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BHABHA ATOMIC RESEARCH CENTRE

REMOVAL OF RADIONUCLIDES FROM RADIOACTIVE
EFFLUENTS OF PUREX ORIGIN USING
BIOMASS BANANA PITH AS SORBANT

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60	<i>Abstract :</i>	Investigations have been carried out on the applicability of dried banana pith (inner stem) for the sorption of various radionuclides viz. U, Pu, ²⁴¹ Am, ¹⁴⁴ Ce, ¹⁴⁷ Pm, ¹⁵²⁺¹⁵⁴ Eu and ¹³⁷ Cs which are generally present at trace level in Purex process waste effluents. The sorption of trivalent radionuclides as well as tetravalent plutonium was found to be high at pH 2, whereas sorption of uranium was found to be maximum at pH 6. Cesium was not found to be sorbed. ²⁴¹ Am sorption was investigated in detail as a representative element of trivalent actinides and fission products to study the general trend. Though its sorption was kinetically slow, near-quantitative sorption was observed on prolonged contact. ²⁴¹ Am sorption was studied in presence of NaNO ₃ (up to 1 M) and Nd(III) up to 500 mg/l. Whereas no significant change in distribution ratios (D) was observed in the presence of NaNO ₃ , it increased with neodymium concentration in the range tested. This indicates the effectiveness of the biomass as sorbent even in presence of sodium salts. Sorbed metal ions could be recovered by leaching with 2 M nitric acid. The dried biomass samples prepared from different sources were found to be stable for months and gave similar results on testing. The biomass was tested for its applicability for sorbing radionuclides present in Purex evaporator condensate and diluted high level waste solution on once through basis. The sorption capacity of banana pith for trivalent actinide-lanthanide is in the range of 60 mg/g banana pith. The results indicate that the biomass can be used effectively for the treatment of Purex Waste effluents for the removal of strontium, tri- and tetravalent actinides and fission products. The biomass was also tested for the sorption of toxic metal ions viz. Sr, Hg, Pb, Cr, Cd, and As from a nitrate solution at pH 2 and 4. D values followed the order Hg > Sr > Cd > Pb at pH 2, with Cr and As showing no uptake. These results indicate the potential of this biomass for use in treating conventional industrial waste.
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REMOVAL OF RADIONUCLIDES FROM RADIOACTIVE EFFLUENTS OF PUREX ORIGIN USING BIOMASS BANANA PITH AS SORBANT

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1. INTRODUCTION

There is an increasing interest in the use of various types of biomass in controlled processing systems for the accumulation and concentration of dissolved metals from the aqueous solution. This is particularly true for the removal of trace amounts of radioactive pollutants in nuclear processing facilities. Most of the reported results have been with the use of micro-organisms⁽¹⁻⁴⁾ either alive or dead, as bioadsorbants. Recently, it has been shown that certain plant tissues are also effective for this type of adsorption process^(5,6)

Banana (*musa*) is grown extensively in tropical and subtropical countries, and 14.37% of the world production is shared by India, the second largest producer in the world. The massive waste generated from banana cultivation, *viz.* the stem, leaves and pseudostem, besides fruit skin, are accumulated in the environment posing serious problems of sorbing and accumulating nutrient from the soil thereby depriving these nutrients to other vegetation. Several attempts have been made to utilize these wastes through ensilaging to eliminate or reduce the negative nutritional effects on the environment⁽⁷⁾. As this plant material is widely available and relatively inexpensive, an investigation on its utility as a bioadsorbant has been carried out. This paper presents the data collected on the use of banana stem as biosorbant for some of the toxic and radionuclides.

2. EXPERIMENTAL

Materials and Method

Banana Pith: After harvesting, the inner stem portions of banana plant (banana pith), grown at Chembur, Mumbai (Bombay) suburb, were laterally cut into small pieces (less than 3 mm thick) and then dried at 90-100°C to constant weight. These dried pieces were used either as such in the form of lumps or after crushing them to 30-50 mesh size. The pseudo stem (waste portion of the stem within the soil) was also used in some experiments after drying as above.

Radioactive Tracers: The radioactive tracers ^{233}U , Pu (mainly 239), ^{241}Am , $^{152+154}\text{Eu}$, and ^{147}Pm were procured and purified by standard procedures^(8,9) and used in the experiments.

Purex HLW: The major radionuclides of acidic Purex High level waste (HLW) (3-4 M HNO_3) are fission products like ^{144}Ce , ^{137}Cs , ^{90}Sr , ^{106}Ru , ^{95}Zr , ^{95}Nb etc., actinides viz. Pu (mainly ^{239}Pu), ^{241}Am and ^{237}Np , corrosion products such as Fe, Cr, Ni and Mn and process chemicals in the form of nitrates. This solution was diluted and used in the experiments.

Purex Condensate: In Purex process, the condensed vapours from a number of evaporators that concentrate wastes as well as products are collected and again concentrated. The condensed liquid from this evaporation (Condensate) contains gross beta and gamma activities $< 0.1 \mu\text{Ci/l}$. Sorption of alpha activities from americium spiked evaporator condensate sample was tested.

Toxic Metal Ions: Composite mixtures of Sr^{2+} , Cr^{3+} , Cd^{2+} , As^{3+} , Pb^{2+} and Hg^{2+} at 25 ppm level each in nitrate solutions at pH 2 and 4 were also prepared by mixing appropriate quantities from their individual stock solution of 1 mg/ml and used in the experiment.

Instrumentation: Alpha activity was assayed using 2π argon gas flow proportional counter standardized against ^{239}Pu . Beta activity was assayed using burshane gas flow proportional

counter standardized against RaDE source whereas gamma counting was carried out with a well type scintillation counter using a NaI(Tl) detector standardized against ^{137}Cs . Individual fission products were determined by gamma spectrometry using a HPGe detector coupled to a 4K multichannel analyser.

Estimation of non-radioactive toxic metal ions were carried out using an Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES).

Analysis of Banana Stem: Preliminary tests were carried out with known weights (~10 g) of banana stem and pseudostem repeatedly heated at 90-100°C to constant weight. Known weights of the sample pieces were incinerated at 800°C and the residue was dissolved in 2 M HNO_3 and analysed for trace metal contents.

Batch Sorption Studies: In all batch experiments, a known volume (generally 5 ml) of the aqueous phase containing trace level metal ions was contacted with biomass of known weight (generally 50 mg) for a known period of time. Wherever necessary, the pH of the solution was adjusted with either 2 M HNO_3 or 0.25 M Na_2CO_3 . Reference tests were also carried out without the biomass for each set of experimental conditions. The supernatant after separating the biomass by centrifugation was assayed for its activity. The distribution ratio was calculated using the formula,

$$D = (A_i - A_f) / A_f \cdot V / M$$

where A_i and A_f are the activities of the tracers in initial and final solutions respectively, V is the volume of the solution in ml and M is the weight of the biomass in gm.

3. RESULTS AND DISCUSSION

1. Volatile and Trace Metal Components

The percentage of volatile content present in stem as well as pseudostem calculated from the loss of weight in heating the biomass to constant weight at 90-100°C is presented in Table 1, which shows comparatively higher volatile content in stem portion than that present in pseudostem. The HNO₃ solution of the residue left after the incineration of banana stem/pseudostem at 800°C shows the presence of trace elements as given in Table 2. Here Ca and Mg are the major constituents that get accumulated in banana pith from nature.

2. Sorption of ²⁴¹Am

As most of the biosorbants are effective only in low acid aqueous solutions, the present studies have been restricted to the uptake of trace level metal ions relevant to Purex process in the acidic pH range. ²⁴¹Am tracer solution with 5573 Bq/ml alpha activity was prepared by diluting the stock solution and adjusting the pH to 2. About 200 mg of dried biomass was contacted with 25 ml of this solution in 100 ml shake flasks on a mechanical shaker at 25±1°C. A reference experiment was also conducted without biomass. The experiment lasted for about 10 days. During the experiment, ²⁴¹Am activity of the supernatant as well as that of the reference solution was monitored at regular intervals and the results are presented in Table 3. The uptake of ²⁴¹Am is observed to be fast initially and the rate of uptake decreases subsequently, and in about 8 days, removal of > 99% of the initial ²⁴¹Am alpha activity is observed. During this test, ²⁴¹Am alpha activity of the reference solution remains constant, thereby showing the integrity of the feed under experimental conditions. The supernatant of the test solution turned slightly yellow with small quantities of floating/suspended particles. All the three phases (the biomass, floating/suspended particles and the clear supernatant) were separated and assayed for their activity. 2 M HNO₃ was used to leach the activity from the banana pith and colloidal suspended portions. Table 4 shows that more than 98% uptake of the activity was found in the biomass

portion. Colloidal portion showed less than 2 % leaving behind only about 0.3 % of the initial ^{241}Am activity in the supernatant. The colloidal portion was checked for its solubility in 16.0 M and 8 M HNO_3 and found to be insoluble.

The dried biomass (banana pith) used in the present experiments was quite stable and could be used over a period of several months (3 months in the present instance). The biomass sorbents prepared from different sources of banana pith gave comparable D values for americium. These results indicate that banana pith from different sources can be collected, oven dried and used as a composite sorbent with long self life. This would facilitate its application on industrial scale.

3. Sorption of Various Radionuclides

Uptake of actinides ^{233}U , Pu (mainly 239) and ^{241}Am and fission products $^{152+154}\text{Eu}$ and ^{147}Pm at varying hydrogen ion concentrations (0.5 M HNO_3 to pH 6) are presented in Table 5. Maximum distribution ratio is observed at pH 6 for U, whereas for Am, Pu and fission products (Eu and Pm) the uptake is highest at pH 2. When tested at slightly higher acidities (0.1 and 0.5 M HNO_3) the uptake of Am decreases with increase in HNO_3 .

4. Sorption of ^{241}Am in presence of NaNO_3

As the waste solutions generated in nuclear fuel reprocessing are expected to contain NaNO_3 , the sorption of ^{241}Am was tested in presence of varying amounts of NaNO_3 (0 - 2.0 M). Results (Table 6) indicate practically no interference of salt upto 2 M concentration on the ^{241}Am uptake.

5. Sorption of ^{241}Am in presence of Nd(III)

To assess the impact of metal ion concentration on the uptake of Am(III) by banana pith, distribution ratio of Am(III) as a function of Nd(III) concentration in the feed was determined.

The results are given in Table 7., where an increase in D_{Am} is observed with the increase in the concentration of Nd(III). The reference experiments without biomass did not show any reduction in Am(III) content of the solution. In the range of Nd(III) concentration tested, there appears to be a salting out effect for Am(III) sorption by biomass.

6. Experiments with ^{241}Am spiked Evaporator Condensate Solutions

Condensate, a low level Purex waste effluent solution, after spiking with ^{241}Am activity was treated with the biomass, as such (0.14 M HNO_3) as well as at different pH (2, 4 and 6). ^{241}Am uptake is found to be very low at 0.14 M HNO_3 , which showed no improvements even after contacting for a longer time, where as at pH 2, 4, and 6, the uptake is substantial and showing steady increase with time of contact (Table 8). After a time interval of nearly 10 days, > 98% activity could be removed. These results are in agreement with the basic studies presented in Table 3.

7. Sorption of Radionuclides from diluted Purex HLW

The sorption behaviour of banana pith for various radionuclides present in a very dilute solution of Purex HLW, adjusted to pH 2, was studied. 25 ml of this solution was contacted for 3 Hrs. with a small lump (50 mg) of the biomass. The results of this experiments are presented in Table 9. In 3 hrs, > 65% of gross alpha activity is removed by the biomass. Sorption of gross beta and gamma activities is substantial with about 72 % ^{144}Ce and no ^{137}Cs uptake is observed.

8. Sorption of Non-radioactive Toxic Metal Ions

The results of the uptake of other toxic elements viz. Sr, Cr, Cd, As, Pb and Hg by the biomass are presented in Table 10. The results show practically no uptake of Cr and As. High uptake of

Sr, Cd, Pb and Hg are observed with a D values following the order $Hg > Sr > Cd > Pb$ at pH 2. These results are promising and the banana pith can thus be utilized to sorb some of these elements from conventional industrial effluents.

9. Capacity of Banana Pith

An attempt was made to determine the sorption capacity of the biomass banana pith for Nd(III) as a representative element of trivalent actinides and lanthanides. The biomass was contacted with Nd(III) solutions of various concentrations at pH 2 for 192 hours and the aqueous phase was analysed periodically to monitor the biomass sorption. The sorption capacity after 192 hours were calculated and the results are presented in Table 11. The sorption capacity appears to be a function of feed concentration. The table shows that at an equilibrium concentration of 4393 mg/l Nd(III), uptake is quite high and is in the range of 60 mg/g of banana pith. Though this does not appear to be the saturation capacity, the value was reached in about 48 hours and only a marginal enhancement was noticed on further contact, as evident from Fig. 1.

Further studies are being continued to assess the capacity of banana pith using column experiments. Efforts are also being made to prepare water-extracts of banana pith as they are expected to contain carboxylic acid based compounds which may be used for selective precipitation of toxic metal ions.

4. CONCLUSION

Preliminary studies indicate that the biomass banana pith can be utilized to sorb radioactive pollutants viz. ^{233}U , ^{239}Pu , ^{241}Am , ^{144}Ce , ^{147}Pm , and $^{152+154}\text{Eu}$ from aqueous solutions at acidic pH. Sorption is maximum at pH 2 for ^{241}Am , ^{144}Ce , ^{147}Pm , $^{152+154}\text{Eu}$ and tetravalent ^{239}Pu and at pH 6 for ^{233}U . The presence of NaNO_3 upto 2 M does not interfere with the uptake of ^{241}Am . The sorption capacity of banana pith for Nd(III) or trivalent actinide-lanthanide in general is

more than 60 mg/g of banana pith. The sorbed activity is recoverable in higher nitric acid (>0.5 M) solutions. This biomass can also be used to remove the toxic metals like Sr, Pb and Hg from waste effluents. Thus banana pith can be utilised as a sorbent for treatment of waste solutions from industries including nuclear industry.

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Table 1. Analysis of Volatile Contents in Banana Tissue

Plant portion	Wet weight (g)	Constant weight after drying at 90-100°C (g)	% Volatile content
Stem	(a) 10.222	0.4442	95.65
	(b) 10.380	0.4670	95.50
Pseudostem	(a) 2.3067	0.2996	87.01
	(b) 5.2480	0.6900	86.85

Table 2. Trace Metal Analysis of Banana Tissue

Elements	Stem Sample 1 (µg/g)	Stem Sample 2 (µg/g)	Pseudostem (µg/g)
Ca	1539.4	1120.0	2690.0
Al	55.6	60.0	70.0
B	10.6	--	--
Cu	8.6	--	--
Fe	135.7	140.0	120.0
Mg	1767.0	>3000	>3000
Pb	105.9	20.0	50.0
Si	231.6	280.0	370.0
Ti	7.3	--	--
Ni	7.9	--	--
Na	--	--	700.0
Mo	--	800.0	980.0
Zn	--	40.0	20.0
Mn	--	12.0	20.0

-- Not Detected

Table 3. Sorption of ^{241}Am by Dry Banana Pith

Experimental Conditions:

Feed Volume : 25 ml, ^{241}Am alpha activity : 5573 Bq/ml.
 pH : 2, Weight of banana pith : 0.1983 g (lump)

Time (Hrs.)	^{241}Am activity reference (Bq/ml)	^{241}Am activity in supernatant (Bq/ml)	D_{Am}	Sorption (%)
0	5573	5573	-	-
3	5570	2425	164	56.49
45	5575	1760	273	68.42
51	5573	1661	297	70.20
71	5580	1131	495	79.71
143	5569	184	3687	96.70
167	5575	82	8485	98.53
190	5578	64	10807	98.85
214	5570	39	17921	99.30
238	5574	18	39797	99.68

Table 4. Material Accounting in ^{241}Am Sorption on Banana Pith

Feed : 5573 Bq/ml ^{241}Am (alpha), 25 ml at pH 2,
 Biomass : 0.1983 g (lump), Contact time ; 238 Hrs.,
 Leach agent : 2 M HNO_3

Sample	Volume (ml)	Am alpha activity (Bq)	% ^{241}Am
Feed	25	139325	100.00
Supernatant	25	425	0.31
Colloid leach	2	1818	1.31
Biomass lump leach	5	136675	98.10

Table 5. Uptake of Various Radionuclides at Different pH

Experimental Conditions :

Volume : 5.0 ml, Weight of the biomass : 50 mg,

Mesh size : 30-50, Time of Contact : 24 Hrs.

Feed activity :

²⁴¹Am (alpha) : 4888 Bq/ml,

¹⁴⁷Pm (beta) : 6900 Bq/ml,

¹⁵²⁺¹⁵⁴Eu (gamma) : 20425 Bq/ml,

²³³U (alpha) : 4176 Bq/ml,

²³⁹Pu (alpha) : 986 Bq/ml,

HNO ₃ /pH	D _{Am}	D _{Pm}	D _{Eu}	D _{Pu}	D _U
0.5 M	10.6	NA	NA	NA	NA
0.1 M	38.2	76.3	NA	NA	NA
PH = 2	278.0	909.3	1187.4	294	34.1
PH = 4	191.9	601.1	237.0	146	149.2
PH = 6	170.4	472.1	119.1	1.7	322.0

NA = Not Analysed

Table 6. ²⁴¹Am Uptake in Presence of NaNO₃

Experimental Conditions :

Feed volume : 5 ml, pH : 2, Weight of biomass : 50 mg,

Mesh size : 30-50, Contact time : 24 Hrs.

²⁴¹Am alpha activity in feed : 5749 Bq/ml.

NaNO ₃ Conc. (M)	Supernatant activity (Bq/ml)	Sorption (%)	D _{Am}
0	1664	71.06	245.5
0.15	1808	68.55	218.0
0.25	1670	70.95	244.3
0.50	1663	71.07	245.7
1.00	1603	72.12	258.6

Table 7. Uptake of ²⁴¹Am in Presence of Nd(III)

Experimental Conditions :

Volume : 5 ml, pH : 2, Wieght of biomass : 50 mg,

Mesh size: 30-50, Contact time : 24 Hrs.

²⁴¹Am alpha activity in the feed : 29125 Bq/ml

Nd(III) Conc. (mg/l)	Supernatant activity (Bq/ml)	Sorption (%)	D _{Am}
0	8149	72.02	257.4
5	8070	72.29	260.9
100	7307	74.91	298.6
200	3604	87.63	708.1
500	2912	90.00	900.2

Table 8. Sorption of ²⁴¹Am spiked Purex Evaporator Condensate

Experimental Conditions:

Volume : 100 ml, Weight of dry banana lump : 1.0 gm.

²⁴¹Am Activity (alpha) spiked : 6206 Bq/ml

Time (hrs)	Percentage Sorption of ²⁴¹ Am at			
	0.14 M	(pH=2)	(pH=4)	(pH=6)
1	8.86	41.17	23.50	20.82
24	13.99	54.16	45.20	27.54
48	13.95	59.48	49.41	32.64
72	13.06	68.94	64.24	56.70
96	13.75	72.79	68.84	67.78
120	13.06	80.68	85.59	82.18
144	13.40	96.49	93.14	92.44
170	13.87	97.60	94.84	93.22
188	13.16	98.02	96.31	94.47
220	13.57	99.04	97.78	96.39
250	13.61	99.8	98.48	97.87

Table 9. Sorption of Activity from Diluted Purex HLW

Experimental Conditions :

Feed : Diluted Purex HLW, Volume : 25 ml, pH : 2,
Weight of biomass : 50 mg (lump), Contact time : 3 Hrs.

Constituents	Feed activity (Bq/ml)	Effluent activity(Bq/ml)	Sorption (%)
Gross alpha	4.33×10^3	1.50×10^3	65.36
Gross beta	1.62×10^7	1.12×10^7	30.86
Gross gamma	2.75×10^6	1.39×10^6	49.45
^{144}Ce	2.55×10^3	6.94×10^4	72.78
^{137}Cs	2.15×10^6	2.15×10^6	Nil

Table 10. Uptake of Toxic Elements using Banana Tissue

Experimental Conditions :

Volume . 5.0 ml, Time of Contact : 24 Hrs.
Weight of the biomass : 250 mg piece
Feed concentration : 25 ppm each of metal ion.

Elements	at pH 2		at pH 4	
	D	E(%)	D	E(%)
Sr^{2+}	249.0	92.6	275.0	93.2
Cr^{3+}	0.5	2.4	Nil	Nil
Cd^{2+}	4.7	19.0	5.9	22.6
As^{3+}	Nil	Nil	Nil	Nil
Pb^{2+}	164.0	89.2	109.0	84.5
Hg^{2+}	447.0	95.7	200.7	90.9

Table 11. Neodymium uptake Capacity of Banana Pith

Experimental Conditions :

Feed Volume : 5.0 ml, pH : 2, Contact time : 192 Hrs.
Weight of Biomass : 50 mg (lump)

S.No.	Concentration of Nd(III) in feed (mg/l)	Equilibrium Concentration of Nd(III) (mg/l)	Sorption Capacity mg Nd /g biomass
1.	750.0	504.7	24.0
2.	1000.0	714.7	28.6
3.	2000.0	1698.3	30.2
4.	5000.0	4393.0	60.7

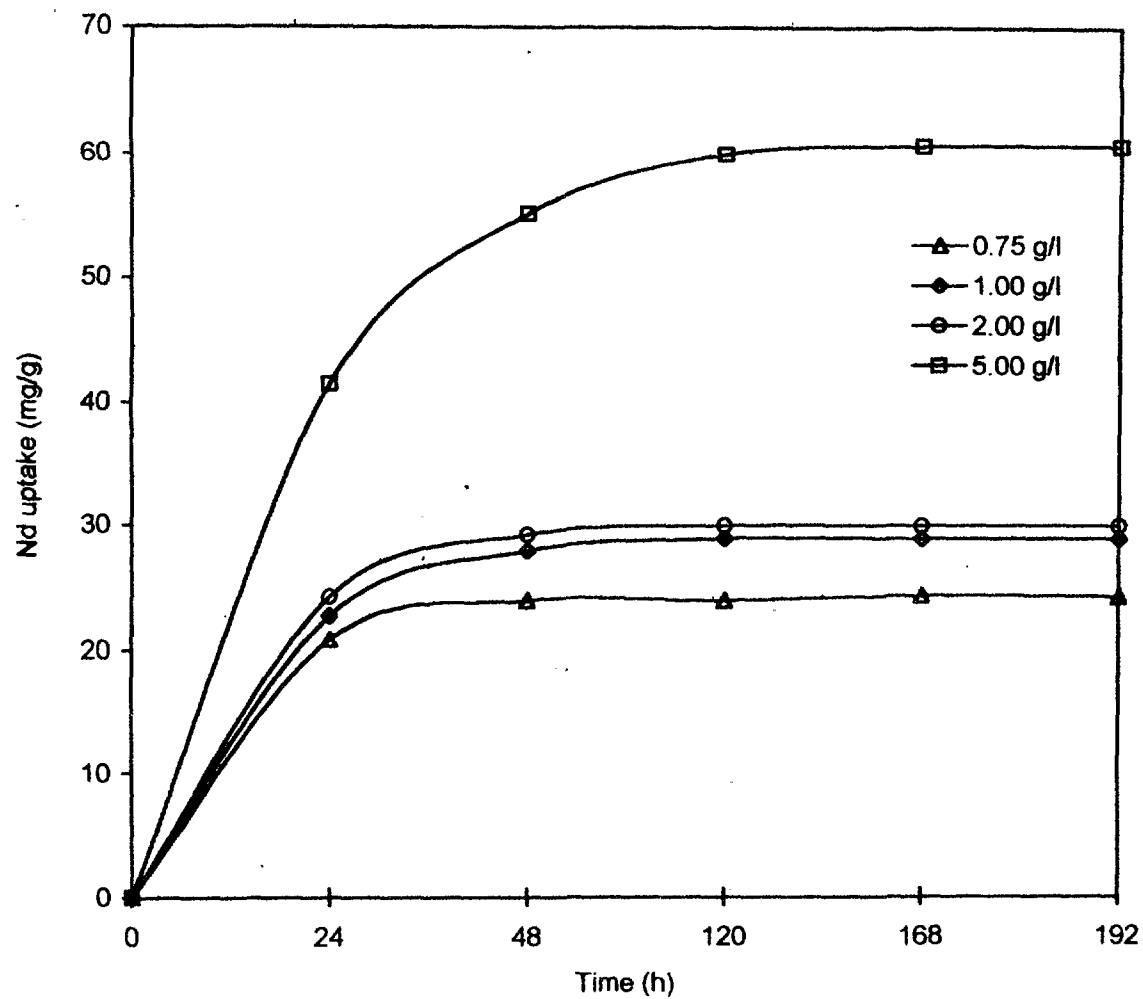


Fig.1. Sorption of Nd(III) as a Function of Time

