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Deionization of Coagulated, Clarified, Turbid *Gauri Shankar* Lake Waters by Using Ion-Exchange Technology

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Abstract: Water collected from a rain fed Gauri Shankar Lake, Bhavnagar, Gujarat, India was used for this study. It was clarified by giving treatment with alum and poly aluminum chloride (PAC) to remove suspended impurities present in it. Waters having turbidity value of 100–750 NTU (Nephelometric Turbidity Units) were treated with PAC and alum. The dosage of PAC required for the treatment was 60% less as compared with alum. The clarified water was passed through columns loaded with strong acid and strong base ion-exchange resins to obtain deionized water. The experiments were carried out for 100 cycles of exhaustion and regeneration. The effect of the coagulation process conditions on ion-exchange capacity, and the physical attrition of the ion-exchange resins were studied intensively. Also, the performance of ion-exchange resins was compared with virgin ground waters from the institute's well. The resins employed in the study were subjected to EDX (Energy Dispersive X-Ray Spectroscopy) analysis to find out the presence of coagulating ions adsorbed on the resins. The resins were also subjected to SEM (Scanning Electron Microscope) analysis to find changes on their surface due to adhering materials if any.

Keywords: Alum; Coagulation; Demineralization; EDX; Poly aluminum chloride; Resin; SEM.

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INTRODUCTION

Lakes, ponds, and streams are the important source of water for many farms, rural camps, households and so on. These water sources contain much more suspended impurities than the ground water and this water is required to be clarified using coagulants. Coagulants commonly used are calcium carbonate (lime), calcium sulfate (gypsum), and alum. Natural materials such as starch and its derivatives, cellulosic compounds, polysaccharide gums, and proteinaceous materials were formerly used as flocculants for removing suspended impurities from water.

The most common coagulant used in water treatment is alum-$Al_2(SO_4)_3 \cdot 18H_2O$ because of its effectiveness in treating a wide range of water types and relatively at low cost. The use of preformed polymerized forms of Aluminum have become more common as alternative coagulants, with polyaluminum chloride (PAC) and polyaluminum sulfate (PAS) are most widely used to treat drinking water (1)/surface water (2) containing the polymeric species- $Al_{13}{}^{7+}([AlO_4Al_{12}(OH)_{24}(H_2O)_{12}]^{7+})$. These polymerized coagulants have the advantage of being more effective over a broader pH range and perform even at lower temperatures than alum (3,4). The coagulant doses required are also less because of the higher charge density of PAC species. Polyaluminum chloride solution contains stable aluminum species, such as "Al_{13}", which are stable and more effective for charge neutralization purposes than the respective monomeric species. PAC also produces generally lower volumes of residual solids (sludge) (3). Polyaluminum chloride coagulants typically demonstrate reduced coagulant demand resulting in chemical saving and longer filter runs. High basicity PACs have been shown to produce decreased head-loss accumulation rates, improved filter effluent turbidity, and improved natural organic matter (NOM) removal for direct filtration applications as compared to alum. Poly-aluminum chloride (PAC) is one of the most widely used inorganic flocculants throughout the world and has proven to be an effective flocculant for the removal of suspended water contaminants, turbidity, and color (5).

It is revealed in literature that when polyaluminum hydroxyl chlorosulfate coagulants are used, the floc formation is rapid which is critical in any water treatment (6) plant with short detention time. Polyaluminum chloride is added to cause small particles to clump together (called "floc") thereby making them heavy enough to settle into a basin from which the accumulated and sediment is removed. The water is then filtered through anthracite coal and sand filter beds to remove remaining particles. As these smaller suspended particles are removed, the turbidity of the water is greatly reduced and clear water is produced. Pre-polymerized inorganic coagulants, such as polyaluminum chloride

(PAC) have several advantages over conventional Al and Fe salts, however, their mode of action is not fully understood and progress in this area has been made largely by empirical approaches (7). Study has been made (8) to find the effect of left over coagulants if any on ion-exchange resins. These authors have studied on the flocculating agents like ferric alum, non ferric alum, and polyaluminum chloride (PAC) and their effect on ion-exchange capacities of resins (INDION). The comparison of data reveals that the use of PAC in the pre-treatment of raw water causes lesser decrement to the total ion-exchange capacities and salt splitting capacities of resins and can be used as a coagulating agent in pre-treatment of water.

MATERIALS

Chemicals

Strongly acidic, strongly basic ion-exchange resins, and polyaluminum chloride (KANPAC-10) were supplied by Kanoria Chemical Industries Ltd. (KCIL). Alum was procured from Merck India, and the hydrochloric acid and sodium hydroxide used was of commercial grade.

Water Sources

Lake water: Water from Gaurishanker lake, Bhavnagar was taken for the study.
Well water: Obtained from CSMCRI's well

Typical Analysis of Lake Water

Total dissolved solids .... 521 mg/L
*Phenolphthalene alkalinity .. 0.0 mg/L
*Methyl orange alkalinity .... 160.1 mg/L
*Hardness .... 224.0 mg/L
*Chloride .... 71.0 mg/L
*Sulphate .... 50.3 mg/L
pH 7.5
*Expressed as $CaCO_3$

EXPERIMENTAL

Water required for the study was brought from Gauri Shankar lake throughout the period of investigation which took nearly a year to complete. The water had high turbidity during the rainy season whereas it was less turbid in winter and summer. The turbidity varied from (100 to 750 NTU). The well known Jar test technique was used for testing PAC and Alum (9).

Test Conditions

1000 ml of water samples were taken in glass beakers for flocculation. In each case after addition of alum and polyaluminum chloride (KANPAC-10), rapid agitation was maintained at 100 ± 10 rpm for 30 sec., followed by slow agitation at $40 + 5$ rpm for 10 min. At the end of slow agitation, the turbidity of water was measured after 20 min.

Dosing of the coagulants was done:

i) by preparing 1% solution in distilled water and then adding with the help of graduated pipette, and
ii) Neat (as received), with the help of micro syringe (for KANPAC-10).

It was observed that when the dosing was done with 1% solution of KANPAC-10 (PAC from KCIL) the treated water showed slight haziness, whereas when the dosing was done with neat PAC, the treated water was clear and free from any haziness. It was estimated that the treated waters after coagulation step contained 0.162 and 0.170 ppm aluminum respectively for waters treated with PAC and Alum.

The results of the jar test carried out in the laboratory are given in Table 1. For testing the resins, 200 liters of lake water was treated separately every time with alum or polyaluminum chloride and decanted and filtered before passing through the resin bed.

Preparation of Ion-Exchange Resin Beds in Columns

Strong acid and strong base resins received from KCIL were conditioned and sieved. A fraction between -18 and $+52$ BSS (British Standard Sieves) was used for this particular study. Glass columns having 2.00 cm internal diameter and a stop-cock and glass wool plug at the bottom and B-29 joint at the top and 1.5 meters length was set up. 60 cm of this column was filled with regenerated and sieved cation-exchange resin and 90 cm of anion-exchange resin. Cation exchanger is taken less in volume than anion exchanger as the cation-exchange capacity of cation exchanger is 2.0 equivalents per liter whereas anion-exchange capacity that of anion exchanger is 1.4 equivalents

Table 1. Comparitive jar test results of KANPAC-10 (PAC) vis-á-vis alum

ALUM			KANPAC-10		
Dose ppm	Turbidity NTU	Final pH	Dose ppm	Turbidity NTU	Final pH
	Turbidity: 750 NTU			pH: 7.2	
20	170	6.9	30	56.0	7.2
30	150	6.8	40	32.0	7.2
50	100	6.8	45	3.4	7.2
60	89	6.7	50	6.2	7.2
70	40	6.8	55	10.0	7.1
80	25	6.6	–	–	–
100	20	6.5	–	–	–
110	5	6.4	–	–	–
120	4	6.4	–	–	–

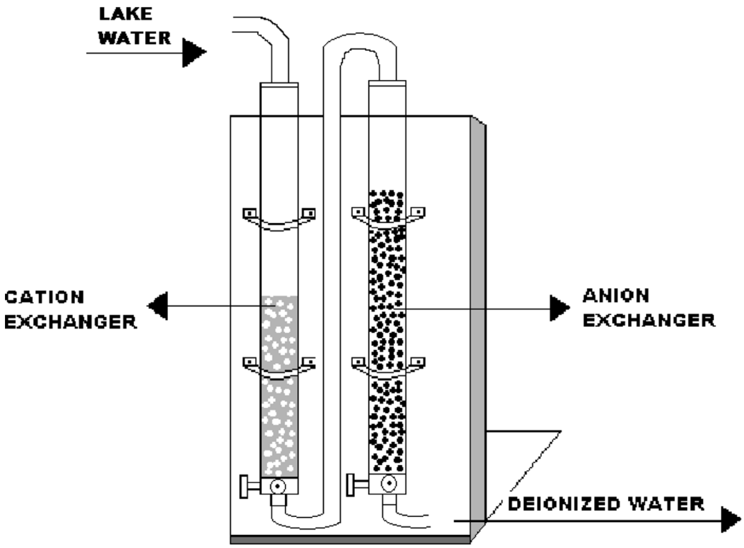
Source: Raw Water-Gauri Shankar Lake, Bhavnagar, Gujarat, India.
Jar Test Conditions.
Flash Mixing: 30 sec., Slow Mixing: 10 min, Setting: 20 min80.

per liter. In order to balance the ionic capacity of both resins the anion exchanger was roughly taken 1.5 times more than the cation exchanger.

Performance of Ion-Exchange Columns

Clarified waters from the polyelectrolyte treatment (alum and poly aluminum chloride separately) were percolated down the column at service flow rate arbitrarily fixed at 10 bed volumes per hour. The eluted fractions were collected and analyzed for the ions present in every fraction. The process was discontinued when the resin bed got saturated, after which the resins were regenerated with 1 N hydrochloric acid and 1 N sodium hydroxide with in between washing using demineralized water. This process was continued for ten cycles of exhaustion and regeneration. At the end of ten cycles the resins were once again regenerated/conditioned and sieved. The resins were evaluated for their ion-exchange capacities and attritional losses each time after such regeneration steps.

The resins used were again subjected to next ten cycles of exhaustion-regeneration and evaluated for ion-exchange capacity and sieve analysis. This process was repeated after every ten cycles till 100 cycles



Scheme 1. Schematic Illustration of the Column. (http://www.lenntech.com/deionised-demineralised-water.htm).

of exhaustion and regeneration were complete. A blank run of 100 such cycles was carried out with virgin well water of the institute for both resins. The results are given in Figs. 1–4. *Schematic Illustration of the Column.*

EDX Analysis of the Used Resins

The resins used in this study were subjected to EDX analysis to determine any deposition of aluminum ions on the resins. The result obtained showed that there was no adsorption of aluminum ions on the resin matrix. The analysis results are shown in the Figs. 5–8.

$$R\sim SO_3^-H^+ + XY \longrightarrow R\sim SO_3^-X^+ + HY$$

$$R\sim NR_3^+OH^- + HY \longrightarrow R\sim NR_3^+Y^- + H_2O$$

X=cations
Y=anions

Scheme 2. Reaction scheme shown here (13).

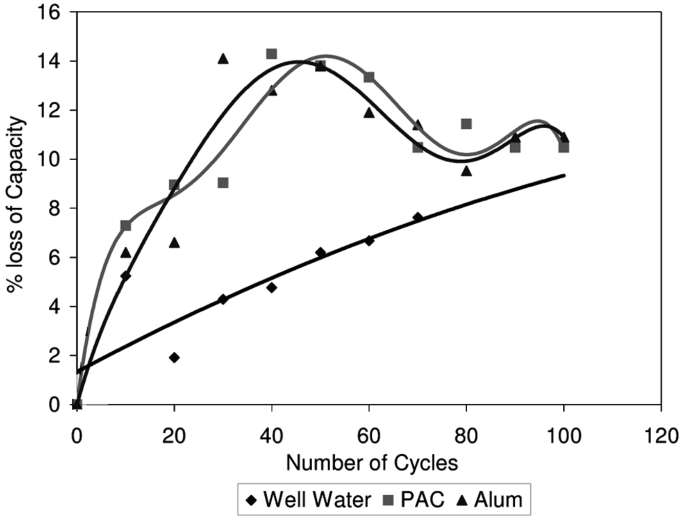


Figure 1. Comparative losses in capacity of strong acid cation exchanger while under going exhaustion and regeneration cycles by waters clarified with PAC, Alum and virgin well waters.

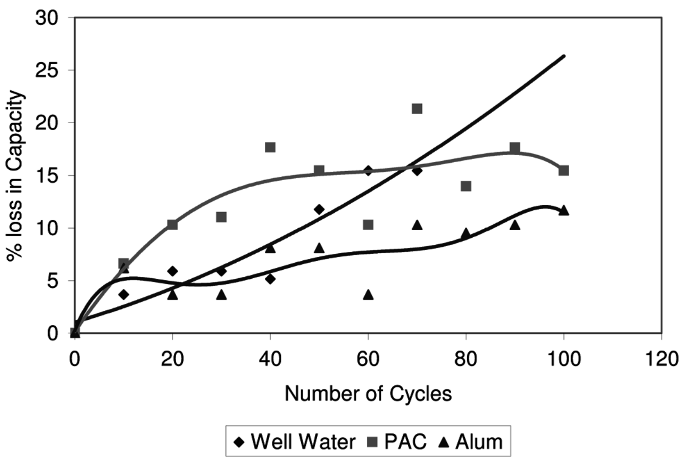


Figure 2. Comparative losses in capacity of strong base anion exchanger while under going exhaustion and regeneration cycles by waters clarified with PAC, Alum and virgin well waters.

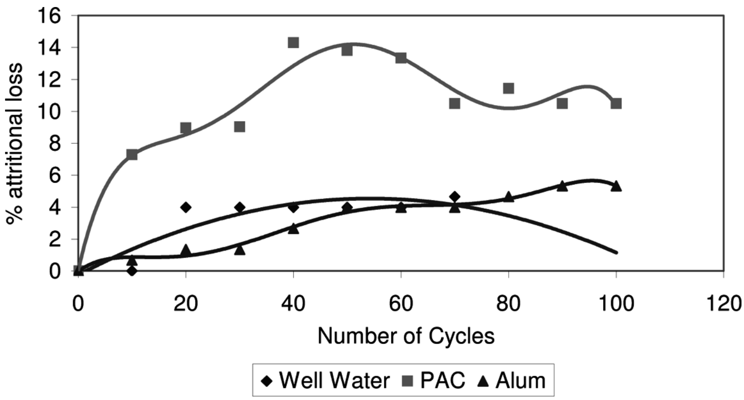


Figure 3. Comparative attritional losses in strong acid cation exchanger while under going exhaustion and regeneration cycles by waters clarified with PAC, Alum and virgin well waters.

SEM Images of the Used Resins

The resins which were used for hundred cycles each for de-ionizing well waters and lake waters treated with alum and PAC to get rid of suspended impurities were subjected to SEM analysis using LEO

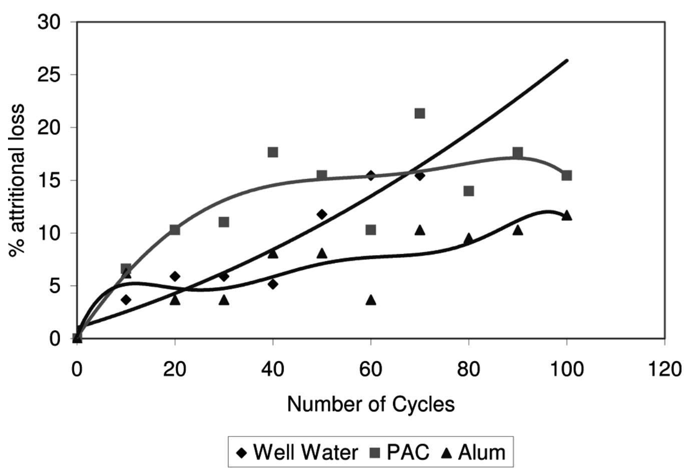


Figure 4. Comparative attritional losses in strong base anion exchanger while under going exhaustion and regeneration cycles by waters clarified with PAC, Alum and virgin well waters.

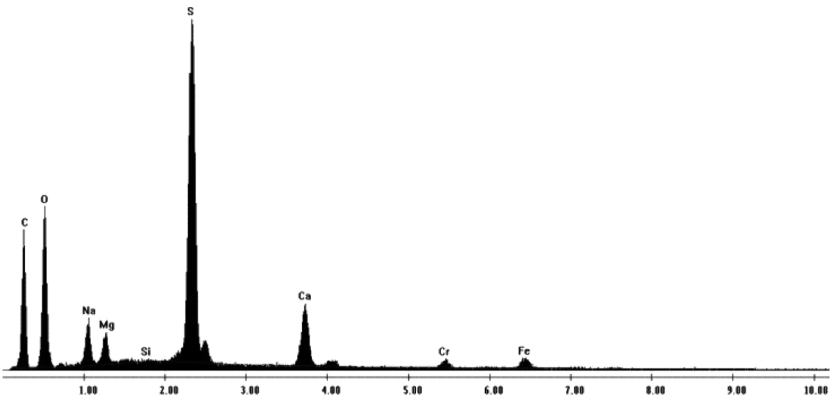


Figure 5. EDX report of cation Exchanger Resin used for removing cations from Alum treated waters.

1430VP instrument model was used for taking the micrographs. SEM micrographs are shown in Figs. 9–10.

RESULTS AND DISCUSSION

It is evident from the jar test study that 100–120 ppm alum is required to get 5 NTU turbidity level whereas only 40 ppm polyaluminum chloride is

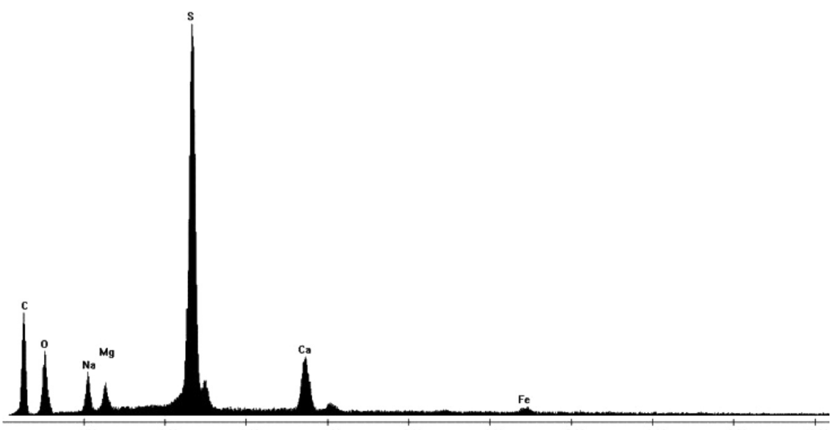


Figure 6. EDX report of cation Exchanger Resin used for removing cations from PAC treated waters.

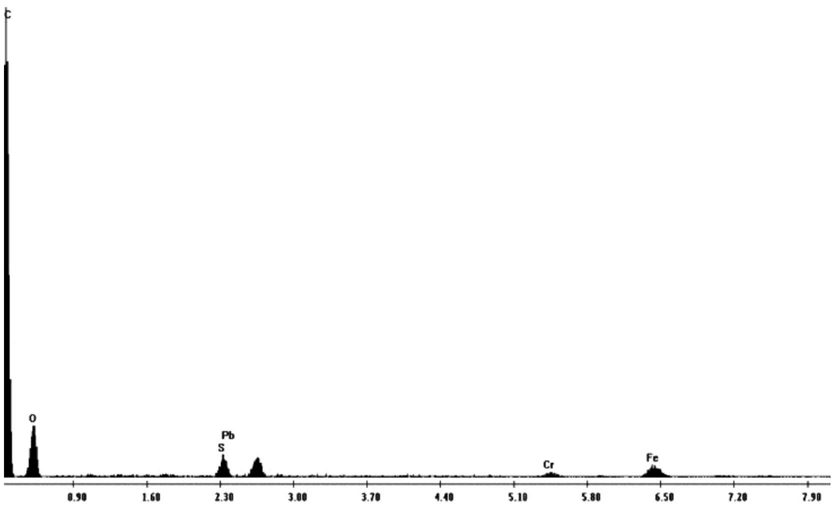


Figure 7. EDX report of anion Exchanger Resin used for removing anions from Alum treated waters.

needed to get the same result. These results indicate that there can be 60% saving in the use of coagulant when waters are treated with polyaluminum chloride (10). This proves that polyaluminum chloride (11) coagulants are superior to alum as they show higher removal of suspended solids and natural organic matter, form larger flocks, and have higher flocculation rates and lower residual aluminum concentration, which is a very important factor especially for the

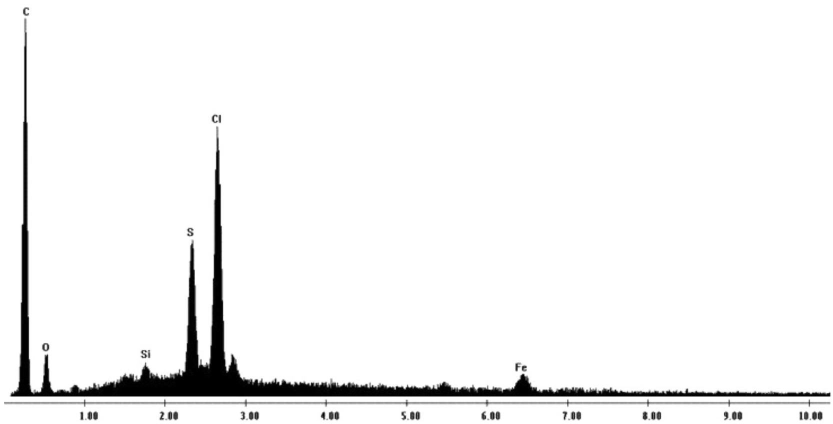


Figure 8. EDX report of anion Exchanger Resin used for removing anions from PAC treated waters.

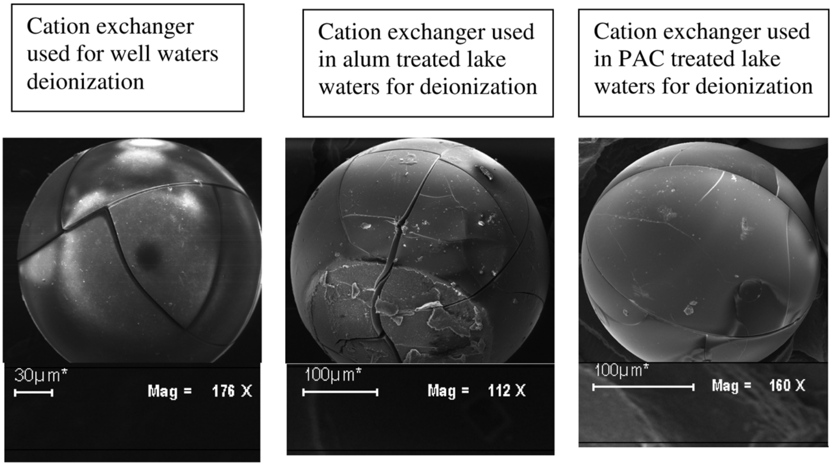


Figure 9. SEM micrographs of the cation-exchangers used in the study.

treatment of surface water meant for potable use (12). Waters treated with alum are acidic in nature whereas they are just neutral in case of poly-aluminum chloride.

Reaction Scheme Shown Here

Strong acid cation exchanger showed 10.5, 10.9, and 9.0 percent loss in capacity and 10.7, 5.3, and 5.5 percent attritional losses after 100 cycles of exhaustion and regeneration with waters clarified with PAC, alum

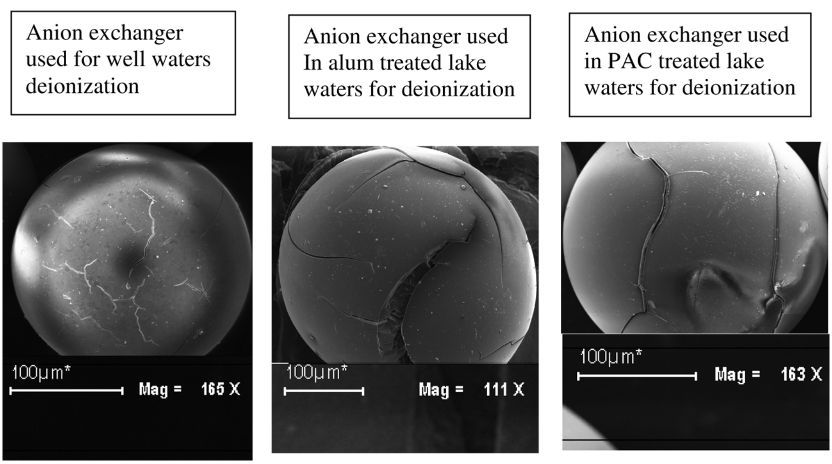


Figure 10. SEM micrographs of the anion-exchangers used in the study.

and well waters respectively (Figs. 1,3). Strong base anion exchanger showed 15.5, 11.7, and 18.4 percent loss in capacity and 12, 10.6, and 8.0 percent attritional losses after 100 cycles of exhaustion and regeneration with waters clarified with PAC, alum and well waters respectively (Figs. 2,4). Literature is full of references (7) wherein researchers have found similar results; in fact 5% loss in capacity and attritional losses are quite common. In a chloro-alkali industry using huge quantity of DM water, resin columns are regenerated once in every week (2), thus in this study, two years of resin's life has been used up.

The EDX analysis of the used resin samples (Figs. 5–8) show no peaks for aluminum thereby indicating that the resins used for the study are unaffected by left over aluminum after coagulation step.

The SEM images in Fig. 9,10 reveal that on cation and anion exchangers there is no noteworthy deposition of ions from well waters but there appears to be some sort of haziness may be due to deposition of left over coagulant on resins which were used for deionization in case of waters treated with PAC and it is still more in waters treated with alum.

CONCLUSIONS

Waters having turbidity value of 750 NTU can be treated with 60% less coagulant doze when PAC is used in place of alum. The resin properties remain unaffected during demineralization process up to 100 cycles of exhaustion and regeneration. The resin is not poisoned by left over aluminum ions as evidenced by EDX and to some extent by SEM images.

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