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ORIENTAL JOURNAL OF CHEMISTRY
An International Open Free Access, Peer Reviewed Research Journal

www.orientjchem.org

ISSN: 0970-020 X
CODEN: OJCHEG
2017, Vol. 33, No. (2):
Pg. 1001-1010

Study on Ion Exchange behavior of Nuclear Grade Resin AuchliteARA9366 Chemically Degraded in HydrogenPeroxide Medium

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http://dx.doi.org/10.13005/ojc/330255

(Received: January 03, 2017; Accepted: March 22, 2017)

ABSTRACT

The ion exchange resins are important material for study of various uni-univalent and uni-bivalent ion exchange reactions for the separation, purification and the treatment of industrial effluent's and nuclear waste. In the current investigation attempts were done to evaluate the performance and the selectivity behavior of peroxide degraded resins viz AuchliteARA-9366 towards the Br^- and I^- ions from the external solution. AuchliteARA-9366 resins in chloride form were subjected to Hydrogen peroxide degradation separately on the magnetic stirrer for 24h at room temperature using 20% (6 volume) and 30% (9 volume) H_2O_2. The ion exchange equilibrium study of the peroxide degraded AuchliteARA-9366 resin in chloride form were done separately with KBr and KI solution of concentrations range from 0.01M to 0.1M at the temperature 30.0 °, 35.0 °, 40.0 ° and 45.0 ° C for 3h and the equilibrium constants (*K*) values for Cl^-/I^- and Cl^-/Br^- ion exchange reactions were determined. It was found that during Cl^-/I^- ion exchange reactions, the *K* values of AuchliteARA-9366 resin degraded using 20% H_2O_2 were found to be decreases from 37.28×10^{-2} to 15.61×10^{-2} which was higher than the *K* values observed for the resin degraded using 30% H_2O_2 which decreases from 20.53×10^{-2} to $15.54 \times 10^{-2}$with rise in temperature from 30.0 - 45.0 °C. Similar results were observed during Cl^-/Br^- ion exchange reactions for the resins AuchliteARA-9366.The equilibrium constant (*K)* values were found to be decreases with rise in temperature shows that both Cl^-/I^- and Cl^-/Br^- ion exchange reactions were exothermic in nature and which was further supported by negative enthalpy (ΔH^0) and entropy (ΔS^0) values. The equilibrium constant (K) values of peroxide degraded resins indicates that resins effectively oxidized with increase in the concentration of H_2O_2 from 20% to 30% which were also seen in SEM micrographs and FTIR spectrum of the resin. The results obtained from the present study will be helpful in understanding the effects of hydrogen peroxide degradation on the performance and halide ion selectivity behavior of ion exchange resins AuchlitARA-9366.

Keywords: hydrogen peroxide degradation; AuchliteARA-9366; anion exchanger; nuclear and industrial grade resin; enthalpy.

INTRODUCTION

The AuchliteARA-9366 was a strong base nuclear grade anion exchange resin having cross linked polystyrene matrix. Strongly basic anion exchange resins can be useful for [1]waste water treatment in large number of food ,chemical and bio industry whereas the anion exchange resin with benzyllic ternary ethyl or methyl ammonium groups were chemically unstable and not suitable above 60^0-70^0C temperatures in the hydroxide form[2]. Organic ion exchange resins were more versatile and selective than inorganic ion exchange resins and readily available to fulfil the demands of the various industries[3].The inorganic ion exchangers were more selective towards the separations of specific radioactive ions like Cs^+ and Sr^+ from nuclear waste than the organic exchangers[5].The inorganic exchangers[6]becomes more useful in nuclear industry than conventional organic resins especially in high purity water applications[7].Considering the extensive industrial applications of ion exchange resin, in the present investigation attempts were made to study the performance and selectivity behavior of hydrogen peroxide (20%&30%) degraded resins AuchliteARA-9366[8].The scanning electron microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR)[9]were used to study the surface morphology and to recognizing the degradation pattern of the resin AuchliteARA-9366 in hydrogen peroxide medium. The above resins in chloride form were subjected to peroxide degradation using 20% (6 volume) and 30% (9 volume) H_2O_2 and ion exchange equilibrium studies of the resins were carried out to determine standard free energy (ΔG^0), entropy (ΔS^0), enthalpy change (ΔH^0) and mode of their chemical degradation.

MATERIALS AND METHODS

The anion exchange resin AuchliteARA-9366 in hydroxide form was purchased from Auchtel chemical Products Ltd.,Lower Parel,Mumbai,India. The various physical and chemical properties of the resins AuchliteARA-9366 was given in Table 1

Chemical degradation of resins

The performance of chemically degraded AuchliteARA-9366 resins was studied by exposing it to hydrogen peroxide (20% H_2O_2&30% H_2O_2) medium. In order to bring about hydrogen peroxide (20% H_2O_2&30% H_2O_2) degradation, 25g of the resins in chloride form are transferred separately in 100mL round bottom flask containing 50mL of 20% H_2O_2 and 30% $H_2O_2$and the mixture was stirred on the magnetic stirrer for 24h. The ished chemically degraded resins in chloride form are air dried over P_2O_5 in desiccators and used for further studies.

Conditioning and equilibration of chemically degraded resins in hydrogen peroxide (20% H_2O_2&30% H_2O_2) medium

In order to completely convert the resins AuchliteARA9366 in chloride form, about 80g of resin are placed in glass column 60cm long and 2.5cm in diameter[10]and eluted with 1L of 1M potassium chloride solution at the rate of 2mL/min. the resin is then washed with distilled water and methanol to remove excess of chloride ions and are air dried over P_2O_5in desiccators, stored in stoppard bottle and used for further study[11]. The ion exchange equilibration study of the above 20% H_2O_2&30% H_2O_2 degraded AuchliteARA9366 in chloride form were carried out separately for 3h with KBr and KI solutions[12]of different concentration from 0.01M, 0.025M, 0.05M, 0.075M and 0.10M in the equilibration temperatures between 30.0°C to 45.0°C as explained[13].After 3 hours, the amount bromide and iodide ions[15] exchange with the resin can be determined potentiometrically[14-15] by using standard 0.1M $AgNO_3$.

$$A\text{-}Cl + Br^-_{(aq)} \rightarrow A\text{-}Br + Cl^-_{(aq)} \qquad ...(1)$$

Table1: Physico-chemical properties ofAuchliteARA-9366

Name of Resin	Type of Matrix	Functional Group	Average particle Size(mm)	Percentage of Moisture	Rage of operating temperature(°C)
Auchlite ARA9366	~Polystyrene- ~ divinyl benzene	~R4N+,OH-~	0.3-0.9	40-50	50-60.0

$$A\text{-}Cl + I^-_{(aq)} \rightarrow A\text{-}I + Cl^-_{(aq)} \quad ...(2)$$

Where A=AuchliteARA-9366

From the results the equilibrium constants *K* for the reaction (I) and (II) were calculated in the equilibration temperature ranging from 30.0°C to 45.0°C.

RESULTS AND DISCUSSIONS

The equilibrium constants (*K*) [16] for reactions I and II were calculated by the equation

$$K = \frac{[A\text{-}X][Cl^-]}{[B - A\text{-}X]\,[X^-]} \quad ...(3)$$

Here, A =AuchliteARA-9366 surface; *B* = anion exchange capacity of the resin; X^- = Br^- and I^- ions. Thus by knowing different concentrations of X^- ions in solution at a given temperature, *equilibrium constant* (K) values were calculated and mean *K-values* for this set of experiment was determined. Similar *K* values were calculated for the Cl^-/Br^- and Cl^-/I^- ion exchange reactions carried out at different temperatures. A graph was plotted in between log *K* and 1/T (°K) (figure1-3), from the slope of a graph, the standard enthalpy change ΔH°, ΔG° and ΔS° in kJ.mol^{-1}of Cl^-/Br^- and Cl^-/I^- ion exchange reactions were determined for *K*-values at different temperatures. Similarly ÄH°, ÄG° and ÄS° and equilibrium constant (K), for the above Cl^-/Br^- and Cl^-/I^- ion exchange reactions were also determined for the resin subjected to degradation in hydrogen peroxide (20% H_2O_2&30% H_2O_2) medium. The thermodynamic parameters calculated for Cl^-/Br^- and Cl^-/I^- reactions of fresh resinsAuchliteARA-9366 and resins exposed to degradation in hydrogen peroxide (20% H_2O_2&30% H_2O_2) medium were represented in Tables 2 to 5.

For Cl^-/I^- and Cl^-/Br^- ion reactions[17], the equilibrium constant (*K)* values for AuchliteARA-

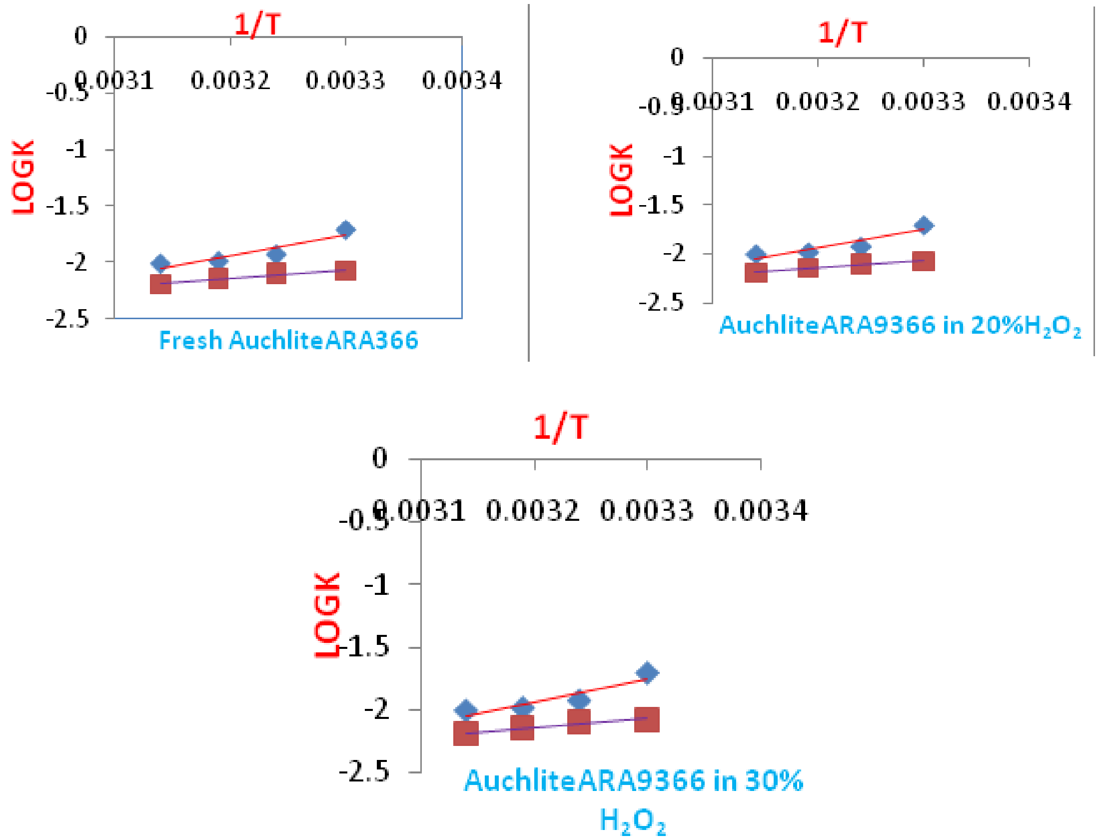

Fig. 1-3: ShowsVariation of equilibrium constant for Cl-/I-&Cl-/Br –reaction.with temperature for fresh, in 20% H2O2, and 30% H2O2 medium for AuchliteARA-9366.

Table 2: Equilibrium constant for the ion exchange Reaction (I) and Reaction (II) using Fresh Resin AuchliteARA-9366. Amount of AuchliteARA-9366 in Cl- form = 500 mg, Anion exchange capacity B= 2.3meq./500mg, Equilibration temperature = 30.0 oC.

AuchiteARA-9366 Fresh Resin ***(before expose to chemical degradation).*** ***$A\text{-}Cl + Br^-_{(aq)} \rightarrow A\text{-}Br + Cl^-_{(aq)}$*** ***Reaction (I)***						***AuchliteARA-9366 Fresh Resin*** ***(before expose to chemical degradation).*** ***$A\text{-}Cl + I^-_{(aq)}$ $A\text{-}I + Cl^-_{(aq)}$*** ***Reaction (II)***					
Initial conc. of Br^- ions (M)	**Final of Conc. Br^- ions (M) [Br^-]**	**Change in Br^- ion conc.**	**Conc. of Cl^- ions exchanged (M) [Cl^-]**	**Amount of interchange Br- ions on the resin meq./ 500mg [A-Br]**	**Equilibrium constant in (K $x10^{-2}$)**	**Initial conc. of I^- ion(M)**	**Final Conc. Of I^- ions (M)[I^-]**	**Change inI^- ion Conc.**	**Conc. Of Cl^- ions interchanged (M)[Cl^-]**	**Amount of iodide ions exchanged on the resin meq./500mg**	**Equilibrium constant in (K $x10^{-2}$) [A-I]**
0.01	0.004	0.006	0.006	0.300	26.47	0.01	0.001	0.009	0.009	0.450	218.93
0.025	0.018	0.007	0.007	0.360	8.79	0.025	0.013	0.012	0.012	0.600	38.50
0.05	0.041	0.009	0.009	0.420	4.79	0.05	0.032	0.018	0.018	0.900	32.14
0.075	0.071	0.004	0.004	0.160	0.34	0.075	0.064	0.011	0.011	0.550	5.96
0.1	0.096	0.004	0.004	0.190	0.37	0.1	0.090	0.010	0.010	0.500	3.31
				MeanK	8.15					Mean K	59.77

Table 3: Equilibrium constant for Reaction (I) and Reaction (II) using AuchliteARA-9366 resin degraded using 20% H2O2.Amount of AuchliteARA-9366 in
Cl^- form = 500 mg, Anion exchange capacity B= 1.804meq./500mg, Equilibration temperature = 30.0 oC.

AuchliteARA-9366 resin degraded in 20% H_2O_2 Reaction (I)						AuchliteARA-9366 resin degraded in 20% H2O2 Reaction (II)					
Initial conc. of Br^- ions (M)	Final Conc. of Br^- ions (M) [Br^-]	Change in Br^- ion conc.	Conc. of Cl- ions exchanged (M) [Cl^-]	Amount of Br^- ions interchange on the resin meq./ 500mg [A-Br]	Equilibrium constant in (K x10^{-2})	Initial conc. of	Final Conc. of I^- ion (M)	Change inI-ion Conc. I^- ions (M)[I^-]	Conc. Of Cl^- ions interchanged (M) [Cl^-]	Amount of iodide ions exchanged on the resin meq./500mg [A-I]	Equilibrium constant in (K x10^{-2})
0.01	0.006	0.004	0.004	0.2	17.81	0.01	0.0028	0.0072	0.0072	0.36	149.5
0.025	0.0172	0.0078	0.0078	0.39	19.20	0.025	0.0168	0.0082	0.0082	0.41	20.43
0.05	0.044	0.006	0.006	0.3	3.38	0.05	0.0406	0.0094	0.0094	0.47	9.91
0.075	0.0694	0.0056	0.0056	0.28	1.59	0.075	0.067	0.008	0.008	0.4	3.67
0.1	0.0944	0.0056	0.0056	0.28	1.21	0.1	0.092	0.008	0.008	0.4	2.77
Mean K	8.64	MeanK	37.27								

Table 4: Equilibrium constant for Reaction (I) and Reaction (II) using AuchliteARA-9366 resin degraded using 30% H2O2.Amount of AuchliteARA-9366 in Cl$^-$ form = 500 mg, Anion exchange capacity B= 1.78meq./500mg, Equilibration temperature = 30.0 oC.

AuchliteARA-9366 resin degraded in 30% H_2O_2 Reaction (I)						AuchliteARA-9366 resin degraded in 30% H_2O_2 Reaction (II)					
Initial conc. OfBr$^-$ ions (M) [Br$^-$]	Final Conc. of Br$^-$ ions (M)	Change in Br$^-$ ion conc.	Conc. of Cl$^-$ ions exch.	Amount of Br- ions interchange on the resin meq./500mg [A-Br]	Equilibrium constant in (K x10^{-2})	Initial conc. of I$^-$ ion(M)	Final Conc. Of I$^-$ ions (M) [I$^-$]	Change in I$^-$ ion Conc.	Conc. Of Cl$^-$ ions interchanged (M) [Cl$^-$]	Amount of iodide ions exchanged on the resin meq./500mg [A-I]	Equilibrium constant in (K x10^{-2})
0.01	0.0056	0.0044	0.0044	0.22	17.23	0.01	0.0046	0.0054	0.0054	0.27	68.96
0.025	0.019	0.006	0.006	0.3	8.21	0.025	0.017	0.008	0.008	0.4	19.65
0.05	0.0454	0.0046	0.0046	0.23	2.40	0.05	0.0412	0.0088	0.0088	0.44	8.75
0.075	0.0704	0.0046	0.0046	0.23	1.04	0.075	0.0678	0.0072	0.0072	0.36	2.90
0.1	0.0954	0.0046	0.0046	0.23	0.79	0.1	0.0926	0.0074	0.0074	0.37	2.35
Mean K	5.94	MeanK	20.52							Average K	

Table 5: Thermodynamics of Cl-/Br-and Cl-/I-reactions using fresh AuchliteARA-9366 and degraded resin in 20% and 30% H2O2medium.

Resin.A-Cl + $Br^-_{(aq)}$ → A-Br + $Cl^-_{(aq)}$ A-Cl + $I^-_{(aq)}$ A-I + $Cl^-_{(aq)}$			(I) (II)				ΔH° (KJ/mol)	ΔG° (KJ/mol)	ΔS° (KJ/mol)
Equilibrium Temperature (0C)			30	35	40	45	---	---	---
Fresh AuchliteARA-9366	Equilibrium Constant	Reaction (I)	8.15	6.6	6.46	6.06	-14.77	6.95	-0.07
	In K x10^{-2}	Reaction (II)	59.77	33.56	27.2	23.77	-47.87	2.82	-0.16
AuchliteARA-9366. Degraded In 20% H_2O_2.	Equilibrium Constant	Reaction (I)	8.64	8.32	5.84	4.97	-32.11	6.96	-0.124
	In K x10^{-2}	Reaction (II)	37.27	22.87	21.89	15.61	-42.57	3.78	-0.148
AuchliteARA-9366.Degraded In 30% H_2O_2.	Equilibrium Constant	Reaction (I)	5.94	5.71	4.95	4.74	-13.05	7.57	-0.065
	In K x10^{-2}	Reaction (II)	20.52	17.77	16.66	15.63	-14.14	4.49	-0.059

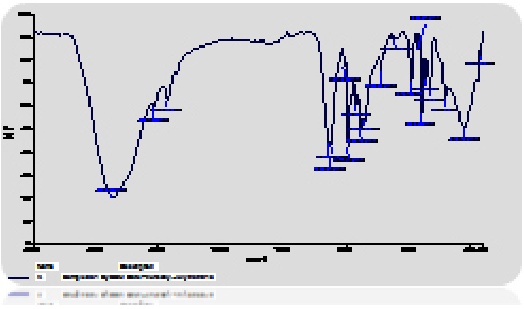

Fig. 4

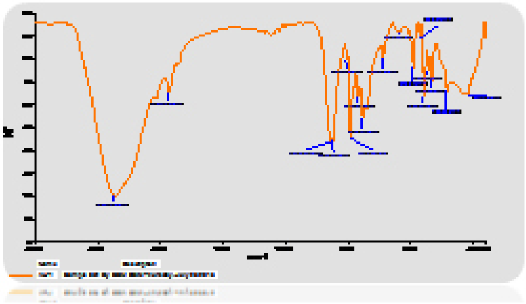

Fig. 5

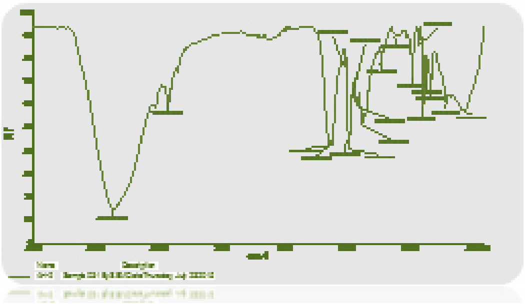

Fig. 6

Fig. 4-6.FTIR Spectrum of freshresin,chemically degraded in 20% and 30% H2O2 medium AuchliteARA-9366.

9366 resins degraded in 20% H_2O_2 medium were found to decreases from 37.28x10^{-2} to 15.61x 10^{-2} and from 8.64 x10^{-2} to 4.97 x10^{-2} respectively with rise in equilibration temperature from 30.0 °C to 45.0 °C. The standard enthalpy change (∆H°), (∆G°) and (∆S°) calculated for Cl^-/I^- ion exchange reactions were -42.58, 3.78 and -0.15 kJ.$K^{-1}mol^{-1}$ respectively which were less than the respective values of -32.12,6.96 and -0.12 kJ.$K^{-1}mol^{-1}$ respectively as that obtained for Cl^-/Br^-ion exchange reactions (Table 5).similarly *(K)* values for Cl^-/I^- and Cl^-/Br^- ion exchange reactions for AuchliteARA-9366 resins degraded in 30% H_2O_2 medium were found to decreases from 20.53x10^{-2} to 15.64x10^{-2} and from 5.94x10^{-2} to 4.74x10^{-2} respectively with rise in equilibration temperature from 30.0 °C to 45.0 °C. The standard enthalpy change (∆H°) ,(∆G°) calculated for Cl^-/I^- ion exchange reactions were -14.14 and 4.50 kJ.mol^{-1}respectively which were lower than the respective values of -13.05 and 7.58 in kJ.mol^{-1}respectively as that obtained for Cl^-/Br^- reactions. However, slightly large value of (∆S°) -0.06 kJ$mol^{-1}$is obtained for Cl^-/I^- ion exchange as compared to -0.07 kJ.mol^{-1} obtained for Cl^-/Br^- ion exchange reactions (Table 5). The large *K* values and standard enthalpy change (∆H°), (∆G°) and (∆S°) values [as given in Tables 5] obtained for Cl^-/I^-ion exchange reactions as compared to the Cl^-/Br^- ion exchange reaction for fresh and peroxide degraded AuchliteARA-9366 in 20% H_2O_2 and 30% H_2O_2 medium shows that iodide ions in the solution were more selective towards the resins AuchliteARA-9366 as compared to that of bromide ions. Similarly for Cl^-/I^- and Cl^-/Br^- reactions of chemically degraded resin AuchliteARA-9366, the K-values obtained were nearly half of equilibrium constants (K) values obtained for Cl^-/I^- and Cl^-/Br^- reactions of fresh resins AuchliteARA-9366 with rise in the concentration of hydrogen peroxide from 20% to 30%. these equilibrium constants (K) values for AuchliteARA-9366 resins decreases with increase in the concentration of hydrogen peroxide indicating that the resin oxidized effectively in peroxide medium which were clearly seen in FTIR spectrum and SEM Micrograph[18]of the resins under similar conditions.

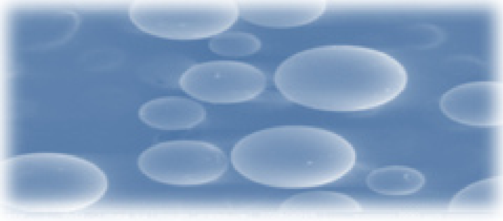

Fig. 7:SEM image offresh resinAuchliteARA9366 in 20% H2O2

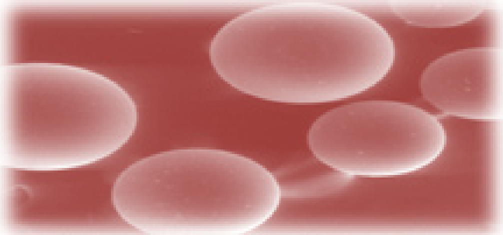

Fig. 8: SEM of chemically degraded

Fig. 9: SEM image of AuchliteARA-9366 resin chemically degraded using 30% H2O2

FTIR spectrum of fresh and chemically degraded AuchliteARA-9366 resin using hydrogen peroxide

FTIR spectra of fresh as well as Hydrogen peroxide degraded samples of AuchliteARA-9366 resins were recorded in KBr pellets (0.002 g resin/ 0.200 g KBr) were using a PerkinElmer 1750 FTIR spectrophotometer. In the FTIR spectrum of fresh resin[19] AuchliteARA-9366, the sharp strong broad band at 3366 cm^{-1} corresponding to the signal vibration of O-H bond of the water or the quaternary ammonium group (R_4-N^+). This may be due to the moisture content of the fresh resins. The sharp band between1380-1349 $cm^{-1}$is for -C-N stretching while a variable absorption bands between 1633-1614cm^{-1} is due to the stretching vibrations of –C=C- of alkenes group. The weak band at 3031cm^{-1} is the characteristic stretching band for aromatic ring. A moderate band at 2925cm^{-1} is due to the C-H stretching band for $–CH_2$ group. A moderate and sharp band at 1416cm^{-1} and 1470cm^{-1} is due to the –C-H bending bands for - CH_2 group. The variable band at1511cm^{-1} is the –C=C- stretching for aromatic ring, the sharp band at 828cm^{-1} and moderate band at 705cm^{-1} is the characteristic bands of p-substituted and o-substituted aromatic rings . On Comparing the IR spectra of fresh resin (figure 4) with the IR spectra's of degraded resin in 20% and 30% peroxide medium (figure 5and 6) indicate the disappearance of characteristic –C-N stretching[19]in quaternary ammonium group band at 1349cm^{-1} and the characteristic Aromatic -C-H stretching band at 3031cm^{-1}, it can be conclude that the resin is slightly oxidized in the peroxide medium and there is no significant change in structure of the resin. However, some of the resin sites are blocked or cracked for the ion exchange reaction with rise in the concentration peroxide degradation medium from 20% to 30% which can also be seen in the SEM images(figures 8- 9) of peroxide degraded resins.

Scanning electron micrographs of both fresh and degraded resin surfaces were obtained with the help of SEM technique using Scanning Electron Microscope of model no. JSM-6380LA (Jeol Ltd., Japan). The resin samples were properly placed on the aluminum sub coated double-sided graphite tape and simultaneously samples were made exposed electronically in a vacuum on thin carbon layer for 60 seconds at 30 W. The images were captured at an excitation voltage of 15 kV[20]under a 90 Pascal pressure and a magnification of x150, x500, x1000, x 2500 and ×5000.The SEM (Figure 7) of fresh ion exchange resins AuchliteARA-9366 shows its plane spherical structure with smooth surface. The scanning electron micrograph of chemically degraded AuchliteARA-9366 resin in 20%H_2O_2medium shows large hair line cracks and thread like rough appearance on the plane surface of the resin indicates the resin may be mildly oxidized in the peroxide medium (Figure 8) while SEM image of chemically degraded AuchliteARA-9366 resin in 30%H_2O_2medium shows heavy cracks and dots on the plane spherical surface of the resin indicates that the resin may be oxidized in the peroxide medium (Figure 9) and the surface of the resin is appearing still rough in the peroxide medium as compare to that of fresh resin (Figure 7).

CONCLUSION

The present research work will be helpful for understanding the degradation effect of various oxidizing and reducing agents on the performance and selectivity behavior of different industrial and nuclear grade resin materials for treatment of waste water effluents. The results and data obtained from such research work will be helpful in selecting the specific ion exchange resins for efficient industrial applications and will be useful in optimization of process parameters to achieve the maximal efficiency of the ion exchange material.

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