

## **Ion-Isotopic Exchange Reaction Kinetics in Characterization of Anion Exchange Resins Dowex 550A LC and Indion-820**

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**Abstract** The present paper involves application of nondestructive radioactive tracer technique in characterization of Dowex 550A LC and Indion-820 anion exchange resins. The characterization study was based on kinetic of exchange reactions between inactive iodide/bromide ions on the resins with radioactive iodide/bromide ions in the solution. During iodide exchange reaction performed at a constant temperature of 40.0<sup>0</sup>C, using 1.000 g of ion exchange resins and labeled iodide ion solution of concentration 0.003 mol/L, for Dowex 550A LC resin the specific reaction rate and initial rate of ion exchange was 0.271 min<sup>-1</sup> and 0.141 mmol/min respectively, while the amount of iodide ions exchanged was 0.522 mmol, and log K<sub>d</sub> was calculated to be 12.1. The above values calculated for Dowex 550A LC resin was higher than the respective values of 0.108, 0.330, 0.036 and 6.0 as obtained for Indion-820 resins. Similar results were obtained for the two resins during bromide exchange reactions, thereby indicating superior performance of Dowex 550A LC resin over Indion-820 resins.

**Keywords:** non destructive technique; radioactive isotopes; Indion-820; Dowex 550A LC; ion-isotopic exchange reaction.

## **Introduction**

Ion exchange technology has been applied for many years in nuclear fuel cycle operations and other activities involving the treatment of radioactive liquids (1). In nuclear power plants ion exchange technology is widely applied in- primary coolant (water) purification, treatment of primary effluents and fuel storage pond water, steam generator blow-down demineralization, for treatments of liquid waste and drainage water, purification of boric acid for recycling, condensate polishing (for nuclear power plants with boiling water reactors) (2-8). Ion exchange materials can be categorized according to their suitability for different applications. Nuclear grade ion exchange resins are normally used when liquids from primary circuits or fuel pools are purified. Nuclear grade ion exchangers are similar to commercial grade resins but have a tighter specification for particle size and composition. The used resins can be restored to the original ionic form by eluting the absorbed radioisotopes with appropriate solutions and can be reused for a number of treatment cycles. The continuous efforts are made to develop new ion exchangers for their specialized applications in nuclear industries (9-14) and different aspects of ion exchange technologies have been continuously studied to make them more economical and efficient in number of industrial applications (15-18). However since the type of ion exchange material to be selected in any of the technological application is based on the requirement of the system and also on their

performance in particular separation process, it is believed that the data based on actual experimental trials will be more significant. Hence it is important to carry out the systematic evaluation of such ion exchange resins in order to understand their performance under various operational conditions. Such types of evaluation will further help in the characterization of different ion exchange resins.

Although literature study reveals existence of wide range of ion exchange characterization techniques (19-21), but the technique involving use of radioactive tracer isotopes are more interesting mainly because of their detection sensitivity which is very high. The radio isotopes used are physico-chemically compatible with the material under study and can be detected in-situ (22, 23). As a result radioisotopes are widely applied in almost all industrial sectors (22, 23) and literature also shows extensive scope of radiotracer methodology (24-30). Considering the extensive technological application of radioactive tracer isotopes, attempts are made in the present study to use the same technique in order to trace the kinetics of ion-isotopic exchange reactions in Dowex 550A LC (nuclear grade) and Indion-820 (non-nuclear grade) anion exchangers. It is believed that the results of present study will be useful in characterization of these resins as well as in standardization of the process parameters in order to bring about the efficient use of the resins in various industrial applications.

## 2. Experimental

**2.1 Ion exchange resins** Dowex 550A LC is a gel Type I strongly basic nuclear grade anion exchange resins in hydroxide form having quaternary ammonium functional group (by Dow Chemical Company, Midland, Michigan) while Indion-820 is a macroporous strong base anion exchange resin in chloride form (by Ion Exchange India Ltd., Mumbai). The information regarding physico-chemical properties of the resins are given in Table 1.

**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)
Dowex 550A LC	Styrene-DVB	$-N^+R_3$	0.3-1.2	63	0-14	60	1.1
Indion-820	Polystyrene Copolymer	$-N^+R_3$	0.3-1.2	50	0-14	40	1.1

**2.2 Treatment of Ion exchange resins** The resins were equilibrated separately with 10 % KI / KBr solution in an ion exchange column in order to convert them to required iodide / bromide form. The loosely adsorbed iodide/bromide ions on the resins were removed by washing them with double distilled water. These conditioned resins were placed in desiccators over P<sub>2</sub>O<sub>5</sub> and dried at room temperature.

**2.3 Radioisotopes** The radioactive isotopes used for the study was supplied by Board of Radiation and Isotope Technology (BRIT), Mumbai, India. The information regarding the isotopes used in our study is given in Table 2.

**Table 2.** Properties of <sup>131</sup>I and <sup>82</sup>Br tracer isotopes (22)

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

\* Sodium iodide in dilute sodium sulphite.

\*\* Ammonium bromide in dilute ammonium hydroxide

**2.4 Kinetics of active and inactive iodide ion exchange reaction** <sup>131</sup>I radioactive solution was used to label 250 mL (*V*) of 0.001 mol/L iodide ion solution placed in a stoppered bottle. The labeling of iodide ion solution by using <sup>131</sup>I as a radioactive tracer isotope was done using a micro-syringe and the radioactivity was measured by using γ-ray spectrometer having NaI (Tl) scintillation detector. The labeling was done in such a way that 1.0 mL of labeled solution will have radioactivity of about 15,000 cpm (counts per minute). It was observed that the concentration of labeled solution remains unchanged since only μL of the radioactive iodide ion solution was used for labeling. The same was confirmed by potentiometer titration against silver nitrate solution. The labeled iodide ion solution of known initial radioactivity (*A<sub>i</sub>*) was placed in a water bath which was previously adjusted to 30.0 °C. The 1.000 g (*m*) of dry, freshly conditioned resins in iodide form was equilibrated with labeled iodide ion solution. The mechanical stirrer was used to stir the solution and the radioactivity of 1.0 mL equilibrated solution was measured at a fixed interval of every 2.0 min. After measuring the radioactivity, the solution was transferred to the bottle having labeled solution. The exchange reaction between inactive and radioactive iodide ions can be represented as:

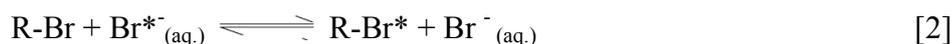


Here R represents resin phase; I\*<sub>(aq)</sub> represents labeled radioactive iodide ions in aqueous solution.

The solution was equilibrated for three hours which was sufficient time to attain the equilibrium (31-35). After the period of three hours, the final radioactivity ( $A_f$ ) of the solution was again measured. The background counts were subtracted from the measured radioactivity to give corrected radioactivity.

The experiments were performed in the similar way by equilibrating ion exchange resins in iodide form (1.000g) with radioactive iodide ion solution of different concentrations up to 0.004 mol/L by keeping temperature constant at 30.0 °C. The experimental sets were performed for different temperatures up to 45.0 °C.

**2.5 Kinetics of active and inactive bromide ion exchange reaction** The study was also performed to understand the kinetics of active and inactive bromide ion exchange reactions by equilibrating ion exchange resin in bromide form (1.000 g) with radioactive bromide ion solution in the same way as explained above. For labeling the bromide ion solution,  $^{82}\text{Br}$  radioactive isotope was used and the procedure adopted for labeling was same as explained above. The exchange reaction between inactive and radioactive bromide ions can be represented as:



Here R represents resin phase;  $\text{Br}^{*-}_{(\text{aq.})}$  represents radioactive labeled bromide ions in aqueous solution.

**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 = 40.0 °C

Concentration of ionic solution (mol/L)	Amount of ions in 250 mL solution (mmol)	REACTION -1								REACTION -2							
		Dowex 550A LC				Indion-820				Dowex 550A LC				Indion-820			
		Specific reaction rate of rapid process min <sup>-1</sup>	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K <sub>d</sub>	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchanged (mmol/min)	Log K <sub>d</sub>	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K <sub>d</sub>	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K <sub>d</sub>
0.001	0.250	0.248	0.163	0.040	10.1	0.091	0.106	0.010	5.1	0.203	0.132	0.027	8.9	0.080	0.085	0.007	2.0
0.002	0.500	0.260	0.336	0.087	11.5	0.099	0.216	0.021	5.6	0.215	0.279	0.060	9.9	0.087	0.178	0.015	2.5
0.003	0.750	0.271	0.522	0.141	12.1	0.108	0.330	0.036	6.0	0.224	0.427	0.096	10.4	0.095	0.270	0.026	3.1
0.004	1.000	0.288	0.710	0.204	12.9	0.119	0.448	0.053	6.5	0.245	0.576	0.141	11.0	0.106	0.386	0.041	3.6

**Table 4.** Temperature effect on Ion-Isotopic Exchange Reactions

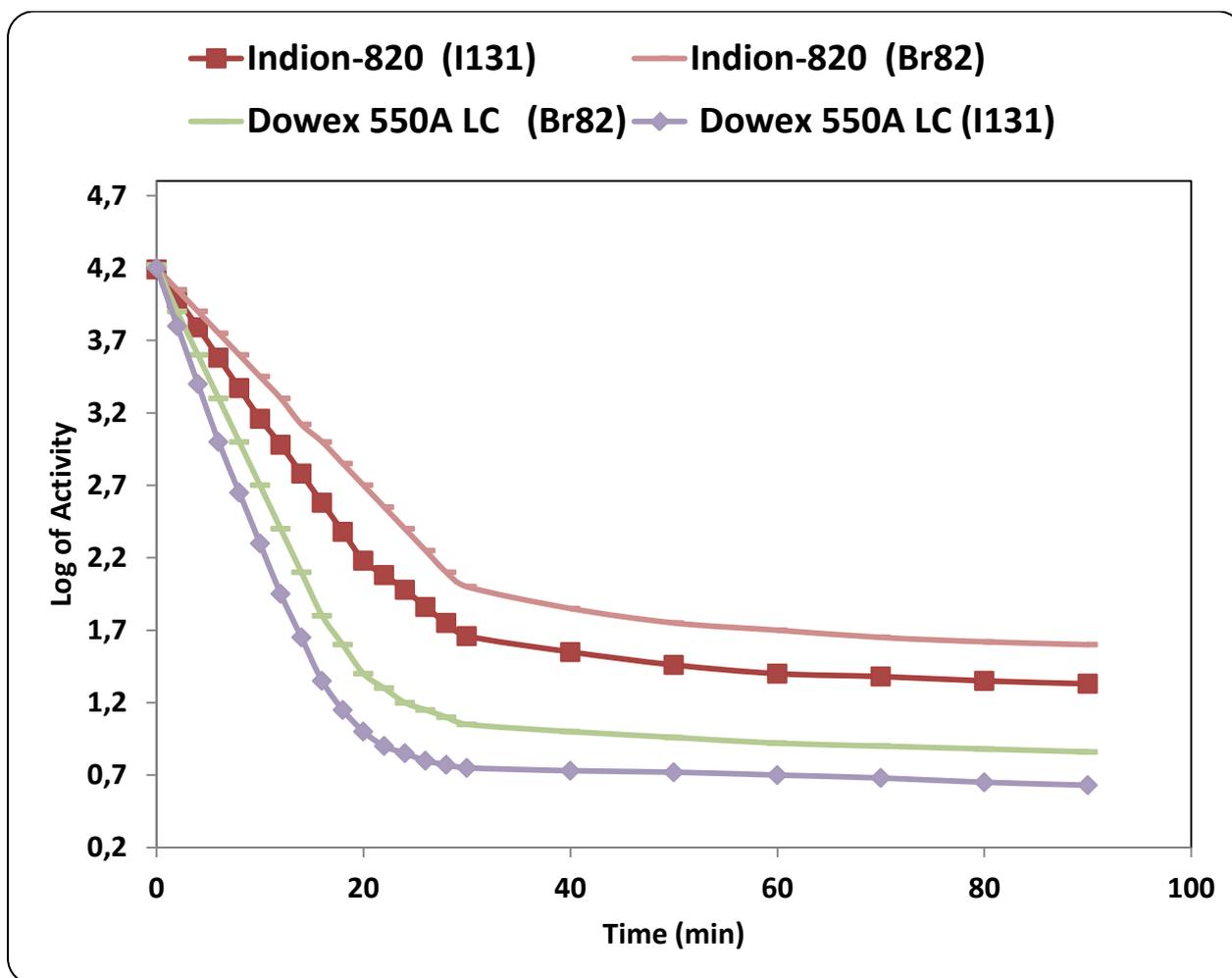
Amount of ion exchange resin = 1.000 g

Concentration of labeled exchangeable ionic solution = 0.003 mol/L

Volume of labeled ionic solution = 250 mL

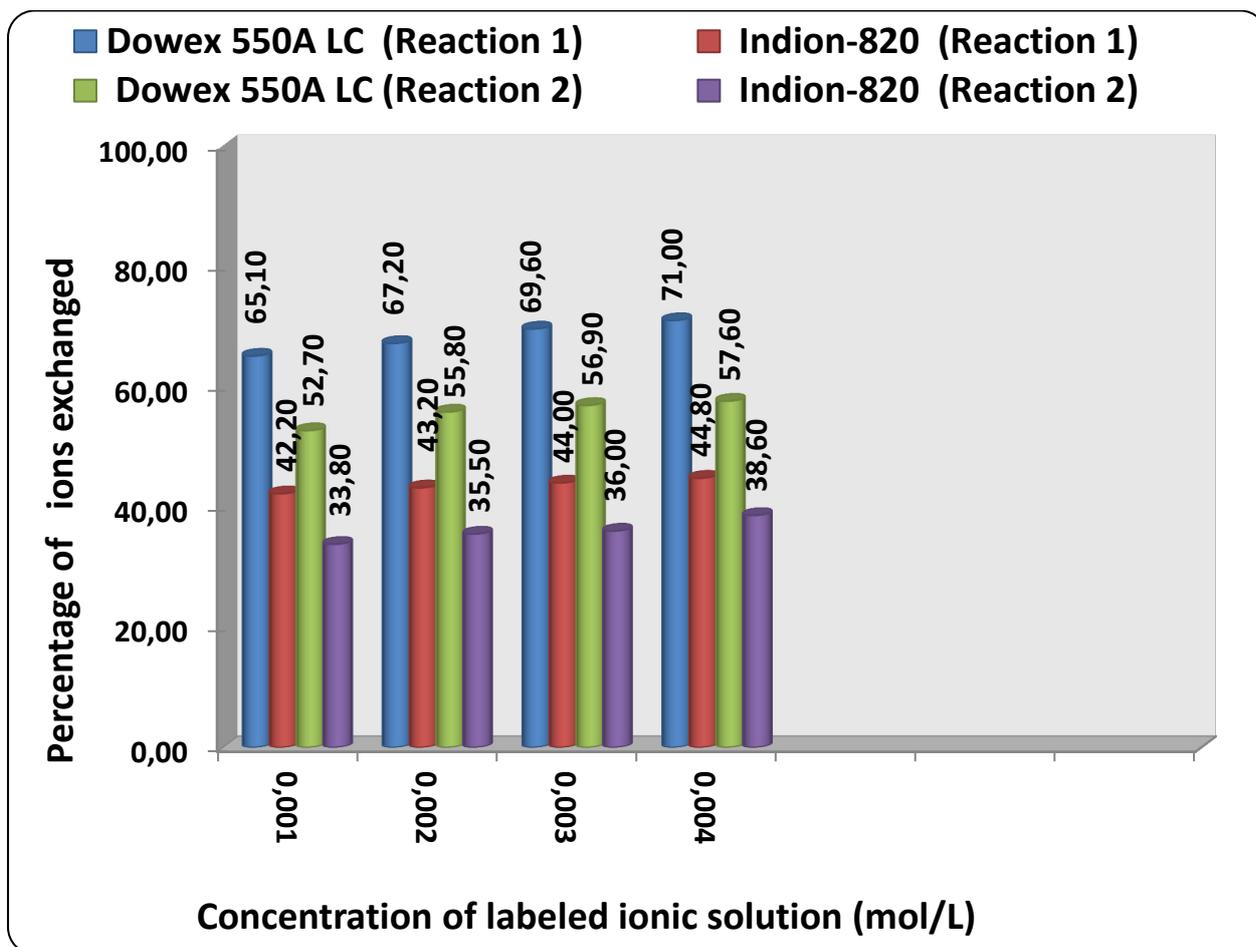
Amount of exchangeable ions in 250 mL labeled solution = 0.750 mmol

Temperature °C	REACTION -1								REACTION -2							
	Dowex 550A LC				Indion-820				Dowex 550A LC				Indion-820			
	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K <sub>d</sub>	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K <sub>d</sub>	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K <sub>d</sub>	Specific reaction rate of rapid process min <sup>-1</sup>	Amount of bromide ion exchanged (mmol)	Initial rate of bromide ion exchange (mmol/min)	Log K <sub>d</sub>
30.0	0.291	0.540	0.157	13.3	0.132	0.343	0.045	7.5	0.251	0.449	0.113	11.3	0.118	0.307	0.036	4.3
35.0	0.282	0.527	0.148	13.0	0.121	0.337	0.041	6.9	0.236	0.435	0.103	10.8	0.103	0.291	0.030	3.4
40.0	0.271	0.522	0.141	12.1	0.108	0.330	0.036	6.0	0.224	0.427	0.096	10.4	0.095	0.270	0.026	3.1
45.0	0.257	0.507	0.130	11.8	0.099	0.327	0.032	5.7	0.216	0.407	0.088	10.0	0.086	0.261	0.022	2.8



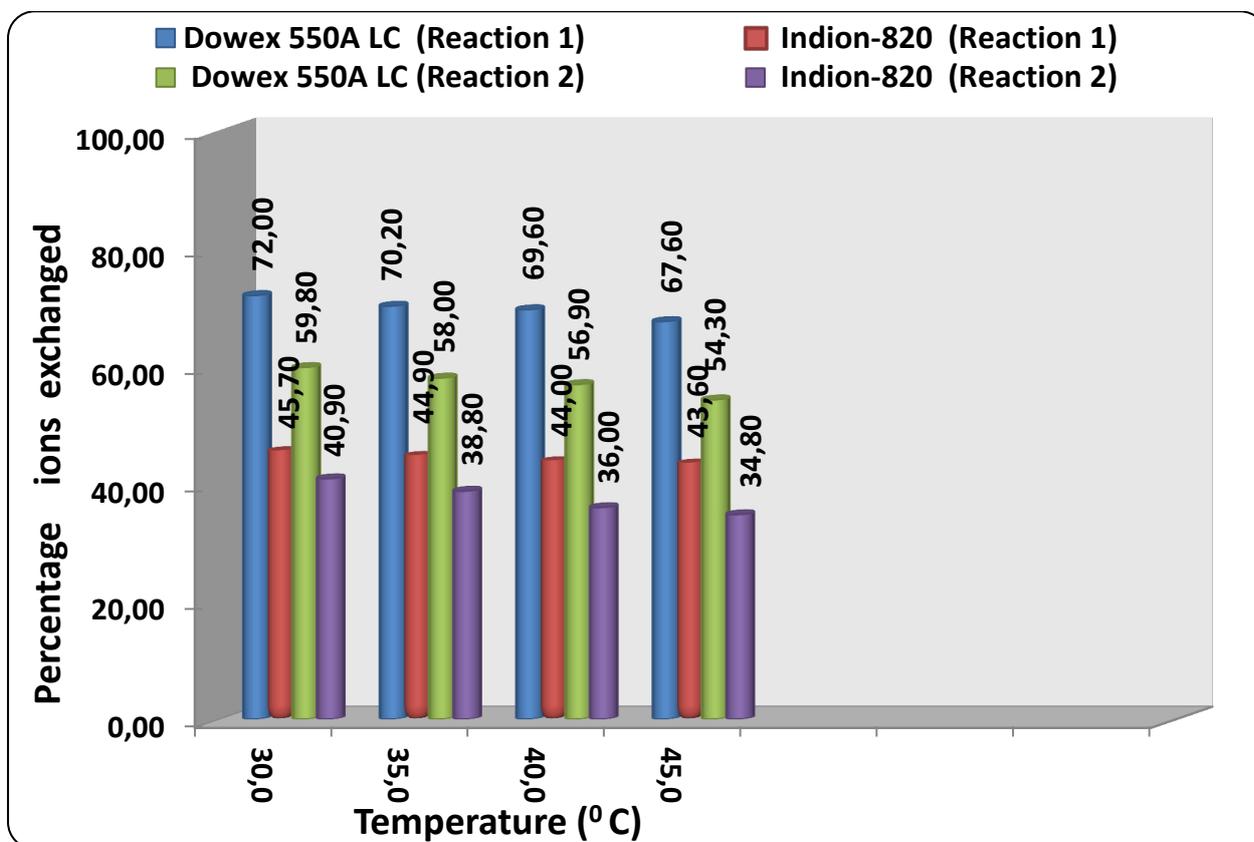
**Figure 1:** Kinetics of ion-isotopic exchange reactions

Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.003 mol/L, Volume of labeled ionic solution = 250 mL, Temperature = 40.0 °C



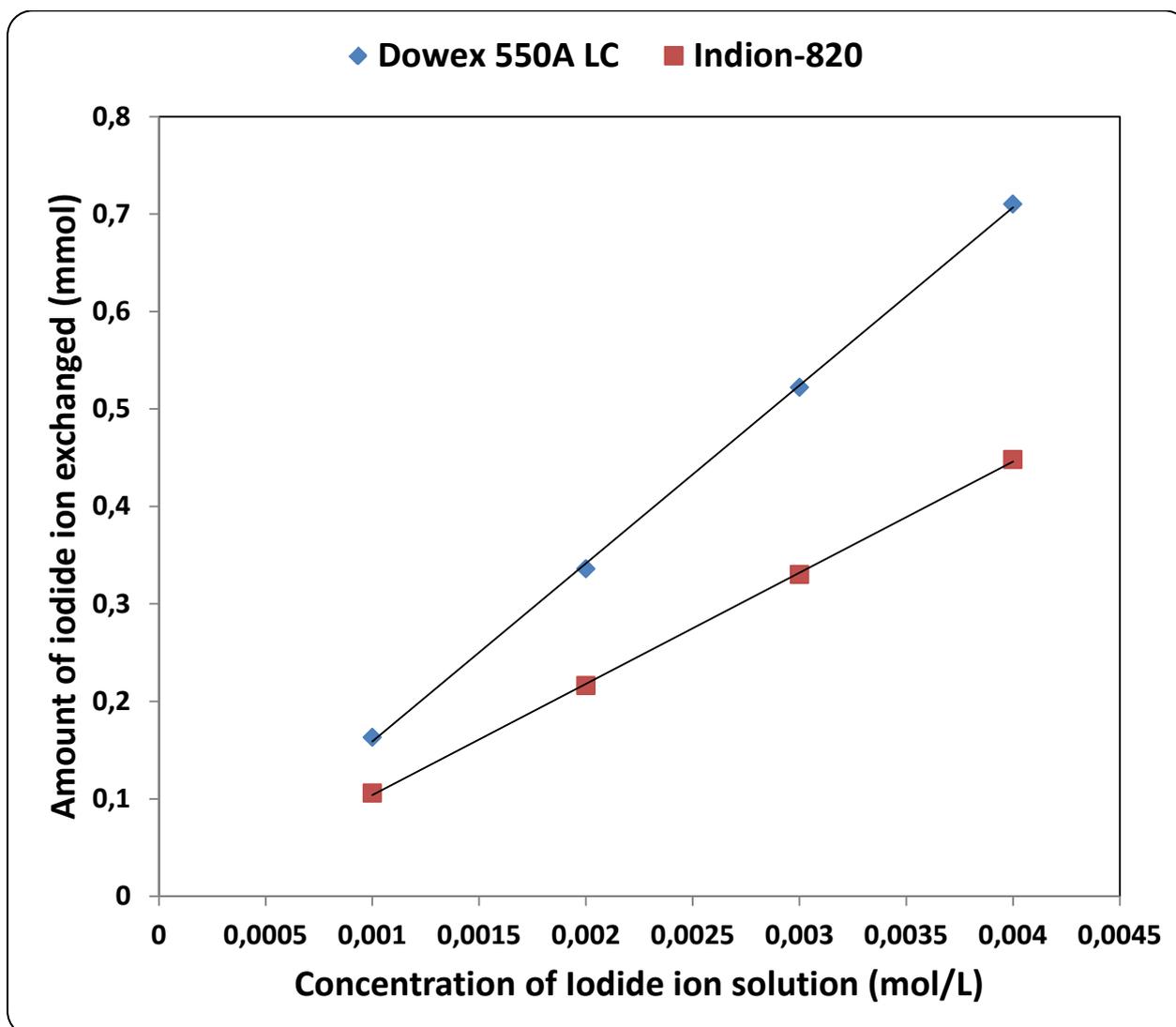
**Figure 2:** Variation in percentage ions exchanged with concentration of labeled ionic solution

Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 40.0 °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 = 0.003 mol/L, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.750 mmol

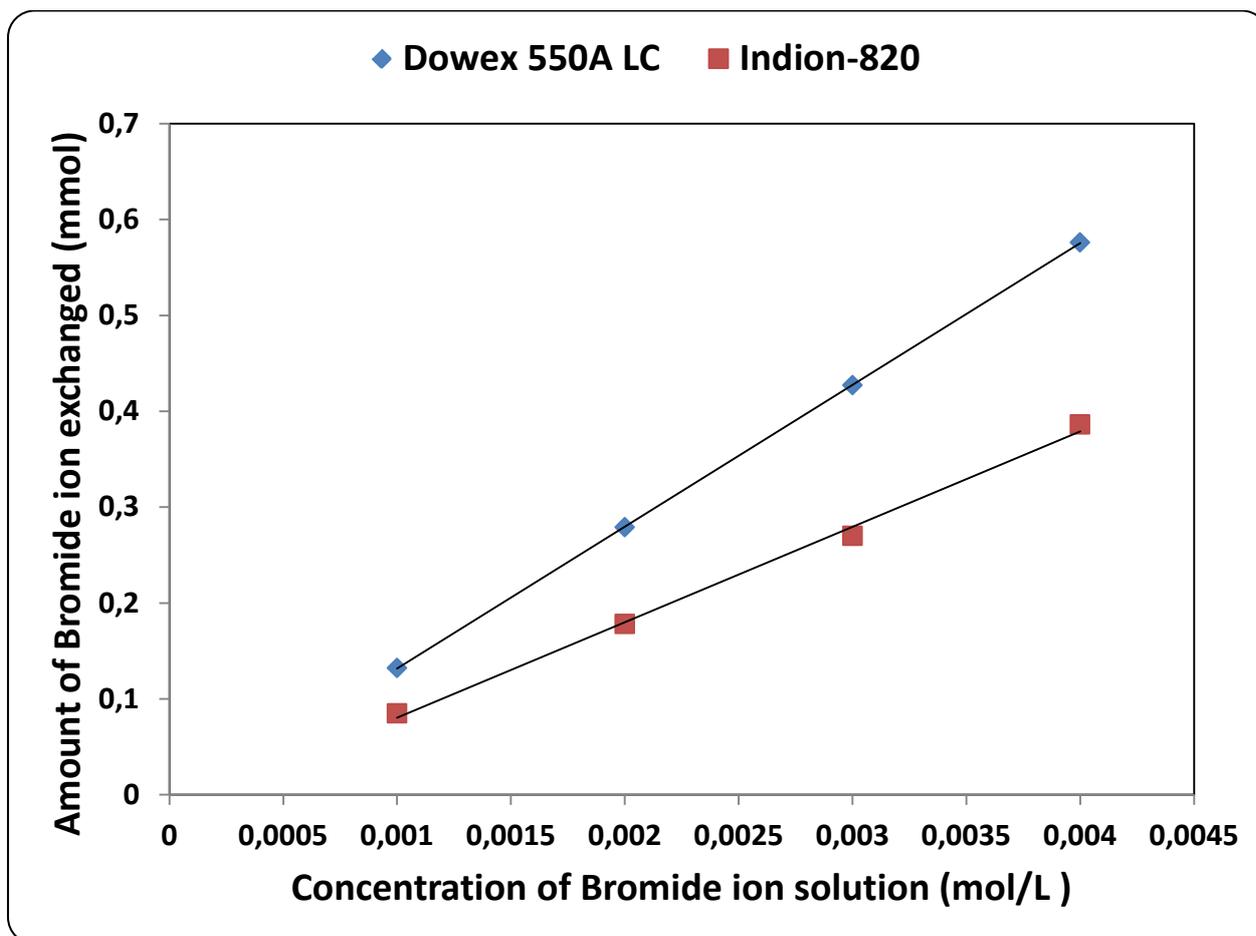


**Figure 4:** Correlation between concentration of iodide ion solution and amount of iodide ion exchanged

Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 40.0 °C

Correlation coefficient (r) for Dowex 550A LC = 0.9998

Correlation coefficient (r) for Indion-820 = 0.9999

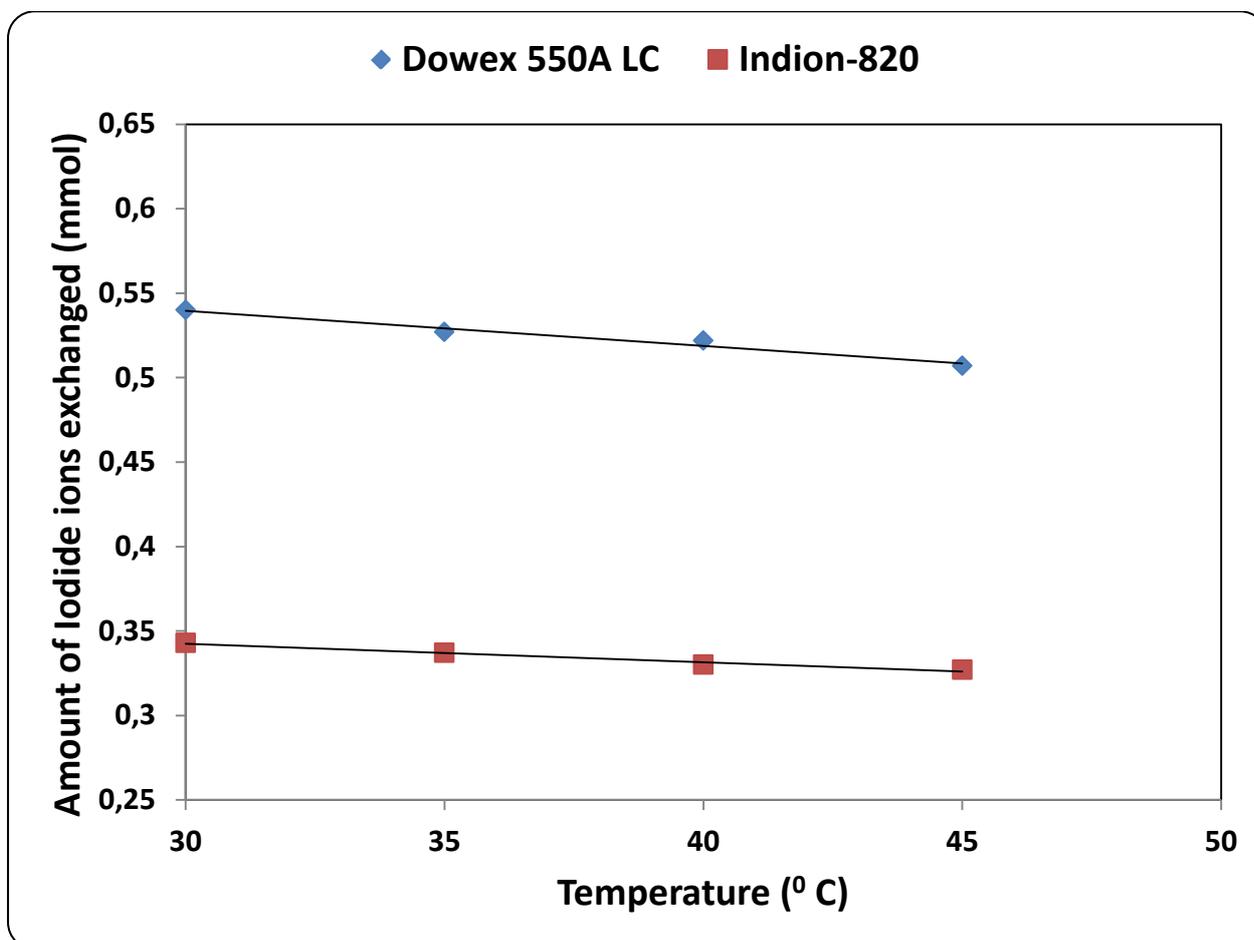


**Figure 5:** Correlation between concentration of bromide ion solution and amount of bromide ion exchanged

Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 40.0 °C

Correlation coefficient (r) for Dowex 550A LC = 1.0000

Correlation coefficient (r) for Indion-820 = 0.9984

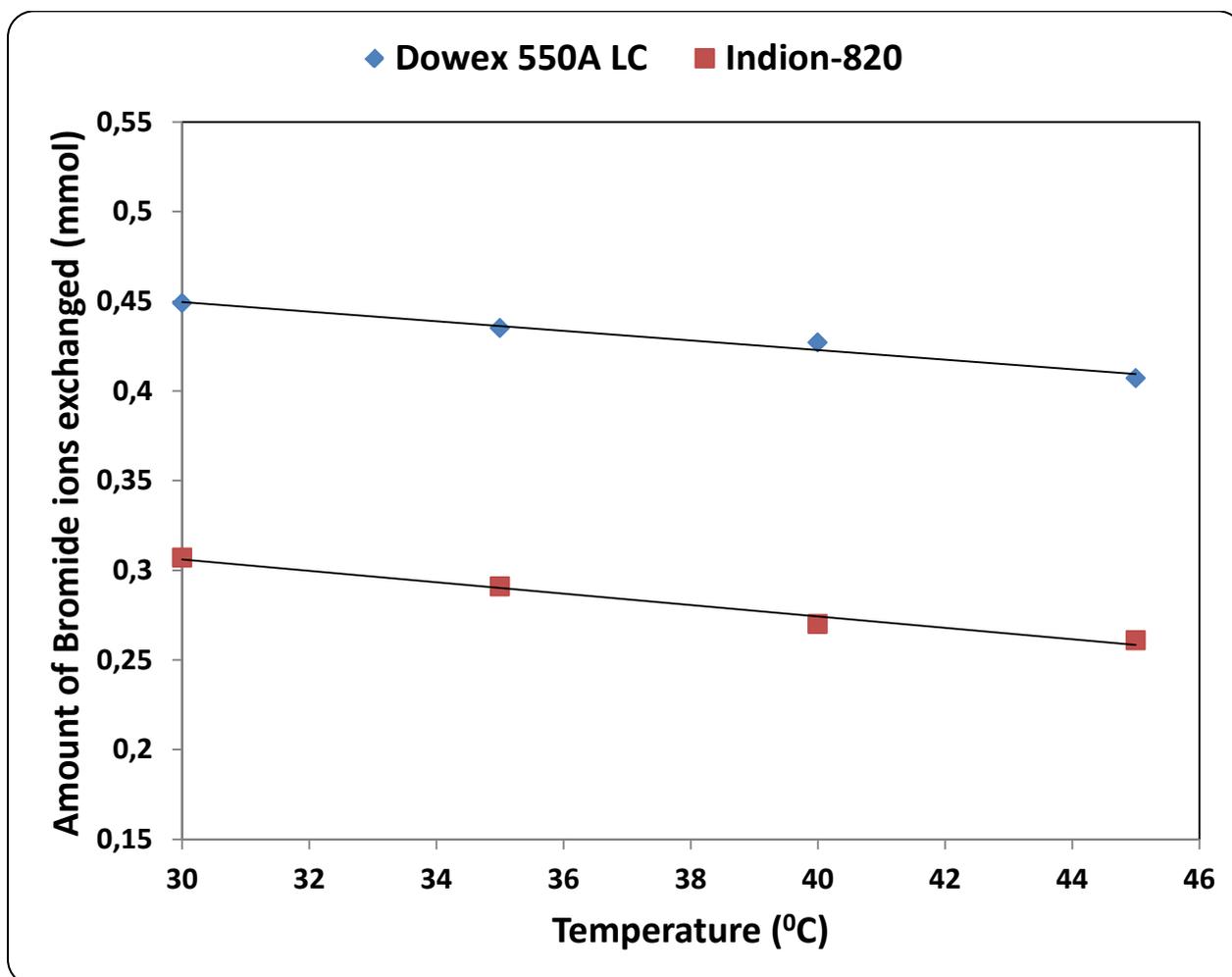


**Figure 6:** Correlation between temperature of exchanging medium and amount of iodide ion exchanged

Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.003 mol/L, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.750 mmol

Correlation coefficient (r) for Dowex 550A LC = -0.9845

Correlation coefficient (r) for Indion-820 = -0.9886



**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 = 0.003 mol/L, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.750 mmol

Correlation coefficient (r) for Dowex 550A LC = -0.9863

Correlation coefficient (r) for Indion-820 = -0.9896

### 3. Results and Discussion

**3.1 Comparative study of iodide and bromide ion-isotopic exchange reactions** In the present study it was observed that the activity of solution decreases rapidly initially due to the rapid ion-isotopic exchange reaction, further the activity of the solution decreases slowly due to the slow ion-isotopic exchange and near the end it remains nearly constant. Therefore a composite curve of log activity against time was obtained in which the activity of solution decreases sharply in the initial stages and thereafter the activity decreases very slowly giving nearly straight line (Figure 1). The graph indicates simultaneous taking place of rapid and slow ion-isotopic exchange reactions. The straight line of the composite curve when extrapolated back to zero time gives the contribution of slow process to the total activity which now includes rapid process also. The activity due to rapid process was obtained by subtracting the activity due to slow process from the total activity at different intervals of time. The specific reaction rates ( $k$ ) of rapid ion-isotopic exchange reaction were calculated from the activity exchanged due to rapid process at various time intervals. From the amount of exchangeable ions in 250 mL of solution and knowing the initial and final activity of solution; amount of iodide / bromide ions exchanged (mmol) on the resin were obtained. The initial rate of ion exchanged (mmol/min) was calculated from the knowledge of amount of ions exchanged on the resin (mmol) and the specific reaction rates ( $\text{min}^{-1}$ ).

Because of smaller solvated size of iodide ions as compared to that of bromide ions, it was observed that the exchange of iodide ions occurs at the faster rate than that of bromide ions. Hence under similar experimental conditions, the higher values of specific reaction rate ( $\text{min}^{-1}$ ), amount of ion exchanged (mmol) and initial rate of ion exchange (mmol/min) were obtained for iodide ion-isotopic exchange reactions as compared to that for bromide ion-isotopic exchange reactions (Tables 3 and 4). For both the ion-isotopic exchange reactions, under similar experimental conditions, the values of specific reaction rate observed to increase with the ionic concentration (Table 3). However, the same values were observed to decrease with hike in temperature (Table 4). Thus in case of Dowex 550A LC at  $40.0^{\circ}\text{C}$  when the concentration of iodide and bromide ions in solution was raised from 0.001 to 0.004 mol/L, the specific reaction rate values for iodide ion-isotopic exchange increases from 0.248 to  $0.288 \text{ min}^{-1}$ , while for bromide ion-isotopic exchange the values increases from 0.203 to  $0.245 \text{ min}^{-1}$ . Similarly in case of Indion-820, during iodide ion-isotopic exchange under same experimental conditions, the values increases from 0.091 to  $0.119 \text{ min}^{-1}$ , while for bromide ion-isotopic exchange the values increases from 0.080 to  $0.106 \text{ min}^{-1}$ . However when the

0.003 mol/L concentration of iodide and bromide ions in solution was kept constant and with rise in temperature from 30.0 °C to 45.0 °C, in case of Dowex 550A LC the specific reaction rate values for iodide ion-isotopic exchange decreases from 0.291 to 0.257 min<sup>-1</sup>, while for bromide ion-isotopic exchange the values decreases from 0.251 to 0.216 min<sup>-1</sup>. Similarly in case of Indion-820, under same experimental conditions, the iodide ion-isotopic exchange specific reaction rate values decreases from 0.132 to 0.099 min<sup>-1</sup>, while for bromide ion-isotopic exchange the values decreases from 0.118 to 0.086 min<sup>-1</sup>. From the results, it appears that bromide ions exchange at the slower rate as compared to that of iodide ions which was related to the extent of solvation (Tables 3 and 4).

From the knowledge of initial activity ( $A_i$ ), final activity ( $A_f$ ), volume of the external ion solution ( $V$ ) and mass of 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 [36, 37] 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.* [38] observed that the swelling pressure of the resin decreased at higher solute concentrations resulting in higher  $K_d$  values. The dependence of  $K_d$  values on temperature using cation exchange resin was studied by Shuji *et al.* [39]; were they observed that the values of  $K_d$  increases as the temperature is decreased. The results of present experimental also indicates that the  $K_d$  values for bromide and iodide ions increases with increase in ionic concentration, however the  $K_d$  values were found to decrease with rise in temperature. Thus in case of Dowex 550A LC at 40.0°C when the concentration of iodide and bromide ions in solution increases, the values of log  $K_d$  for iodide ions increases from 10.1 to 12.9, while for bromide ions the values increases from 8.9 to 11.0. Similarly in case of Indion-820, under same conditions of experiment, the values of log  $K_d$  for iodide ions rises from 5.1 to 6.5, while for bromide ions the values increases from 2.0 to 3.6. However when the concentration 0.003 mol/L of iodide and bromide ions solution is kept constant and temperature is increased from 30.0 °C to 45.0 °C, in case of Dowex 550A LC the log  $K_d$  values for iodide ions decreases from 13.3 to 11.8, while for bromide ions the values decreases from 11.3 to 10.0. Similarly in case of Indion-820, under same conditions of experiment, the values of log  $K_d$  for iodide ions decreases from 7.5 to 5.7, while for bromide ions the values decreases from 4.3 to 2.8. 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 Dowex 550A LC and Indion-820 anion exchange resins** From the results presented in Table 3 and 4, it is observed that during iodide ion-isotopic exchange reaction by using Dowex 550A LC resin, the values of specific reaction rate ( $\text{min}^{-1}$ ), amount of iodide ion exchanged (mmol), initial rate of iodide ion exchange (mmol/min) and  $\log K_d$  were 0.271, 0.522, 0.141 and 12.1 respectively, which was higher than 0.108, 0.330, 0.036 and 6.0 respectively as that obtained by using Indion-820 resins under identical experimental conditions of  $40.0^\circ\text{C}$ , 1.000 g of ion exchange resins and concentration of labeled iodide ion solution 0.003 mol/L. The similar trend was indicated for the two resins during bromide ion-isotopic exchange reaction.

From Table 3, it is observed that at a constant temperature of  $40.0^\circ\text{C}$ , with increase in concentration of labeled iodide ion solution, the iodide ions exchanged percentage increases from 65.10 % to 71.00 % using Dowex 550A LC resins and from 42.20 % to 44.80 % using Indion-820 resins. Similarly in case of bromide ion-isotopic exchange reactions under identical conditions of experiment, the bromide ions exchanged percentage increases from 52.70 % to 57.60 % using Dowex 550A LC resin and from 33.80 % to 38.60 % using Indion-820 resin. The effect of ionic concentration on percentage of ions exchanged is graphically represented in Figure 2.

From Table 4, it is observed by using 0.003 mol/L labeled iodide ion solution, as the temperature increases from  $30.0^\circ\text{C}$  to  $45.0^\circ\text{C}$ , the percentage of iodide ions exchanged decreases from 72.00 % to 67.60 % using Dowex 550A LC resins and from 45.70 % to 43.60 % using Indion-820 resins. Similarly under identical experimental conditions, in case of bromide ion-isotopic exchange reactions, the bromide ions exchanged percentage decreases from 59.80 % to 54.30 % using Dowex 550A LC resin and from 40.90 % to 34.80 % using Indion-820 resin. 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, Dowex 550A LC resins shows higher percentage of ions exchanged. Thus Dowex 550A LC resins show superior performance over Indion-820 resins under identical operational parameters.

**3.3 Statistical analysis** The results of present investigation show a strong positive linear correlation between amount of ions exchanged and concentration of ionic solution (Figures 4, 5). In case of iodide ion-isotopic exchange reaction, the respective values of correlation coefficient ( $r$ ) for Dowex 550A LC and Indion-820 resins were calculated as 0.9998 and

0.9999, while in case of bromide ion-isotopic exchange reaction, the  $r$  values was calculated as 1.0000 and 0.9984 respectively for the two resins.

There also exist a strong negative correlation 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 Dowex 550A LC and Indion-820 resins were -0.9845 and -0.9886 respectively. Similarly in case of bromide ion-isotopic exchange reactions the  $r$  values calculated were -0.9863 and -0.9896 respectively for the two resins.

#### **4. Conclusion**

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 radioisotopic tracer technique used here can also be applied further for characterization of different nuclear as well as non-nuclear grade ion exchange resins.

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