0.169 to 0.151 min⁻¹. In case of Indion GS-300 resins during iodide ion isotopic exchange reaction, the enthalpy and energy of activation values calculated were -357.0 and 2224.5 J.mol⁻¹ respectively, which were less than the respective values of 296.6 and 2878.1 J.mol⁻¹ obtained during bromide ion-isotopic exchange reaction. Similar trend was observed for Indion GS-400 resins during the two ion-isotopic exchange reactions (Table 4). 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

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

Previous studies [17, 18] on halide ion distribution coefficient on strong and weak basic anion exchange resins indicate that the selectivity coefficient between halide ions increased at higher electrolyte concentrations. Adachi *et al.* [19] 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.* [20]; in which it was 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 Indion GS-300 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 19.0 to 20.9, while for bromide ions the values increases from 14.1 to 16.0. Similarly, in case of Indion GS-400, under identical experimental conditions, the log K_d values for iodide ions increases from 16.9 to 19.1, while for bromide ions the values increases from 12.0 to 13.9. 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 Indion GS-300 the log K_d values for iodide ions decreases from 20.6 to 18.4, while for bromide ions the values decreases from 15.1 to 13.5. Similarly, in case of Indion GS-400, under identical experimental conditions, the log K_d values for iodide ions decreases from 18.4 to 16.5, while for bromide ions the values decreases from 12.9 to 11.5. It was also observed that the K_d values for iodide ion-isotopic exchange reactions were calculated to be higher than that for bromide ion-isotopic exchange reactions (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 reactions under identical experimental conditions of 35.0°C, 1.000 g of ion exchange resins and 0.002 M labeled iodide ion solution, by using Indion GS-300 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 0.324, 0.381, 0.124 and 19.7 respectively, which was higher than 0.206, 0.274, 0.057 and 17.7 respectively as that obtained by using Indion GS-400 resins. The identical trend was observed for the two resins during bromide ion-isotopic exchange reactions.

In case of Indion GS-300 resins during the iodide ion-isotopic exchange reaction, the enthalpy and energy of activation values calculated were -357.0 and 2224.5 J.mol⁻¹ respectively, which were less than the respective values of 2677.1 and 5258.6 J.mol⁻¹ obtained for the same reaction using Indion GS-400 resins. Similar trend was observed for the two resins during bromide ion-isotopic exchange reaction (Table 4). The low enthalpy and energy of activation values calculated during both the ion-isotopic exchange reactions for Indion GS-300 resins indicate more efficient ion exchange as compared to Indion GS-400 resins.

From Table 3, it is observed that using Indion GS-300 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 75.32 % to 79.98 %. While using Indion GS-400 resins under identical experimental conditions the percentage of iodide ions exchanged increases from 52.58 % to 61.14 %. Similarly, in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged increases from 64.12 % to 68.78 % using Indion GS-300 resin, while for Indion GS-400 resin it increases from 47.50 % to 51.49 %. The effect of ionic concentration on percentage of ions exchanged is graphically represented in Figure 2.

From Table 4, it is observed that using Indion GS-300 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 77.80 % to 75.45 %. While using Indion GS-400 resins under identical experimental conditions the percentage of iodide ions exchanged decreases from 57.66 % to 52.55 %. Similarly, in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged decreases from 66.60 % to 64.30 % using Indion GS-300 resin, while for Indion GS-400 resin it decreases from 50.75 % to 47.90 %. 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 GS-400 resins, Indion GS-300 resins shows higher percentage of ions exchanged. Thus Indion GS-300 resins show superior performance than Indion GS-400 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 0.9997 and 0.9991 for Indion GS-300 and Indion GS-400 resins respectively, while for bromide ion-isotopic exchange reaction, the respective values of r were calculated as 0.9997 and 0.9999.

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 for Indion GS-300 and Indion GS-400 resins were calculated as -0.9327 and -0.9447 respectively. Similarly, in case of bromide ion-isotopic exchange reactions, the r values were calculated as -0.9691 and -0.9592 respectively for both the resins.

4. CONCLUSION

Although there are number of techniques reported in the literature on characterization of ion exchange resins, radio isotopic tracer technique has emerged as one of the effective non-destructive analytical techniques. The present research paper therefore is a successful demonstration on application of radio tracer isotopes for performance evaluation of Indion GS-300 (nuclear grade) and Indion GS-400 (non-nuclear grade) resins. The experimental work carried out in the present investigation will help to standardize the operational process parameters like temperature and the ionic concentration of the external exchanging medium 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. It is anticipated that the results of such work will be useful in the selection of those resins for stipulated industrial application.

ACKNOWLEDGEMENT

The author is thankful to Professor Dr. R.S. Lokhande (*Retired*) for his valuable help and support by providing the required facilities so as to carry out the experimental work in Radiochemistry Laboratory, Department of Chemistry, University of Mumbai, Vidyanagari, Mumbai -58.

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