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Removal of Indium Turbidity from Water Using a Magnesium Ion Loaded Weak Acid Cation Exchanger and Fibrous Graphite Electrode

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ABSTRACT

Indium turbidity generated due to some corrosion of indium metal used for sealing over pressure rupture device (OPRD) of a RAPS-I moderator heavy water system was found to create a high radiation exposure problem due to activation of ^{115}In to In^{116m} ($t_{1/2} = 54\,\text{min}$, $E_\gamma => 1\,\text{MeV}$). The removal of indium turbidity, due to the small size, weak charge, and low concentration [(ln) $\approx 100-150\,\text{ppb}$] of the suspended particles, was not possible by conventional filtration or by normal ion exchange purification systems. Indium turbidity was generated and its removal was demonstrated on magnesium hydroxide precipitated weak acid cation exchanger resin bed with 97% efficiency. A fibrous graphite electrochemical cell was also tested in the laboratory for indium turbidity

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removal by electrosorption and was used to remove satisfactorily indium turbidity from a RAPS moderator system on an experimental basis. The results of these studies are presented.

INTRODUCTION

It is very difficult to remove metal ion based colloidal turbidity from cooling water systems using normal filters, which can remove particles of greater than a few micron size (2 to 5 μm). In the context of the operation of nuclear power stations, the generation of such turbidity containing an element that is activated by neutrons can create a radiation exposure problem to the operation and maintenance staff. For example, a leaky calendria over pressure rupture device (OPRD) of a moderator system of Rajasthan Atomic Power Station (RAPS-I) was sealed using indium (In) metal in March 1998. Under normal operating conditions, the moderator does not come in contact with this indium sealant. However, the condensing moisture on this sealing surface containing some radiolytically produced HNO_3 caused corrosion of this indium metal and introduced indium in the moderator heavy water. In the neutral pH regime of a moderator, the indium seemed to have formed colloidal indium turbidity. The ^{115}In (95.7% isotopic abundance) got converted to radioactive In^{116m} ($t_{1/2} = 54$ min, $E_\gamma => 1$ MeV) due to its fairly high neutron absorption cross section (170 barns). This created operational problems while handling moderator heavy water samples for analysis of chemistry parameters. Hence, it was necessary to remove the indium turbidity from the moderator system. As the size of turbidity particles was very small ($\ll 1$ μm) and indium concentration was very low (100 to 150 ppb), it could not be removed by normal filtration or by the system purification ion exchangers. Two approaches were studied to remove this indium turbidity. A $Mg(OH)_2$ precipitated weak acid cation exchanger resin in Mg form, which was earlier demonstrated by us in laboratory and in the actual reactor system for removal of aluminum turbidity,[1] was tried and found to satisfactorily remove this indium turbidity. Removal of indium turbidity was also attempted by electrosorption on fibrous graphite.

EXPERIMENTAL

Preparation of Indium Turbidity

Indium turbidity for laboratory investigations was generated by stirring finely chipped acid-washed (with 1:1 HCl) indium metal with distilled water at

70 to 80°C using a magnetic stirrer for several hours. The generated turbidity was filtered through Whatman 540 filter paper and the filtrate was used for further experiments. Turbidity of the filtrate was measured, using ELICO nephelometer model CL-52. The indium nephelometer measures the reflected light intensity at right angles to the incident light beam (Tyndall effect) and, hence, yields concentrations of precipitate in suspension (turbidity) in nephelometric turbidity units (NTU) relative to standard suspensions used for its calibration. A 400 NTU standard turbid solution was prepared by mixing 5 mL of a 1% solution of hydrazine sulfate [$(NH_2)_2{\cdot}H_2SO_4$] with 5 mL of a 10% solution of hexamethylene tetramine [$(CH_2)_6N_4$], allowing it to stand for 24 h at 300 K, and then diluting it to 100 mL full volume. Using this stock turbid solution, calibration standard in the range of 4 to 40 NTU were prepared.

The indium concentration was measured by dissolving the turbidity in concentrated HNO_3 (5 mL HNO_3 + 20 mL turbid solution) and then analyzing it by square-wave voltammetry using an ECO-Chemie electrochemical system. Thus, the indium remaining in suspension was measured in terms of both turbidity concentration in NTU units and corresponding metal ion concentration. Particle size was measured by using BIC-BI-90 particle sizer and zeta potential was measured by using Malvern Zeta meter model 3000.

Preparation of $(RCOO)_2{\cdot}Mg{\cdot}Mg(OH)_2$ Precipitated Ion Exchanger

The $(RCOO)_2{\cdot}Mg{\cdot}Mg(OH)_2$ precipitated ion exchanger was prepared as per our earlier studies with this resin for the removal of aluminum turbidity.[1]

The procedure for this preparation can briefly be summarized as follows:

1. A polyacrylic copolymer based carboxylic acid resin (exchange capacity 9.5 Meq/g on dry (basis) as the starting material was employed. The following scheme was used to precipitate $Mg(OH)_2$ on the ion-exchanger:
    - $2RCOOH + MgCl_2 \rightarrow (RCOO)_2Mg + HCl$
    - The resin was freed from HCl by washing with distilled water.
    - $(RCOO)_2Mg + 2KOH \rightarrow 2RCOOK{\cdot}Mg(OH)_2$
    - $2RCOOK{\cdot}Mg(OH)_2 + MgCl_2 \rightarrow (RCOO)_2Mg{\cdot}Mg(OH)_2 + 2KCl$
2. The resin was washed free of KCl.
3. The $Mg(OH)_2$ precipitated weak acid resin represented by $(RCOO)_2Mg{\cdot}Mg(OH)_2$ was employed in the indium turbidity removal.

A resin column was prepared with 5 mL of this precipitated ion exchange resin sandwiched between 10 mL MB (mixed bed = 1:1 volume wise strong base anion and strong acid cation exchangers), at bottom, and 3 mL at the top, with a glass wool plug at the bottom. The diameter of the column was 10 mm. Solutions of varying turbidity were passed through the column. Inlet and outlet of the column was measured for turbidity, pH, and indium concentration. All reagents used were AR grade and distilled water was used for all the experiments.

Fabrication of an Electrochemical Filter

The basic design of the electrochemical filter in this study is based on an earlier work regarding removal of aluminum turbidity from D_2O in a nuclear reactor.[2] The filter fabricated in this study had 14 cathode anode pairs of fibrous graphite felt disks (each felt of ~ 6 mm thickness and of 35 mm dia). The felt disks were separated and supported by 2-mm thick perforated polyethylene plates. A central SS rod connecting a set of 14 such felts and a outer SS cylinder connecting another set of 14 felts served as the electrode leads for impressing the potential to make the two felt sets as anodes and cathodes. The filter could be operated at a flow rate of 23 mL/min, so as to give a contact time of 2 min.

RESULTS AND DISCUSSION

The particle size of the turbidity generated in the laboratory was found to be 230 to 250 nm. By measuring zeta potential at a constant ionic strength (10 mM) and varying pH (Fig. 1), the point of zero charge (pzc) of turbidity particles was measured to be 2.3. X-ray diffraction (XRD) pattern (Fig. 2) of the residue obtained after evaporation of the filtered turbid solution indicated the composition of turbidity particles to be $In(OH)_3$ and In_2O_3.

$(RCOO)_2 \cdot Mg \cdot Mg(OH)_2$ Resin Method

Results of the column run are shown in Fig. 3. As described earlier, the $(RCOO)_2 \cdot Mg \cdot Mg(OH)_2$ resin was sandwiched between two mixed beds (MB). Top MB was to prevent flow disturbance to MAGWAC and bottom MB was to take care of magnesium slip. Flow rate was kept at 5 to 6 mL/min. Because freshly prepared turbidity was desired for the experiments, though the turbidity concentration levels that could be obtained were varying from on to another

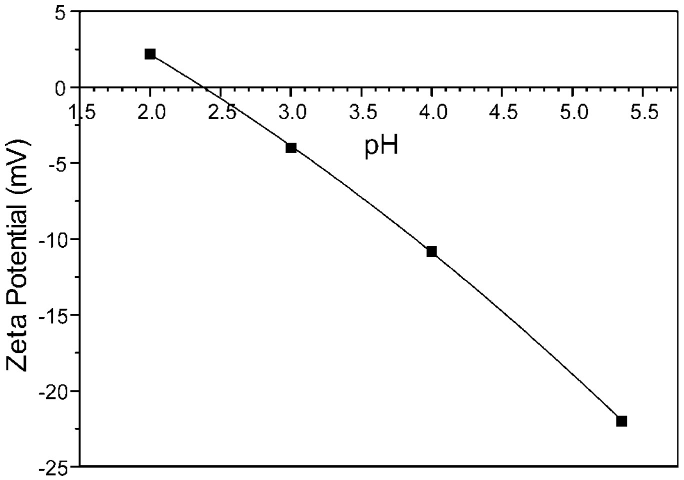

Figure 1. Zeta potential–pH relationship of indium turbidity (ionic strength 10 mM): pzc (point of zero charge) of indium turbidity = 2.35.

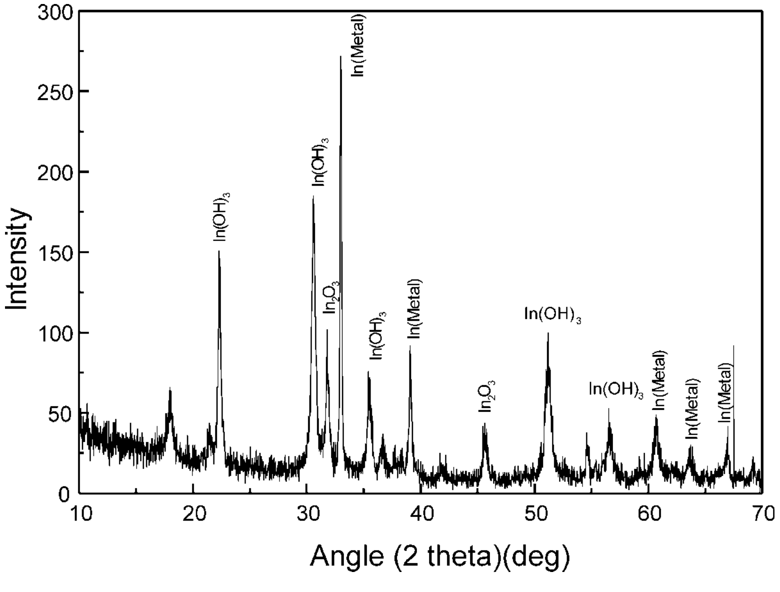

Figure 2. XRD spectra of indium turbidity.

(typically each lot would account for half to 1 L volume of turbid solution), different lots of indium turbidity with indium concentration in the range 0.9 to 6.4 ppm, were passed through the column. Depending on the indium concentration, inlet turbidity of the solution passed also varied from 1 to 7 NTU. The pH of inlet solution was always near neutral (6.0 to 6.5). Approximately 1.5 to 2.0 NTU turbidity of a freshly prepared solution corresponded to 1 ppm In. But this relation cannot be very accurate because indium turbidity of a particular solution measured in NTU units depends on the particle size and its distribution in the solution with both these parameters changing with time. Overall, a volume of 50 L of indium turbidity of average concentration 3.16 ppm In/L was passed through the column. This amounts to the passing of 3.16×10^4 bed volumes ppm of In for the 5 mL of the $=Mg \cdot Mg(OH)_2$ bed.

Indium concentration in the column outlet varied between 0.013 to 0.35 ppm, with an average of 0.09 ppm (< 0.2 NTU). With the exception of initial values of 0.150 to 0.350 ppm, throughout the run, the outlet value was between 0.01 to 0.03 ppm. This indicates a 97% indium turbidity removal efficiency of the column. After use, the original color of the column [pale yellow MB and white $Mg(OH)_2$ loaded resin] turned to

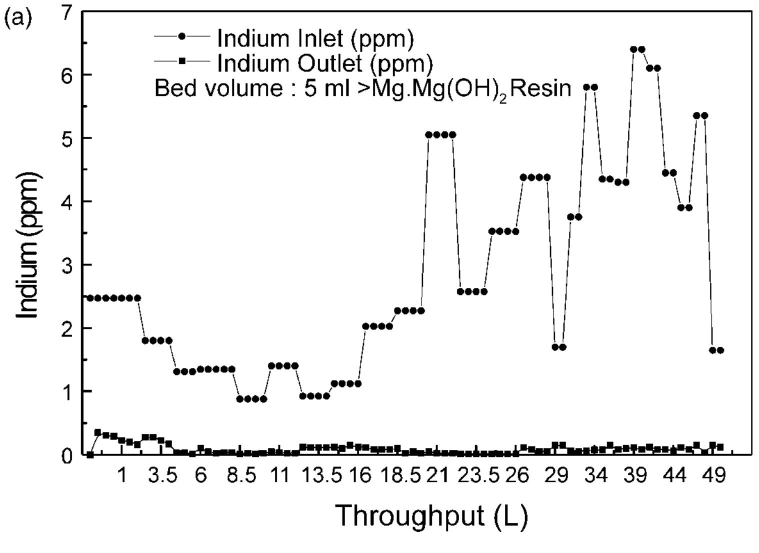

Figure 3. (a) Indium turbidity removal by $(RCOO)_2 \cdot Mg \cdot Mg(OH)_2$ resin. (b) $(RCOO)_2 \cdot Mg \cdot Mg(OH)_2$ resin column photograph, (A) used (indium loaded), and (B) fresh.

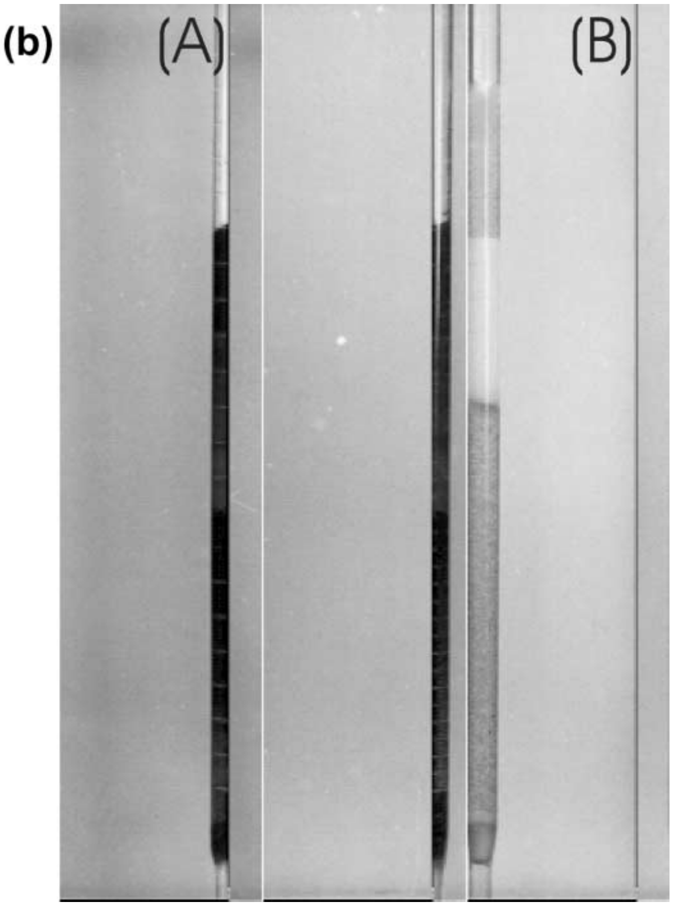

Figure 3. Continued.

black, indicating that turbidity removal may be mostly by adsorption rather than exchange. Outlet pH was always 0.5 to 0.6 units less than inlet, indicating some Mg slip from the $Mg(OH)_2$ loaded resin. Results also show that the wide variation in the inlet turbidity does not affect the outlet indium concentration and, hence, the column performance. Figure 3 shows a macrophotograph of columns having used (indium loaded) (A) and fresh bed of $(RCOO)_2{\cdot}Mg{\cdot}Mg(OH)_2$ resin (B). In the fresh bed, the $Mg(OH)_2$ precipitated resin is seen as a white column sandwiched between the top and bottom mixed beds.

Though one may consider that powdex resins may be suitable for turbidity removal, our laboratory results showed that the alumina turbidity removal capacity of H^+ form of strong acid powdex resin was found to correspond to only $\sim 0.2\%$ of total exchange capacity (TEC), while that of weak acid powdex resin is $\sim 0.5\%$ of TEC.[1] Hence, one has to resort to the principle of electrostatic attraction between the stationary and mobile phases for turbidity removal.

$Mg(OH)_2$ has a high pzc of 12. In solution of $pH < 12$, as in the present case, surface dissociation of —Mg—OH group can give a positively charged surface capable of adsorbing weakly negatively charged indium turbidity.[3] For this purpose, a weak acid cation exchanger resin was modified by incorporating $=Mg \cdot Mg(OH)_2$ to yield magnesium hydroxide precipitated magnesium-ion-loaded weak acid cation resin represented as $(RCOO)_2$-$Mg \cdot Mg(OH)_2$. The resin presents a $Mg(OH)_2$ bearing surface to the solution contacting it. Indium turbidity removal mechanism is explained as follows.

As the pH of initial solution entering resin bed is (6 to 7) less than pzc of $Mg(OH)_2$, the resin surface, having precipitated $Mg(OH)_2$, gets modified to positively charged surface.

$$\underset{\text{(Solid phase)}}{-\mathrm{Mg}-\mathrm{OH}} \xrightarrow[\text{pH<pzc}]{H^+} -\mathrm{MgOH}_2^+ \leftrightarrow -\mathrm{Mg}^+ + \mathrm{H_2O}$$

Due to definite solubility of $Mg(OH)_2$ ($K_s = 5.61 \times 10^{-12}$ moles/lit^2), solution pH changes from near neutral to 10 to 10.2, which is more than the pzc of indium turbidity, i.e., $In_2O_3/In(OH)_3$, and modifies the charge of turbid particles to sufficiently negative value to be adsorbed on the positively charged resin surface.

$$\underset{\mathrm{In_2O_3/In(OH)_3}\ \text{suspension}}{> \mathrm{In}-\mathrm{OH}} \xrightarrow[\text{pH>pzc}]{OH^-} > \mathrm{In}-(\mathrm{OH})_2^- \leftrightarrow > \mathrm{InO}^- + \mathrm{H_2O}$$

Earlier, $Mg(OH)_2$ loaded weak acid resin was used for removing aluminum turbidity generated by the interaction of heavy water (D_2O) with aluminum fuel clad of the Dhruva reactor in India.[1] An electrochemical filter using carbon felt was also used to remove the aluminum turbidity from reactor water.[2] But in both the cases, the system turbidity and, hence, aluminum concentration was much higher (10 to 20 ppm) compared to the RAPS system indium turbidity (0.1 to 0.15 ppm In). The pzc of aluminum turbidity was 5.4 and that of indium turbidity is 2.3. Hence, indium turbidity particles are more negatively charged. So more coulombic attraction between $Mg(OH)_2$ precipitated resin surface and turbidity particles resulted in better column performance, even at an indium concentration lower by an order of magnitude.

Fibrous Graphite Electrode Method

Lab Generated Turbidity Removal

Results of a run performed on lab generated turbidity with electrochemical carbon felt cell are shown in Fig. 4. At high (4.6 ppm In) and low (0.23 ppm In) indium inlet turbidity level, outlet indium was less than 0.001 ppm, except the initial slip of 0.03 ppm at the beginning of low concentration run. Under closed circuit conditions, applied field during low concentration run, 50 V emf was applied to cell and a current of 20 mA was observed. During the high concentration run, 16.5 V emf was required to get 20 mA.

Removal of Moderator Turbidity

A block diagram of the experimental setup as connected to the moderator sampling station as shown in Fig. 5. As shown, the filter was operated with the inlet moderator D_2O flowing from the bottom of the column to top. The exiting heavy water was collected in a plastic carboy. The valve arrangements made in the hook up of this column to the moderator sample point permitted a minimum flow rate of about 30 to 35 mL/min. A ≈ 1.2 m height semicircular

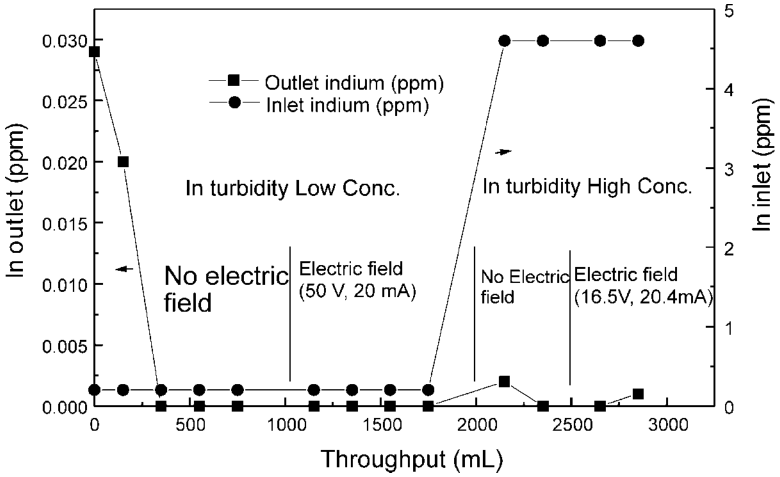

Figure 4. Indium turbidity removal from lab generated turbidity using an electrochemical filter.

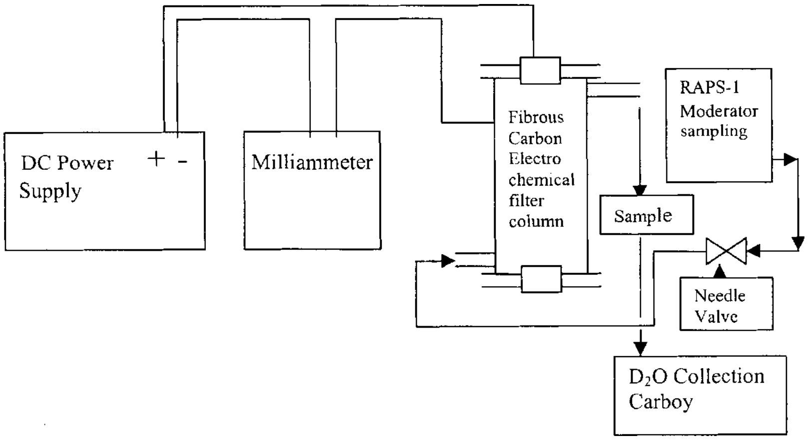

Figure 5. Block diagram of indium turbidity test removal setup connected to the RAPS moderator sampling point.

lead screen mounted on a trolley was employed to isolate the experimental set up in the moderator sampling area. The turbidity removal was followed by monitoring the ^{116m}In radioactivity in the electrochemical filter column inlet and outlet solutions. The 2112.3 keV gamma (yield 15%) was chosen to minimize the Compton scattering interference in the photo peak area determination of this isotope. A 65 cc active volume HPGe (high-purity germanium) detector coupled to a 4 K multichannel analyzer was used for gamma counting. Additionally, the dose rate measurements near the bottom (close to inlet), middle, and top (close to outlet) zones of the column were carried out to assess the progress of indium turbidity removal. This was done using an teletector γ dose monitor. Since the different zones of the column could not be shielded, a zone with a higher field (expected to be the bottom inlet zone) contributing to the dose measured at the upper lower dose zones (middle and top) is anticipated.

To minimize the radiation exposure to personnel involved in the experiments, it was felt prudent to connect the electrochemical filter to the system immediately after a shutdown of the reactor and monitor both inlet and outlet solution indium radioactivity levels at regular intervals of time on 4-12-01 at 1342 h. The electrochemical filter column was connected to the system within 90 minutes after reactor shutdown (i.e., 1510 h). Since the reactor was shutdown, the normal ion-exchange inlet sampling point (SS#3) could not be made use of for providing the moderator D_2O to the electrochemical filter column and, instead, the adjuster rod moderator D_2O cooling water line sampling point was used for this trial removal study. It takes more than 40 minutes to get the heavy water flowing through this sample line from the core to the sampling point. The first column inlet and outlet samples were taken at 1620 h, i.e., 70 minutes after valving-in the column. The electrochemical filter was operated at an applied potential of 40 V and a current of ≈ 0.8 to 1.4 mA could be realized. The temperature of the moderator was close to 303 K during this study.

Table 1 shows the performance of the electrochemical filter column in removing indium from the moderator in terms of ^{116m}In radioactivity levels at the column inlet and outlet, percentage indium removed, cell currents, and cell voltage. As seen in the data, the inlet activity was decreasing with time since the experiments began after the shutdown of the reactor. Except for the first inlet value (1620 h), the other values seem to follow closely the half-life of ^{116m}In. The first value (3700 μCi/L) appears to be ≈ 20% lower than its would-be actual value, which is calculated from the second and further activity values using the radioactive decay equation after applying the decay correction. The adjuster rod cooling water-sampling point is not normally used for moderator sampling during reactor operation. Hence, the somewhat lower first value could possibly be because of some mixing of stagnant heavy water in the sample line with the actual moderator sample. The waiting period of 70 minutes after valving-in (at 1510 h) and before sample collection (at 1620 h) appears to be somewhat less than the actual time lapse required for a representative sample collection after valving-in the column. Once a steady flow of the actual moderator heavy water was established through the column,

Table 1. Test removal of indium from moderator of RAPS-1: radioindium data.

				^{116m}In activity (μCi/L)		Percentage indium removal
Number	Sample time (h) (on 4-12-2001)	Cell voltage (V)	Cell current (mA)	Column inlet	Column outlet	Removal factor (RF)[a]
1	1620	40	1.3	3700	200	94.6/18.5
2	1732	40	1.0	2258	189	91.6/11.9
3	1835	40	0.9	918	89	90.3/10.3
4	1955	40	0.8	425	65	84.7/6.5
5	2155	40	1.4	143	24	83.2/6.0

[a] Removal factor (RF) = column inlet activity/column outlet activity; Percentage removal = [{1 − (1/RF)}] × 100.

samples were collected at any time from both the inlet and the outlet positions of the column. It can be noted that it takes about 6 minutes for the inlet water to come out of the column. In this period, there could be an $\approx 8\%$ reduction in the activity of outlet samples compared to the sample entering the inlet. Hence, any reduction in the outlet activity greater than 7.5% is to be attributed to the sorption capacity of the column. As such, the activity of the outlet samples (see Table 1) was not corrected for decay for this period of transit (6 min) through the column. However, the radioactivity values of inlet and outlet samples were decay corrected for the time elapsed between sample time and count time.

The percentage indium removal can be expressed by Eq. (1):

$$\%\,\text{removal} = [\{1 - (1/\text{RF})\}100] \tag{1}$$

where RF, the removal factor = column inlet activity/column outlet activity.

Due to electroadsorption of colloidal indium, the electrochemical filter column outlet activity values are lower by a factor (RF) ranging from 18.5 to 6. As seen from the Table, the indium removal decreased from $\approx 95\%$ in the initial stages to $\approx 83\%$ at the end of about 6 h of run. It can be noted that for this 12% decrease in removal, the RF value decreased by 66%, i.e., from an initial value of 18.5 to the end of run value of 6. The decrease in percentage removal, as indicated by radioactivity measurements, may be envisaged to be arising due to a decrease in the inlet activity values and not due to any reduction in the efficiency of column to sorb the indium. It is well-known in chemical decontamination operation that locations with higher initial radioactivity tend to show higher decontamination factors (higher % oxide removal) than those where the initial activity is lower. We, thus, infer that an 85% to 90% removal efficiency can be assigned to the electrochemical filter column. We can also argue that the efficiency could have been higher than 90% had the column been operated at its designed flow rate of ≈ 20 mL/min against the minimum flow rate of 30 to 35 mL/min that could be achieved in this test removal experiment. We could ascribe this reason because in a pilot run conducted a day earlier when the reactor was operating at constant power of 90 Mwe and when the flow rate could only be adjusted to a minimum of 60 to 70 mL/min, no sorption of indium activity by the column was observed.

Table 2 shows the radiation field accumulated on the column with the progress of the test removal run. The radiation field on the column at any time, t, is a resultant of the earlier sorbed indium activity decaying with time and freshly sorbed activity, the latter being at a lower value at t as compared to the value at an earlier time ($t - \Delta t$). Accordingly, the radiation field observed at the bottom of the column showed a lower decrease with time than the decrease warranted by half-life. However, the radiation field at the column outlet

Table 2. Radiation field on the electrochemical filter during test removal of indium from the moderator of RAPS-1.

		Radiation field on electrochemical filter column (mR/h)		
Number	Sample time (h) (on 4-12-2001)	Bottom (inlet) of column[a]	Middle of column	Top (outlet) of column
1	1620	>10000	2000	400
2	1732	5000	1800	350
3	1835	3000	800	300
4	1955	2500	700	300
5	2155	1400	450	300

[a] The terms bottom, middle, and top of the column refer to about 10 to 12 cm

remained nearly steady at ≈ 300–400 mR/h (3 to 4 mSv/h) without showing a decrease similar at the bottom of the column. This is due to the background γ shine from the bottom of the column contributing to the dose measurements at the top of the column. The high field at the bottom of the column shows that it is the initial 4 to 5 anode/cathode pairs that essentially contributed to the sorption process. The cell current of ≈ 1 mA applied to the electrochemical cell induced a voltage of 40 V. Because the moderator had a sp. conductivity of $0.5\,\mu\mathrm{S\,cm^{-1}}$, it is likely that most of the voltage drop occurs across the solution resistance and only a small portion of the applied voltage is experienced by the electrical double layer to drive the adsorption process. Unlike the experiment with laboratory generated turbidity, the present experiment with moderator indium turbidity could be not be conducted without applying the field (since radioactivity in the moderator was decaying and the cell could only be operated in the desired applied field mode to collect the turbidity removal data) to assess as to what extent the applied field has helped. In the case of laboratory generated indium turbidity, the removal was quantitative, even without applying the electrical field. However, it is reported that in the removal of aluminum turbidity (colloidal Al_2O_3) by this fibrous carbon electrode method, the application of an electric field had helped in clearing the turbidity to the clean water level. Without the field, about 90% reduction in turbidity was noticed.[2]

Zeta potential of carbon felt particles suspended in distilled water was found to be -25 mV at neutral pH and was similar to indium turbidity (-22 mV). This indicates that during open circuit conditions, turbidity removal is by physical adsorption. During closed circuit conditions, it is removed by electrostatic attraction and physical sorption.

CONCLUSION

Indium turbidity was generated and characterized for pzc, particle size, and form. More than 97% of turbidity removal was achieved by using a $Mg(OH)_2$ loaded weak acid cation resin in Mg form and fibrous graphite felt electrochemical cell. Attachment to a RAPS-I moderator system on an experimental basis, the electrochemical cell was tested. A large-size electrochemical filter will be required for the complete removal of indium turbidity from the moderator.

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