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Synthesis and characterization of cobalt ferrocyanides loaded on organic anion exchanger

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ABSTRACT

Transition metal ferrocyanides have important applications in the selective removal of radioactive caesium from low level and intermediate level radioactive liquid waste streams. The microcrystalline nature of these materials renders them useless for application in column mode operations. Special preparation procedures have been developed to prepare granular solids by in situ precipitation of metal ferrocyanides on organic anion exchangers, which is suitable for column mode operations. The elemental compositions of the metal ferrocyanides precipitated inside the pores of anion exchanger were determined by analysing the dissolved samples using ICP–AES system and flame photometer. From the XRD and EDX analyses and the elemental composition of the synthesized materials, the nature of the compound formed inside the anion exchanger was found to be cobalt ferrocyanide. From SEM analysis of the samples, the particle size of the cobalt ferrocyanide precipitated inside the anion exchanger was found to be much less than that of cobalt ferrocyanide precipitated outside. The efficiency of these materials for removal of Cs was evaluated by measuring the distribution coefficient (Kd), ion exchange capacity and kinetics of Cs uptake. The Kd of the materials loaded on anion exchanger was found to be of the order of 10^5 ml/g. The Cs uptake kinetics of the materials loaded on anion exchanger was slower than that of precipitated materials. The ion exchange capacity of the cobalt ferrocyanide loaded on anion exchanger was found to be much higher than that of the precipitated cobalt ferrocyanide.

1. Introduction

Radioactive liquid waste generated during reprocessing of spent fuel is broadly classified as high level waste (HLW), intermediate level waste (ILW) and low level waste (LLW). The volume of HLW generated from nuclear fuel cycle is much smaller than the volumes of ILW and LLW. Vitrification followed by disposal in deep geological repositories is the universally accepted treatment for HLW. Vitrification of HLW also generates substantial volumes of ILW and LLW. In view of the presence of significant quantities of non-radioactive constituents of these wastes, a concentration step before suitable matrix fixation is desirable. The volume reduction (concentration) is achieved by the use of highly selective sorbents and ion exchange materials.

Natural zeolites were the first materials to be used in large-scale processes to treat nuclear waste solutions. The ion exchange capacity of natural and synthetic zeolites may be as high as organic resins. But because of their relatively low selectivity, zeolites are not adequate for the removal of Cs from waste solutions having medium or high salt concentrations [1]. Their use is also limited to a narrow pH range. Transition metal ferrocyanides have pronounced selectivity for Cs in waste solutions of high salt concentrations and high stability towards radiation. They also have a wide pH range for uptake of Cs [2]. However, the microcrystalline nature of these materials renders them useless for application in column mode operations. Several methods have been proposed for making granulated ferrocyanides for use in large-scale treatment of liquid radioactive waste in column mode [3,4]. Campbell prepared granular solids by slow addition of potassium ferrocyanide solution to a cobalt salt solution [5]. Loose-Neskovic prepared large particles of ferrocyanides by slow growth of ferrocyanides on the surface of potassium or sodium ferrocyanide crystals [6]. Some results are reported for the formation of macro porous ion exchange beads by mixing ferrocyanide powders with liquid organic exchangers and polymerizing it [7]. One procedure to react porous gel spheres such as titanium oxide with ferrocyanide solution to convert the oxide into the corresponding ferrocyanide is also reported by some scientists [8,9]. Many investigators have used several types of support materials such as silica gel [10–12] to develop metal ferrocyanide with large stable particles. The kinetics of the ion exchange depends on the mass transfer within the particle and is slower as the particle size increases. There is also a tendency for preferential reaction of the surface and the average capacity may decrease as

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the surface area decreases [2]. Some researchers have also tried the loading of these ferrocyanides on organic ion exchange resins [13,14].

This study gives details of preparation, characterization and evaluation of ion exchange properties of cobalt ferrocyanide loaded on anion exchanger. The prepared ion exchange materials are highly specific for Cs in solutions of high salt concentration. Also they have wide pH range for Cs exchange [15]. The radiation stability of the materials loaded on anion exchanger is very good, as organic ion exchangers are stable to hard gamma radiation up to a dose of 10 mega rads [16].

2. Experimental

All the chemicals used for the experiments were of AR grade. Strong base anion exchange resin (Indion FFIP) having iso-porous structure was used for loading cobalt ferrocyanide. The resin samples were subjected to a preconditioning process to remove any water-soluble residue or undesired anions remaining in the anion exchange resin. The anion exchange capacity of the anion exchange resin was determined by conventional method [17].

2.1. Synthesis of cobalt ferrocyanide ion exchange materials

Potassium cobalt ferrocyanide powder was prepared by the conventional precipitation method. Synthesis of pure cobalt ferrocyanide ($Co_2Fe(CN)_6$) is difficult by conventional precipitation route and the product formed is invariably $K_2CoFe(CN)_6$. Hence, the precipitation of pure $Co_2Fe(CN)_6$ and loading of $Co_2Fe(CN)_6$ on anion exchange resin were carried out by the innovative method as given in our earlier publication [18]. The anion exchanger loaded with cobalt ferrocyanide was treated with 0.1 M KCl solution to get potassium cobalt ferrocyanide loaded ion exchanger.

2.2. Evaluation of prepared ion exchangers

2.2.1. Evaluation of compositions of synthesized cobalt ferrocyanide ion exchangers

The elemental compositions of the prepared materials were determined by dissolving them in sulphuric acid as per the procedure described by Lehto et al. in their report titled mechanism of caesium ion exchange on potassium cobalt hexacyanoferrates(II) [19].

(a) In the case of precipitated materials without anion exchanger, known weight of samples was taken and dissolved in concentrated sulphuric acid by heating. The acid solution was then evaporated to dryness and the residue was dissolved in concentrated hydrochloric acid.
(b) In the case of anion exchanger loaded resin hydrogen peroxide was added to the acid digested residue to destroy the organic materials of the anion exchanger and heated.

In both cases, the solutions were made up to known volume. The concentrations of iron and cobalt were determined by ICP–AES system (ARL, model 3520). Potassium concentration was determined by flame photometer. In order to minimise the matrix effect during the analysis of Co, Fe and K, the blank and standard solutions for ICP–AES and flame photometer were prepared in a similar manner as that of sample.

2.3. XRD analysis

The crystalline phases formed in the synthesized ion exchangers were determined by X-ray diffraction. Cu Kα radiation and graphite monochromator were used. The instrument used is Siemen's make, model KRISTALLOFLEX D-500.

2.4. SEM/EDX analysis

The crystallite size of the precipitated cobalt ferrocyanide and cobalt ferrocyanide loaded on anion exchanger was determined by SEM analysis. The EDX analysis of the precipitated and cobalt ferrocyanide loaded on anion exchanger was carried out to determine the composition of the materials formed. The instrument employed for the study is Philips ESEM, model – XL30 coupled with EDAX.

2.5. Determination of Cs uptake kinetics

Cs uptake kinetics of the prepared materials was determined. For this, known amount of the prepared exchanger materials were equilibrated with the known activity of ^{137}Cs spike solution for different lengths of time with continuous stirring. The supernatant was filtered and analysed for ^{137}Cs activity on a single channel analyzer having NaI(Tl) scintillation detector. From the initial and final activity of the solutions percentage uptake of Cs was calculated.

2.6. Evaluation of distribution coefficient (Kd) with respect to Cs uptake

For Kd determination precisely weighed quantities of dried samples were equilibrated with the known activity of ^{137}Cs spike solution by continuous stirring for 1 h at pH 8. The supernatant was filtered and analysed for ^{137}Cs activity, and the Kd value of the materials was calculated.

2.7. Evaluation of equilibrium ion exchange capacity

Precisely weighed quantities of dried samples of the prepared ion exchange materials were equilibrated with the known volume of 0.052 M $CsNO_3$ solution having ^{137}Cs spikes by continuous stirring for 1 h at pH 8. The supernatant was filtered and analysed for ^{137}Cs activity. From the concentration of Cs in the initial solution and the initial and final activity of ^{137}Cs in the equilibrated solution, the amount of Cs loaded on the ion exchanger and thereby, the Cs exchange capacity of the exchangers was calculated.

3. Result and discussion

During the loading of cobalt ferrocyanide on the anion exchanger, in the first step, when treated with potassium ferrocyanide solution, $[Fe(CN)_6]^{-4}$ ions got loaded onto the anion exchanger by ion exchange mechanism. In the second step, cobalt ferrocyanide solid got precipitated inside anion exchanger when treated with Co^{+2} ions. This precipitation reaction depends on the time of equilibration and concentration of Co ions in the solution. Determination of chemical formula of the compound formed and their phase identification are explained below.

3.1. Chemical formula of the synthesized materials

On the basis of ICP–AES and flame photometer analysis results, the elemental compositions and the corresponding chemical formulae of the synthesized ferrocyanides were calculated, and are given in Table 1. The chemical formula of the synthesized ferrocyanide compounds was derived by considering Fe concentration to unity and normalizing the Co concentration accordingly. In the case of potassium cobalt ferrocyanide, precipitated by conventional method, the proportion of K was calculated on the basis of

Table 1
Elemental analysis of prepared ion exchange materials and their formula.

Ion exchange material	Concentration, mmol/g of material formed			CoFC content (%)	Formula of the compound
	Co	Fe	K		
Precipitated KCoFC	2.713	2.439	4.428	19	$K_{1.78}Co_{1.11}Fe(CN)_6$
Precipitated CoFC	3.641	1.833	BDL	100	$Co_{1.99}Fe(CN)_6$
CoFC loaded on anion exchanger	0.523	0.657	BDL	100	$Co_2Fe(CN)_6$
KCoFC loaded on anion exchanger	0.521	0.631	0.491	53	$K_{1.28}Co_{1.36}Fe(CN)_6$

BDL, below detectable limit; KCoFC, potassium cobalt ferrocyanide; and CoFC, cobalt ferrocyanide.

the iron and cobalt values and taking into account electrical neutrality [19]. The concentration of K calculated, taking into account the electrical neutrality, is matching with the K concentration in the dissolved sample analysed (Table 1) within the error limits of analysis. In this case, since the compound formed is a mixture of $K_2CoFe(CN)_6$ and $Co_2Fe(CN)_6$ [19], the derived formula ($K_{1.8}Co_{1.1}Fe(CN)_6$) is non-stoichiometric. The $Co_2Fe(CN)_6$ content in the compound formed is about 19% (Table 1). In the case of pure cobalt ferrocyanide synthesized, the derived formula ($Co_{1.99}Fe(CN)_6$) is exactly matching with the ideal formula $Co_2Fe(CN)_6$ within the limit of experimental errors. The absence of K in the sample further confirms the purity of the sample (Table 1).

In the case of cobalt ferrocyanide and potassium cobalt ferrocyanide loaded on anion exchanger, the concentration of Fe cannot be taken as unity to derive the formula of the ferrocyanide precipitated inside the pores of the anion exchanger. This is due to the excess concentration of Fe on the anion exchanger fully loaded with ferrocyanide ions. In the case of cobalt ferrocyanide loaded on anion exchanger, the formula was derived from the concentration of Co only (Table 1). For potassium cobalt ferrocyanide present inside the anion exchanger, the amount of $K_2CoFe(CN)_6$ in the sample was calculated from the K concentration. The concentration of Co in the mixture was found to be much in excess than that required to satisfy the electrical neutrality of $K_2CoFe(CN)_6$ content. This indicates that the conversion of $Co_2Fe(CN)_6$ to $K_2CoFe(CN)_6$ by ion exchange was not complete during KCl treatment. The amount of $Co_2Fe(CN)_6$ in the mixture was calculated from the excess Co concentration. The Fe concentration required for the formation of mixture of $Co_2Fe(CN)_6$ and $K_2CoFe(CN)_6$ in the sample was calculated. The average formula of the mixture ($K_{1.25}Co_{1.38}Fe(CN)_6$) was derived by normalizing the concentrations of K and Co considering Fe concentration as unity as explained earlier (Table 1). The content of the remaining $Co_2Fe(CN)_6$ in the metal ferrocyanide inside the anion exchanger is about 53% (Table 1).

The chemical analysis results of pure cobalt ferrocyanide precipitated, and that loaded on anion exchanger shows the absence of K in these samples (Table 1). The EDX spectrum of these compounds (Figs. 1 and 2) also confirms the above observation as there is no peak corresponding to K in the spectrum. The semi quantitative wt% analysis of precipitated pure cobalt ferrocyanide calculated from the EDX spectrum (Fig. 1) in terms of the ratio of Co and Fe (1.94) is in good agreement with the theoretical value (2.10) calculated using the formula $Co_2Fe(CN)_6$ (Table 2). This also is in good agreement with the ratio of Co and Fe calculated (2.09) from chemical analysis (Table 2). This again supports the formula derived ($Co_2Fe(CN)_6$). Phase identification of this material by XRD analysis (Fig. 3(a)) also shows that the compound formed is $Co_2Fe(CN)_6$ as the phases identified are $Co_2Fe(CN)_6 \cdot 10H_2O$ (JCPDS Card No. 36-505), $Co_2Fe(CN)_6 \cdot 2H_2O$ (JCPDS Card No. 23-189) and $Co_2Fe(CN)_6$ (JCPDS Card No. 30-47).

In the case of cobalt ferrocyanide loaded on anion exchanger, the ratio of Co and Fe calculated from chemical analysis (0.84) and EDX analysis (0.78) is comparable (Table 2). This ratio is less compared to the theoretical value (2.10) due to the higher loading of ferrocyanide ions on the anion exchanger than that required to form $Co_2Fe(CN)_6$. The XRD pattern of ferrocyanide ion loaded anion exchanger is amorphous (Fig. 3) with a broad hump in the 2θ range of 15–25°. The phases identified from the XRD pattern of the cobalt ferrocyanide loaded on anion exchanger (Fig. 4(b)) were similar to that of precipitated pure cobalt ferrocyanide. This XRD trace had higher background due to the presence of ferrocyanide ion loaded anion exchanger. This confirms that the compound formed inside

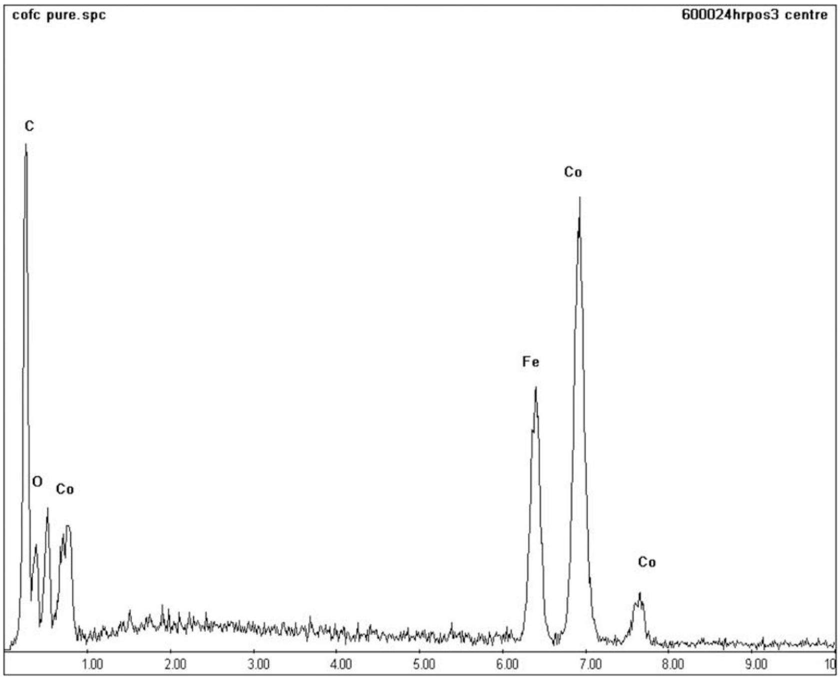

Fig. 1. EDX spectrum of precipitated $Co_2Fe(CN)_6$.

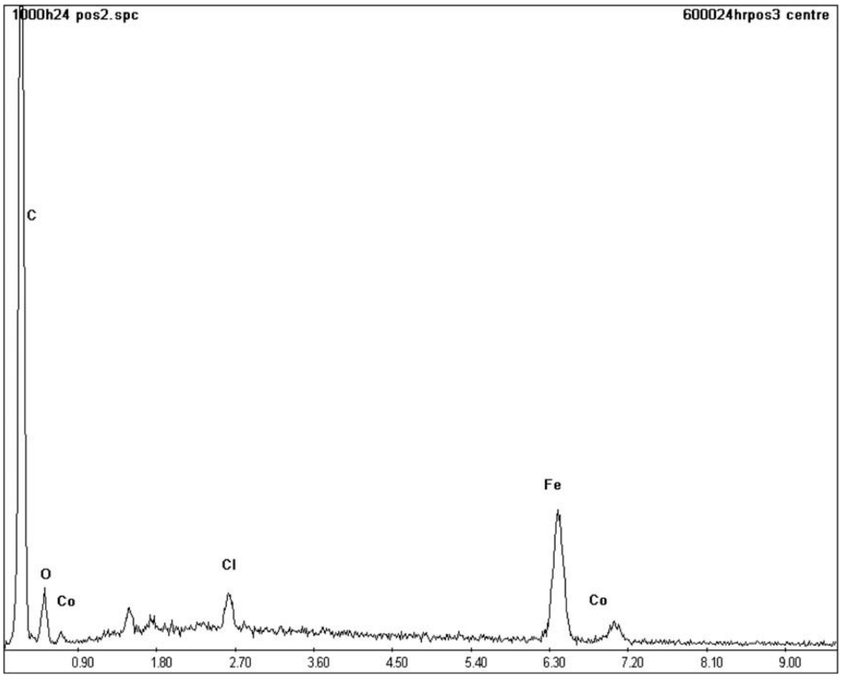

Fig. 2. EDX spectrum of cobalt ferrocyanide loaded on anion exchanger.

Table 2
Ratio of Co and Fe in the prepared ion exchange materials.

Ion exchange material	Percentage of material on the exchanger	Co/Fe ratio		
		Chemical	EDX	Theoretical
Precipitated KCoFC	–	1.18	–	1.05
Precipitated CoFC	–	2.09	1.94	2.10
CoFC loaded on anion exchanger	13.3	0.84	0.78	2.10
KCoFC loaded on anion exchanger	15.6	0.87	–	1.05

KCoFC, potassium cobalt ferrocyanide; and CoFC, cobalt ferrocyanide.

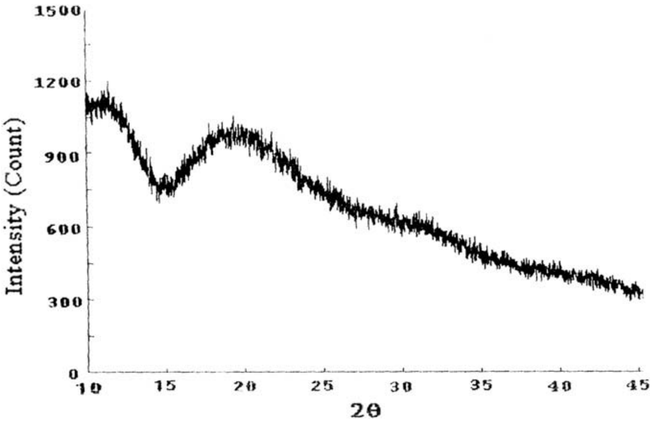

Fig. 3. XRD scan of ferrocyanide ion loaded anion exchanger.

the anion exchanger is also pure $Co_2Fe(CN)_6$ only. Therefore, the preparation of pure $Co_2Fe(CN)_6$ can be achieved by the innovative precipitation method or by loading it on anion exchanger, which is not possible by conventional precipitation method as the product formed will be always a mixture of $K_2CoFe(CN)_6$ and $Co_2Fe(CN)_6$.

The XRD pattern of the precipitated potassium cobalt ferrocyanide and that loaded on anion exchanger are given in Fig. 5(a) and (b), respectively. The major phase identified was $K_2CoFe(CN)_6$ (JCPDS Card No. 31–1000) in both cases. The broad hump at 20° 2θ of Fig. 5(b) is due to the presence of ferrocyanide ion loaded anion exchanger. In the XRD trace 'a' the line width for the peaks is more compared to that in 'b'. This is due to the smaller size of the crystals (Fig. 5(a)) of potassium cobalt ferrocyanide precipitated by conventional method (30–50 nm) [20] than that (Fig. 5(b)) present inside the pores of anion exchanger (0.5–1 μm), which is formed by exchanging Co ions of cobalt ferrocyanide with K ions.

The percentage of material formed was calculated from the concentrations of Co and K and the derived formula of the compound (Table 2). Molecular weights of potassium cobalt ferrocyanide and cobalt ferrocyanide were taken as 349 and 510, respectively, for the calculation. From Table 2, it can be observed that the amount of cobalt ferrocyanide loaded on anion exchanger is 13.3% and the amount of potassium cobalt ferrocyanide loaded on the anion exchanger is 15.6%.

3.2. Evaluation with respect to Cs uptake

In order to use the prepared ion exchanger for selective removal of Cs from radioactive waste solutions having Cs concentration in ppb level, the material was evaluated for their performance with respect to Cs uptake kinetics, Kd, capacity, etc. As the wastes to be treated by the exchanger have pH in the range of 8–9 the studies were carried out at pH 8.

In an earlier work carried out by us, the adsorption isotherm for the sorption of Cs ions by cobalt ferrocyanide loaded anion exchanger was studied by varying Cs ion concentration in the equilibrating solution. The data obtained were interpreted using various adsorption models (paper submitted for publication).

3.2.1. Cs uptake kinetics of synthesized materials

Cs uptake kinetics of precipitated potassium cobalt ferrocyanide and cobalt ferrocyanide are very fast (Table 3). The uptake was al-

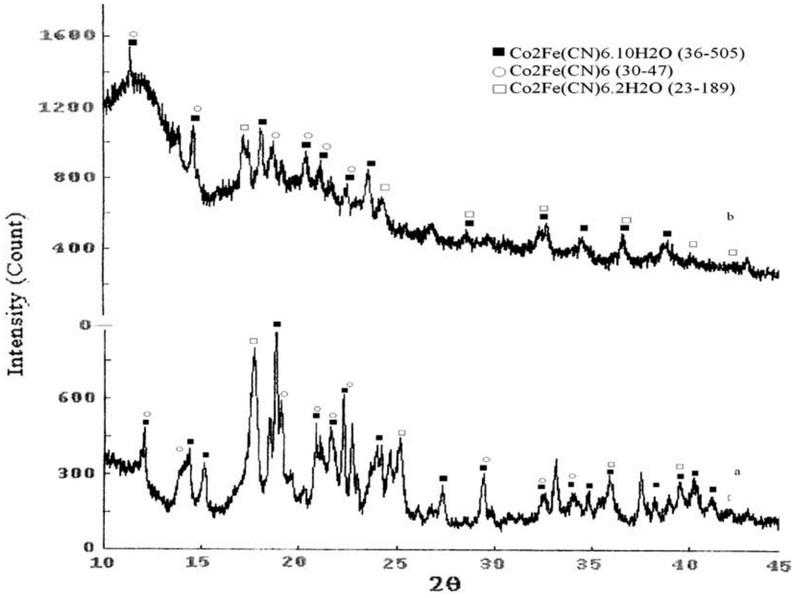

Fig. 4. XRD scan of (a) precipitated $Co_2Fe(CN)_6$ and (b) $Co_2Fe(CN)_6$ loaded on anion exchanger.

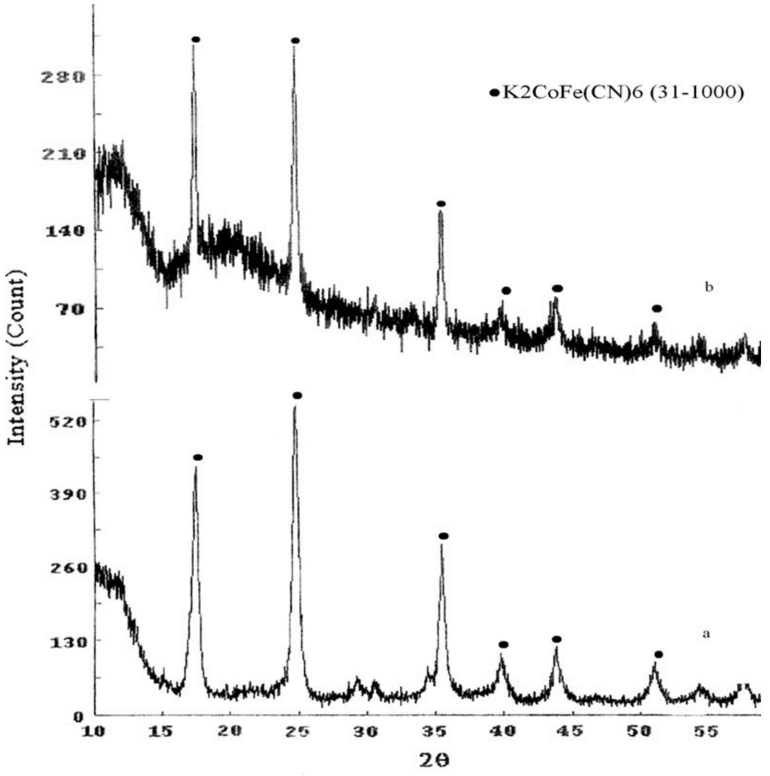

Fig. 5. XRD scan of (a) precipitated $K_2CoFe(CN)_6$ and (b) $K_2CoFe(CN)_6$ loaded on anion exchanger.

most complete within 1 min of equilibration time. Whereas Cs uptake of the metal ferrocyanides loaded on anion exchanger is slow (Fig. 6) due to the slow diffusion of exchangeable ions through the pores of anion exchanger. Compared to the Cs uptake of cobalt ferrocyanides loaded on anion exchanger, Cs uptake of potassium cobalt ferrocyanide loaded on anion exchanger is faster. This is in agreement with the higher feasibility of exchange of one Cs ion for one K ion than the exchange of two Cs ions for one Co ion. Therefore, loading of cobalt ferrocyanide on anion exchanger retarded the kinetics of Cs uptake.

3.2.2. Distribution coefficients (Kd) of the synthesised ion exchangers

The distribution coefficients (Kd) of the synthesised ion exchangers with respect to Cs uptake are given in Table 4. The Kd value of precipitated potassium cobalt ferrocyanide is very high and is in good agreement with the literature values [2]. The Kd value of precipitated cobalt ferrocyanide is also found to be very high which is not reported earlier. Kd values of metal ferrocyanides loaded on anion exchanger are about one order less than the precipitated ferrocyanides (Table 4). However, considering the percentage loading (calculated considering the dry sample) of metal

Table 3
Cs uptake kinetics of synthesised ion exchange materials.

Time (min)	Percentage uptake			
	Precipitated $K_2CoFe(CN)_6$	Precipitated $Co_2Fe(CN)_6$	Resin loaded $K_2CoFe(CN)_6$	Resin loaded $Co_2Fe(CN)_6$
1	99.5	99.3	65.9	39.8
3	99.6	99.5		
5	99.3	99.2	81.3	46.8
15			94.0	59.1
30			95.4	77.4
60			96.2	90.0
120			98.1	95.4
240			99.6	99.1

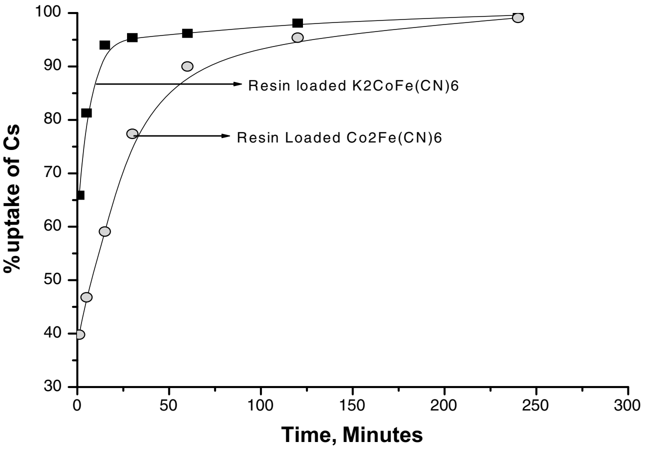

Fig. 6. Cs uptake kinetics of the anion exchanger loaded materials.

Table 4
Kd values and capacity of prepared ion exchange materials.

Ion exchange material	Kd (ml/g)	Normalised Kd (ml/g)	Ion exchange capacity (meq/g)	Normalised capacity (meq/g)
Precipitated KCoFC	266962	266962	0.747	0.747
Precipitated CoFC	217461	217461	1.151	1.151
KCoFC loaded on anion exchanger	26041	146300	0.420	2.35
CoFC loaded on anion exchanger	23380	193223	0.583	4.82

KCoFC, potassium cobalt ferrocyanide; and CoFC, cobalt ferrocyanide.

ferrocyanides on the anion exchanger (Table 2), the normalized Kd value approaches that of the precipitated materials. Hence, loading of metal ferrocyanide on the anion exchanger did not affect the distribution coefficient of the material, though the kinetics of uptake of Cs becomes slow.

3.2.3. Cs exchange capacities of the synthesized materials

The Cs exchange capacities of the synthesised metal ferrocyanides are given in Table 4. In comparison to the Cs exchange capacity of precipitated cobalt ferrocyanide, the Cs exchange capacity of precipitated potassium cobalt ferrocyanide is less [2]. This is true for the metal ferrocyanides loaded on anion exchanger also. The Cs exchange capacity of the metal ferrocyanides loaded on anion exchanger is less compared to that of the precipitated materials. But considering the percentage loading (calculated considering the dry sample) of potassium cobalt ferrocyanide and cobalt ferrocyanide on the anion exchanger (Table 2), these values are very high and has approached the theoretical exchange capacities (5.73 and 6.05 meq/gm, respectively) calculated on the basis of single Co^{2+} exchange for two Cs^+ or two K^+ for two Cs^+ ions, respectively. The higher capacity of these ion exchangers may be due to the higher surface area of the material inside the anion exchanger pores, because the ion exchange capacity is a function of surface area in the case of ferrocyanides [2]. Therefore, to determine the particle size (thereby the surface area) of the precipitated cobalt ferrocyanide and that loaded on anion exchanger, SEM analysis of these materials was carried out (Figs. 7 and 8). From these SEM micrographs, the size of the particles of precipitated pure cobalt ferrocyanide was found to be about 4–5 μm (Fig. 7) and that of cobalt ferrocyanide loaded on anion exchanger was about 0.5–1 μm (Fig. 8). The reduction in size of the precipitates is due to hindered growth of crystals within the pores of resin. The reduction in size of the particles in the latter case caused the increase in the surface area of the exchangeable material that in turn caused the higher capacity for Cs uptake. Hence, the drastic increase in the ion exchange capacity of cobalt ferrocyanide loaded on anion exchanger is attributed to ~20–30-fold increase in surface area of the material due to smaller particle size of cobalt ferrocyanide precipitated within the anion exchange resin (0.5–1 μm) as compared to cobalt ferrocyanide precipitated outside (4–5 μm).

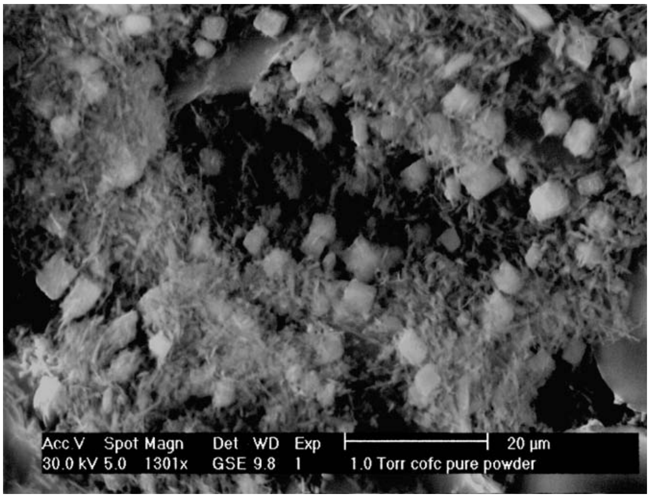

Fig. 7. SEM micrograph of precipitated $Co_2Fe(CN)_6$.

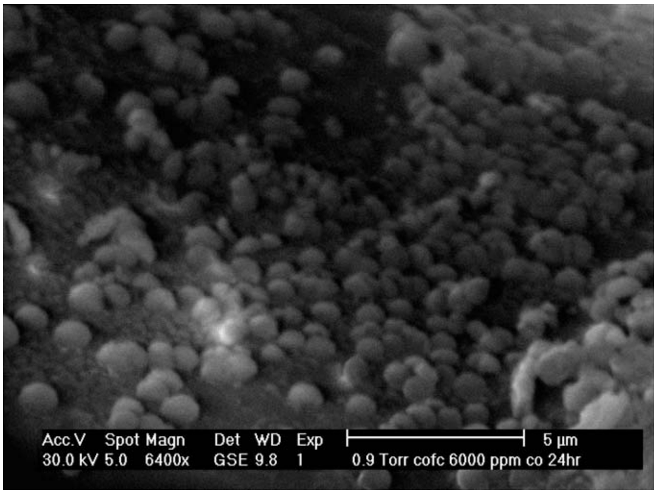

Fig. 8. SEM micrograph of $Co_2Fe(CN)_6$ loaded on anion exchanger.

4. Conclusion

The conventional precipitation method of potassium ferrocyanide produced a mixture of $K_2CoFe(CN)_6$ and $Co_2Fe(CN)_6$ and the $Co_2Fe(CN)_6$ content in the precipitated compound $K_{1.83}Co_{1.12}Fe(CN)_6$ was found to be ≈19%. The cobalt ferrocyanide prepared by precipitation and by loading on anion exchanger was found to be pure $Co_2Fe(CN)_6$. Hence, preparation of pure $Co_2Fe(CN)_6$ can be achieved by the innovative precipitation method or by loading it on anion exchanger, which is not possible by conventional precipitation method.

Cs uptake of exchanger loaded with metal ferrocyanides is slow compared to that of precipitated pure metal ferrocyanides. Also Cs uptake of potassium cobalt ferrocyanide loaded on anion exchanger is faster compared to that of cobalt ferrocyanide loaded on anion exchanger. The Kd values of precipitated potassium cobalt ferrocyanide and cobalt ferrocyanide are very high. The normalized Kd value of materials loaded on anion exchanger is similar to that of precipitated materials. Loading of metal ferrocyanide on the anion exchanger did not affect the distribution coefficient of the material, though the kinetics of uptake of Cs becomes slow.

The capacity of the materials loaded on anion exchanger is very high considering the percentage loading of the materials on the anion exchanger and is approaching the theoretical exchange capacities. The drastic increase in the capacity in the case of materials loaded on anion exchanger is attributed to the increase in surface area of cobalt ferrocyanide precipitated within the anion exchanger. Though the purpose of loading of cobalt ferrocyanide on organic anion exchanger is to get column grade material for ion exchange operations, increase in the Cs exchange capacity nearing theoretical value is a remarkable achievement.

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