Comparative Study of Anion Exchange Resins Indion H-IP and Indion-NSSR by Application of ^{131}I and ^{82}Br as a Tracer Isotope

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Abstract   The present investigation deals with characterization of weakly basic and strongly basic industrial grade anion exchange resins Indion H-IP and Indion-NSSR. The characterization was performed on the basis of iodide and bromide ion-isotopic exchange reaction kinetics by application of ^{131}I and ^{82}Br tracer isotopes. It was observed that due to solvation effect the iodide ions are exchanged at faster rate as compared to bromide ions when the experimental conditions are kept identical. For both the ion-isotopic exchange reactions it was observed that the reaction rate is having negative correlationship with temperature and positive correlationship with concentration of exchangeable ions in solution. Also under identical experimental conditions, Indion-NSSR shows higher percentage of ion exchange than Indion H-IP resins.

Keywords   Indion H-IP, Indion-NSSR, Solvation Effect, Ion-isotopic Exchange Reactions, Tracer Applications, Reaction Kinetics, Radioactive Isotopes, ^{131}I, ^{82}Br

1. Introduction

The use of ion exchange procedures in chemical processing, water and wastewater treatments was well developed by the time the technique was first applied in the nuclear industry. Since then much progress has been made in improving the technology, and ion exchange methods have been widely used to remove soluble radionuclides from liquid waste. Recent developments in ion exchange technology and applications are described in other publications[1, 2]. The process involves the exchange of ionic species between a liquid solution and a solid matrix containing ionizable polar groups. Efforts to develop new organic ion exchangers for specific applications are continuing[3-7] and various aspects of ion exchange technologies have been continuously studied to improve the efficiency and economy of their application in various technological applications[8-11]. 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. Hence in the present investigation, it is proposed to test the performance of industrial grade ion exchange resin 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

Ion exchange resin Indion H-IP is a weekly (intermediate) basic anion exchange resin while Indion-NSSR is a strongly basic anion exchange resin supplied by Ion Exchange India Ltd., Mumbai. Both the resins as supplied by the manufacturer were in chloride form having quaternary ammonium -N$^+$R$_3$ functional group. 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$_2$O$_5$ 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.

Table 1.   Properties of ion exchange resins

Ion exchange resin	Resin Type	Matrix	Particle Size (mm)	Moisture content (%)	Operating pH	Maximum operating temperature (℃)	Total exchange capacity (meq./mL)
Indion H-IP	Iso porous	Crosslinked Polystyrene	0.3–1.2	50	0 – 7	80	1.1
Indion-NSSR	Macro porous	Crosslinked Polystyrene	0.3–1.2	53	0 – 14	100	1.2

Table 2.   Properties of ^{131}I and [82]Br tracer isotopes[12]

| Isotopes	Half-life	Radioactivity / mCi	γ-energy / MeV	Chemical form	Physical form
[131]I	8.04 days	5	0.36	Iodide*	Aqueous
[82]Br	36 hours	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 (Tl) 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 AgNO₃ solution. The above labeled solution of known initial activity *(A_i)* was kept in a thermostat adjusted to 30.0℃. 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:

$$R\text{-}I + I^{*-}_{(aq.)} \rightleftharpoons R\text{-}I^* + I^-_{(aq.)} \tag{1}$$
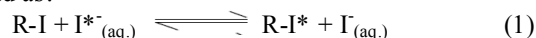
Here R-I represents ion exchange resin in iodide form; $I^{*-}_{(aq.)}$ represents aqueous iodide ion solution labeled with ^{131}I radiotracer isotope.

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 3hours which was sufficient time to attain the equilibrium[13-18]. 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℃. The same experimental sets were repeated for higher temperatures up to 45.0℃.

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:

$$R\text{-}Br + Br^{*-}_{(aq.)} \rightleftharpoons R\text{-}Br^* + Br^-_{(aq.)} \tag{2}$$
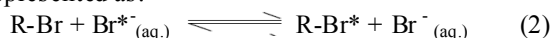
Here R-Br represents ion exchange resin in bromide form; $Br^{*-}_{(aq.)}$ represents aqueous bromide ion solution labeled with [82]Br radiotracer isotope.
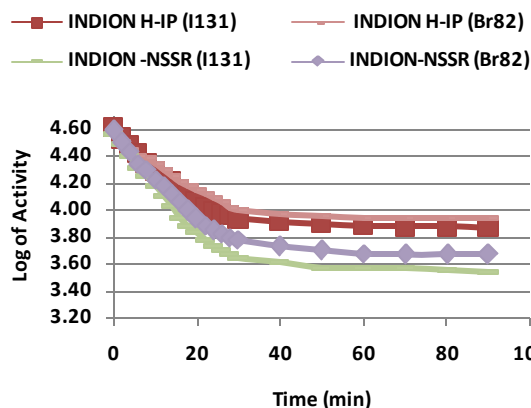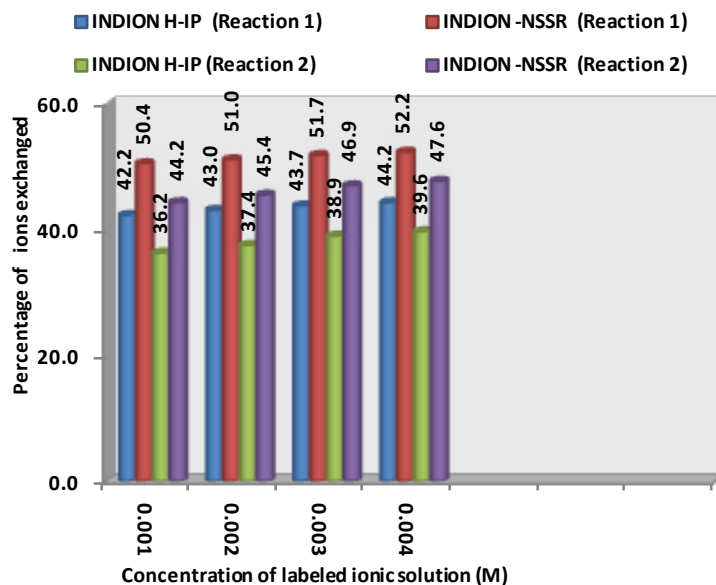

Figure 1.   Kinetics of Ion-Isotopic Exchange Reactions Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.001M, Volume of labeled ionic solution = 250 mL, Temperature = 30.0℃

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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 = 30.0℃

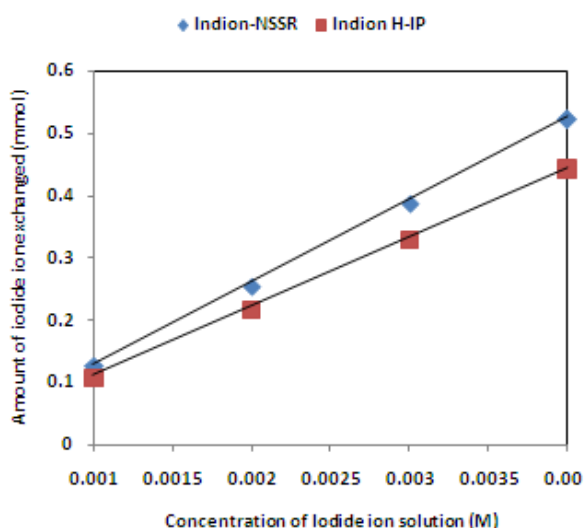| Concentration of ionic solution (M) | Amount of ions in 200 mL solution (mmol) | REACTION -1 | | | | | | | | REACTION -2 | | | | | | | |
| | | INDION H-IP | | | | INDION-NSSR | | | | INDION H-IP | | | | INDION-NSSR | | | |
		Specific reaction rate of rapid process min⁻¹	Amount of iodide ion exchanged (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	0.091	0.106	0.010	5.5	0.162	0.126	0.020	7.6	0.086	0.091	0.008	2.9	0.130	0.111	0.014	3.8
0.002	0.500	0.104	0.215	0.022	6.0	0.176	0.255	0.045	8.2	0.100	0.187	0.019	3.6	0.139	0.227	0.032	5.0
0.003	0.750	0.116	0.328	0.038	6.6	0.187	0.388	0.073	8.8	0.110	0.292	0.032	3.9	0.148	0.352	0.052	5.5
0.004	1.000	0.128	0.442	0.057	7.0	0.200	0.522	0.104	9.2	0.119	0.396	0.047	4.2	0.154	0.476	0.073	5.7

Table 4. Temperature effect on Ion-Isotopic Exchange Reactions Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.001M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.25 mmol

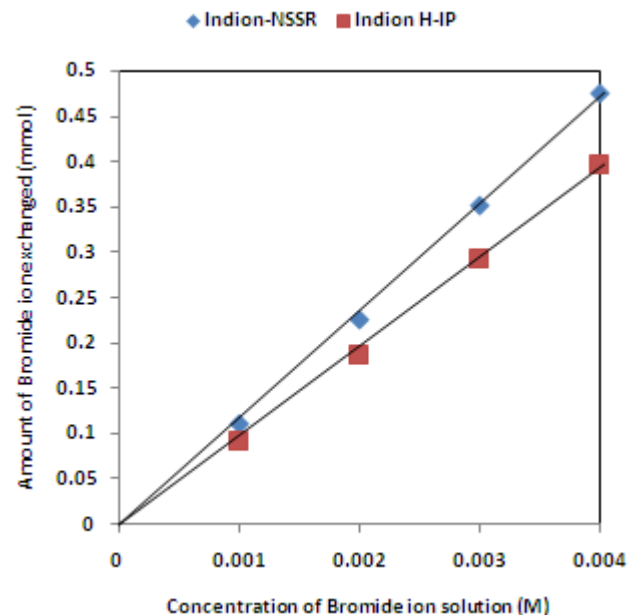| Temperature °C | REACTION -1 | | | | | | | | REACTION -2 | | | | | | | |
| | INDION H-IP | | | | INDION-NSSR | | | | INDION H-IP | | | | INDION-NSSR | | | |
	Specific reaction rate of rapid process min⁻¹	Amount of iodide ion exchanged (mmol)	Initial rate of iodide ion exchange (mmol/min)	Log K_d	Specific 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	0.091	0.106	0.010	5.5	0.162	0.126	0.020	7.6	0.086	0.091	0.008	2.9	0.130	0.111	0.014	3.8
35.0	0.083	0.103	0.009	4.9	0.155	0.123	0.019	7.1	0.080	0.084	0.007	2.1	0.125	0.104	0.013	3.3
40.0	0.072	0.101	0.007	4.0	0.143	0.121	0.017	6.5	0.074	0.080	0.006	1.8	0.120	0.100	0.012	3.1
45.0	0.065	0.100	0.006	3.6	0.132	0.120	0.016	6.0	0.068	0.074	0.005	1.5	0.114	0.094	0.011	2.9

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 = 30.0℃
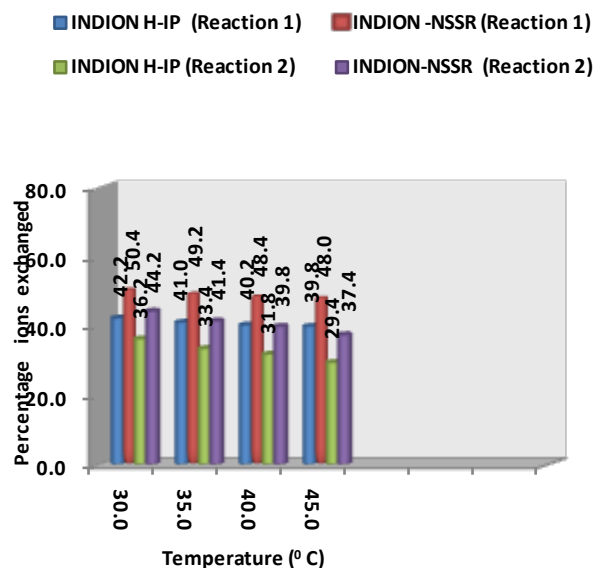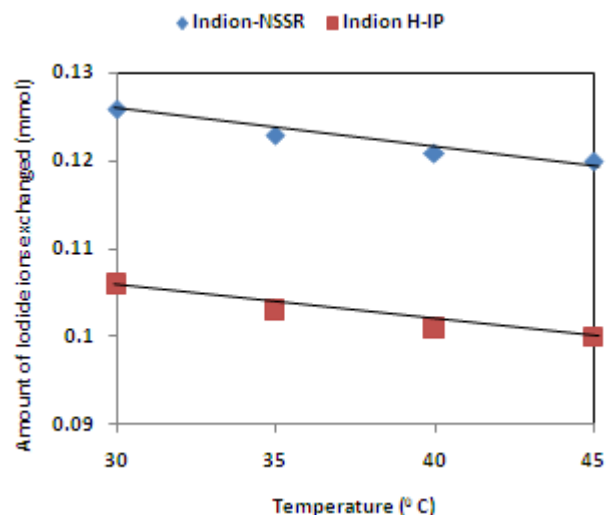
Figure 3. Variation in Percentage Ions Exchanged with Temperature of labeled Ionic Solution Amount of ion exchange resin = 1.000 g, Concentration of labeled exchangeable ionic solution = 0.001M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.250 mmol

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 = 30.0 ℃ Correlation coefficient (r) for Indion H-IP = 0.9998 Correlation coefficient (r) for Indion-NSSR = 0.9999
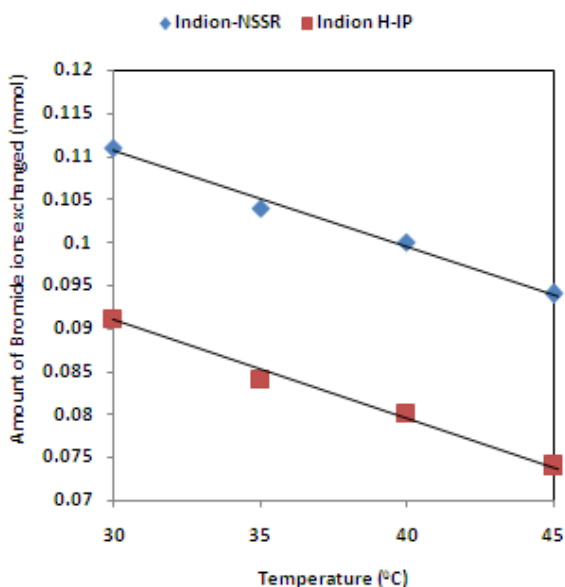

Figure 4. Correlation between concentrations of iodide ion solution and amount of iodide ion exchanged Amount of ion exchange resin = 1.000 g, Volume of labeled ionic solution = 250 mL, Temperature = 30.0 ℃ Correlation coefficient (r) for Indion H-IP =1.0000 Correlation coefficient (r) for Indion-NSSR =1.0000

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 = 0.001M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.250 mmol Correlation coefficient (r) for Indion H-IP = -0.9759 Correlation coefficient (r) for Indion-NSSR = -0.9759

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 = 0.001M, Volume of labeled ionic solution = 250 mL, Amount of exchangeable ions in 250 mL labeled solution = 0.250 mmol Correlation coefficient (r) for Indion H-IP = -0.9951 Correlation coefficient (r) for Indion-NSSR = -0.9951

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[19, 20]. 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[13-18]. 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[21]. 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℃ to 45.0℃, the specific reaction rate was observed to decrease (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

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

Heumann et al.[22] 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.[23] 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.[24]; 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. 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, it is observed that for iodide ion-isotopic exchange reaction by using Indion-NSSR 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 0.162, 0.126, 0.020 and 7.6 respectively, which was higher than 0.091, 0.106, 0.010 and 5.5 respectively as that obtained by using Indion H-IP resins under identical experimental conditions of 30.0^{0}C, 1.000 g of ion exchange resins and 0.001 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 Indion-NSSR resins, at a constant temperature of 30.0 ^{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 50.4 % to 52.2 %. While using Indion H-IP resins under identical experimental conditions the percentage of iodide ions exchanged increases from 42.2 % to 44.2 %. Similarly in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged increases from 44.2 % to 47.6 % using Indion-NSSR resin, while for Indion H-IP resin it increases from 36.2 % to 39.6 %. 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-NSSR resins, for 0.001 M labeled iodide ion solution, as the temperature increases 30.0 ^{0}C to 45.0 ^{0}C, the percentage of iodide ions exchanged decreases from 50.4 % to 48.0 %. While using Indion H-IP resins under identical experimental conditions the percentage of iodide ions exchanged decreases from 42.2 % to 39.8 %. Similarly in case of bromide ion-isotopic exchange reaction, the percentage of bromide ions exchanged decreases from 44.2 % to 37.4 % using Indion-NSSR resin, while for Indion H-IP resin it decreases from 36.2 % to 29.4 %. 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, Indion-NSSR resins shows higher percentage of ions exchanged. Thus Indion-NSSR resins show superior performance than Indion H-IP resins.

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 using Indion-NSSR and Indion H-IP resins, the values of correlation coefficient *(r)* were found to be 1.0000 for both the resins, while for bromide ion-isotopic exchange the values of *r* were calculated as 0.9999 and 0.9998 respectively. There also exist a strong negative co-relationship between amount of ions exchanged and temperature of exchanging medium (Figures 6, 7). For Indion-NSSR and Indion H-IP resins, during iodide ion-isotopic exchange the values of *r* were found to be -0.9759; while for bromide ion-isotopic exchange the values were calculated as -0.9951 for the two 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.

ACKNOWLEDGEMENTS

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