**Original Research Article****DOI - 10.26479/2018.0403.11****SYNTHESIS OF A NEW HYBRID SORBENT 5-AMINO-1,3,4-THIADIAZOLE-2-THIOL FUNTIONALIZED SBA-15 AND ITS APPLICATIONS IN SOLID PHASE EXTRACTION OF FE(III) FROM WATER SAMPLES****K. Imran¹, B. Ramesh Naik¹, N. Satish Kumar¹, K. Sessaiah^{1*}, K. Ramanjaneyulu²****1.**Inorganic and Analytical Chemistry Division, Department of Chemistry,

Sri Venkateswara University, Tirupati, India.

2.First year Engineering Department, Sir C.R. Reddy college of Engineering, Eluru-5534007.

ABSTRACT: A mesoporous hybrid material, 5-Amino-1,3,4-Thiadiazole-2-Thiol-SBA-15 (SBA-15-ATT) was synthesized by anchoring 5-Amino-1,3,4-Thiadiazole-2-Thiol (ATT) on to the surface of SBA-15. The prepared sorbent was characterized by using Fourier-transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), X-ray diffraction (XRD) and Nitrogen adsorption-desorption isotherm. The sorbent, SBA-15-ATT was applied for the separation and preconcentration of Fe(III) in water samples. The maximum preconcentration capacity ($0.82 \pm 0.01 \text{ mmol g}^{-1}$) for Fe(III) was observed at pH=9. Fe(III) collected onto the sorbent was eluted out with 10 mL of 1 M HNO₃ and the concentration of Fe(III) in eluent was determined by flame atomic absorption spectrometry. This has resulted a preconcentration factor of 50. From the results it was concluded that the presented method is very much suitable for the determination of Fe(III) ions in water samples with LOD of $0.32 \mu\text{g L}^{-1}$.

KEYWORDS: SBA-15, Fe(III), SBA-15-ATT, Preconcentration.

***Corresponding Author: Dr. K. Sessaiah Ph.D.**

Inorganic and Analytical Chemistry Division, Department of Chemistry,

Sri Venkateswara University, Tirupati, India.

*Email Address: sessaiahsvu@gmail.com

1.INTRODUCTION

In recent years, environmental pollution is an undoubted growing global problem that should needs to be controlled. Numerous toxic chemicals are discharged into the environment from various sources of industries. The released contaminants from the industries mainly contaminating the air, soil, water and other constituents of environment. Among the released pollutants, heavy metal ions stand in the major position in causing several problems to the mankind. Heavy metal ions come from different sources like chemical manufacturing, mining, extractive metallurgy, nuclear painting and coating, and other industries [1]. The metal ions such as Fe(III), Cr(III), Pb(II), Cd(II), Cu(II), Co(II), Ni(II), Zn(II), Hg(II) in water samples has received great attention over the past few years and increasing their effect day by day to show great negative impact on our living systems [2]. Among the toxic metal ions, Fe(III) plays an important role in causing major problems to the environment which creates major impact on human health. Iron is an essential element for the living systems but its deficiency leads to cause several health problems in the living organisms. The deficiency of Iron in blood causes Anemia in human beings whereas the high concentration of iron in blood causes a variety of health problems like dysfunction of endocrine, heart diseases, liver diseases and diabetes [3]. So it is essential to preconcentrate the Fe(III) metal ions from the water samples before going to discharge into the natural waterbodies. Several techniques were used to preconcentrate the heavy metal ions from waste water includes liquid-liquid extraction, cloud point extraction, ion exchange, co-precipitation and solid phase extraction (SPE). Most of the techniques were failed in the analysis of the heavy metals in their ultra-trace levels. Among the above said procedures, the most commonly used method to preconcentrate the heavy metal ions is solid phase extraction. The solid phase extraction technique has a main advantage in which preconcentration of metal ions from water samples from larger volumes to minimum levels with elimination of matrix interference [4-5]. In SPE, the sorbent should be insoluble material along with anchored organic functional groups which chelate metal ions, having good sorption, rapid phase separation and regenerability for better research. Some of the sorbents which are using in preconcentration of heavy metal ions are activated carbon nanotubes, resins, modified silica gel, polytetrafluorethylene beads and turnings and polyurethane foam [6-7]. In SPE, choosing of sorbent plays an important role on the effect of preconcentration of trace metal ions from the waste water. Thus, there is a need in the development of a sorbent material with high stability, high extraction efficiency and recovery of sorbent. For the last few years, analysts made an attempt on the mesoporous materials like MCM-41, MCM-48, MCM-50 and SBA-15 which are having unidimensional hexagonal, cubic, lamellar, 2D hexagonal meso structures respectively. Among the mesoporous materials SBA-15 was applied in various fields of water purification, catalysis, and separations. SBA-15 has a two-dimensional hexagonal arrangement having uniform, large, thicker pore walls which is a major advantage in the

preconcentration of trace metal ions [8-9]. In the present work, the development of mesoporous sorbent material was studied and it was used as a sorbent in the SPE method for the preconcentration of trace elements in environmental samples. The surface of the SBA-15 mesoporous silica was modified by anchoring 5-Amino-1,3,4-Thiadiazole-2-Thiol and the prepared sorbent was named as SBA-15-ATT. The functionalized sorbent was characterized by Fourier-transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), X-ray diffraction (XRD) and Nitrogen adsorption-desorption isotherm. Application of the prepared material as a sorbent for the preconcentration of Fe(III) in water samples prior to the determination by flame atomic absorption spectrometry was investigated.

2. MATERIALS AND METHODS

2.1 Chemicals

The chemicals which are used in the present study were of Analytical grade. Doubled distilled water was used for the solubility and dilutions. Poly (ethylene glycol) block-poly (propylene glycol) block-poly (ethylene glycol) (pluronic P123 $\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$, $M_w=5800$), TEOS (tetraethylorthosilicate), 3-chloropropyltrimethoxy silane, 5-Amino-1,3,4-Thiadiazole-2-Thioethyltri-amine and Iron nitrate nonahydrate were supplied by Sigma Aldrich. Hydrochloric acid and Nitric acid were purchased from Merck life sciences Pvt.Ltd, Mumbai. Acetic acid was purchased from Thermo Fisher Scientific India Pvt. Ltd, Mumbai. Sodium hydroxide was supplied from Merck life sciences Pvt.Ltd, Mumbai.

2.2 Synthesis of SBA-15

SBA-15 was synthesized by taking 3.5 g of P-123 copolymer dispersed in distilled water and stirred for 2 hours at room temperature. To the resultant solution, 120 g of 2M HCl was added with continuously stirring for another 2 hours. Finally, Tetraethylorthosilicate was added and subjected to hydrothermal treatment for 48h under static conditions. The mixture was filtered, washed double distilled water and dried in hot air oven at 70°C for 12 hours. The resulted mixture was then calcined at 560 °C for 7 hours in air for the removal of template completely.

2.3 Synthesis of mesoporous silica SBA-15 5-Amino-1,3,4-Thiadiazole 2-thiol derivative

Initially SBA-15-Cl was prepared by taking 2.0 g of SBA-15 in 40 ml of toluene and 0.5 g of 3-chloropropyl trimethoxy silane was added to it with stirring the reaction mixture at 80 °C for 24 h in inert nitrogen atmosphere. The formed solid SBA-15-Cl was filtered, washed with dichloromethane (DCM) and dried at 60 °C for 12 h in hot air oven. 5-Amino-1,3,4-Thiadiazole 2-Thiol was taken into the mixture of dry ethanol and tri methyl amine (Et_3N). Then SBA-15-Cl was added and refluxed for 24 h in inert N_2 atmosphere. The final product, SBA-15-5-Amino-1,3,4-Thiadiazole 2-Thiol (SBA-15-ATT) was collected after the removal of solvent through filtration and drying in hot air oven at 60 °C for 12 h. The schematic mechanism of preparation of SBA-15-ATT

is shown in fig 1

2.4 Instrumentation

Powder X-ray diffractograms (XRD) of materials were recorded using a PANalytical X'pert Pro dual goniometer diffractometer. The radiation was used $\text{Cu K}\alpha$ (1.5418 \AA) with a Ni filter and the data collection was carried out using a flat holder in Bragg-Brentano geometry ($0.5\text{--}5^\circ$; $0.2^\circ \text{ min}^{-1}$). Scanning electron microscope (Model: EVO ma 15 manufactured by Carl Zeiss) was used to study the morphological features of the sorbents. For HRTEM analysis, the samples were subjected to JEOL JEM-3010, Tecnai (Model F30) microscopes operating at 300 kV. N_2 adsorption-desorption isotherms, pore size distributions were determined by using a Micrometrics ASAP 2020 instrument Autosorb 1C Quanta chrome USA. Infrared Spectrometer (Model: Bruker Alpha FT-IR spectrometer) was used for the identification of functional groups on the surface of the sorbents. The spectrum was recorded in the range of $4000 - 400 \text{ cm}^{-1}$. An Elico pH meter (Model: LI-120, India) with combined glass electrode was used for measurements of pH. The pH meter was calibrated using standard buffer solutions of pH 4.0, 7.0 and 9.2. Flame atomic absorption spectrophotometer (Model: Shimadzu AA-6300, Japan) with single element hollow cathode lamps of respective elements operating with an air-acetylene flame, was used to analyze the concentrations of heavy metal ions.

2.5 Determination of metal ions by solid phase extraction procedure.

A column process was employed to study the ability of SBA-15-ATT material as sorbent in preconcentration of Fe(III) from aqueous solutions. A glass column of $10\text{cm} \times 1\text{cm}$ dimensions was filled with 0.2 g of SBA-15-ATT. The aqueous solution of Fe(III) was prepared by adjusting the pH to 9.0. The prepared aqueous solution of Fe(III) was passed through the column at a flow rate of 3.5 mL/min. Then, the column was washed with 20 mL of Milli-Q water and 10 mL of 1M nitric acid was passed through the column to desorb the bound Fe(III) ions into the solution. The concentration of Fe(III) ions present in the eluent was determined by flame atomic absorbance spectroscopy. The same procedure was repeated about 6 times by using suitable blank solutions.

2.6 Flow rate effect on sorbent

500 mL of $10 \text{ }\mu\text{g/mL}$ aqueous solution containing Fe(III) ions was taken in a separate beaker and adjusted the pH to 9.0 by using dilute HCl and dilute NaOH. Accurately weighed 0.2 g of dried SBA-15-ATT sorbent was filled in glass column of $10\text{cm} \times 1\text{cm}$ dimensions and passed the aqueous solution at different flow rates of 0.5 mL/min, 1 mL/min, 1.5 mL/min, 2 mL/min, 2.5 mL/min, 3 mL/min, 3.5 mL/min and 4 mL/min (20 - 160 rpm) with a peristaltic pump. The column was washed with 20 mL of Milli-Q Water for washing the remaining free metal ions present in the column. Then, 10 mL of 1M nitric acid was passed through the column to desorb the chelated metal ions with ligands in the SBA-15-ATT sorbent.

2.7 Maximum adsorption capacity of the sorbent.

0.2 g of SBA-15-ATT sorbent filled in glass column 10cm×1cm dimensions and 500 mL of 10µg/mL Fe(III)solution adjusted to pH 9.0 was passed through the column at a flow rate of 10 mL/min with a peristaltic pump. Washed the column with 20mL of Milli-Q water. Finally, to desorb the bound Fe(III)ions, 10mL of 1M nitric acid solution was used and resultant solution was determined by AAS. The maximum amount Fe(III)was taken up by the SBA-15-ATT ($N_{f\max}$) was calculated by the following equation:

$$N_{f\max} = \frac{n_r}{m}$$

Where n_r is the amount of Fe(III)retained in the SBA-15-ATT after the equilibrium and m is the mass of sorbent (grams).

2.8 Determination of volume breakthrough

0.2 g of SBA-15-ATT sorbent filled in glass column having 10cm×1cm dimensions. 50, 100, 200, 300, 400 and 500 mL of 10µg of Fe(III)solutions adjusted to pH 9.0 was passed through the column at a flow rate of 3.5 mL/min with a peristaltic pump. Finally, to desorb the bound Fe(III)ions, by using 10mL of 1M nitric acid solution and eluent in the nitric acid solution was determined by flame atomic absorption spectroscopy. Up to 500 mL the recovery percent of Fe(III)was found to be ≥ 96.5 . From this procedure the preconcentration factor was calculated to be 50.

2.9 Reusability of the material

0.2 g of SBA-15-ATT sorbent filled in glass column having 10cm×1cmdimensions and 500mL of 10µg/mL of Fe(III)solution adjusted to pH 9.0 was passed through the column at a flow rate of 3.5mL/min with a peristaltic pump. To regain the sorbent, wash the column with 10mL of 1M HNO₃ solution followed by 20mL of Milli-Q water. The concentration of Fe(III)in the eluated solution was determined by Flame atomic absorption spectrophotometer. The sorption/desorption process was Repeated up to six cycles.

3. RESULTS AND DISCUSSION

3.1 IR Spectra

FTIR spectrometry was used to identify the functional groups which are present on the surface of the sorbent. The IR spectra of SBA-15-Cl was shown in Fig 2. The IR spectra of SBA-15-Cl shows asymmetric and symmetric stretching vibrations of Si-O-Si were at 1071cm⁻¹ and 801 cm⁻¹ respectively. A broad peak at 3410 cm⁻¹ indicating the free -OH stretching vibrations and a peak at 1640 cm⁻¹represents the free -OH bending vibrations [10]. From Fig 2A, the peaks at 2934 cm⁻¹ and 740 cm⁻¹ indicates the C-H and C-Cl stretching vibrations respectively. From Fig 2Bit is clear that the absence of C-Cl stretching peak in modified SBA-15-ATT confirms the thiol substitution in the

place of Cl group. In the modified SBA-15-ATT spectra, stretching frequencies at 3264 cm^{-1} and 3349 cm^{-1} which represents the amine (NH_2) groups remains same in the position. So that it was concluded that the amine group was not involved in the bonding with SBA-15-Cl. Finally, the absence of S-H stretching peaks confirms the modification of SBA-15-Cl. From Fig 2D it was clear that the shifting amine stretching peaks from 3264 cm^{-1} and 3349 cm^{-1} to 3334 cm^{-1} and 3253 cm^{-1} respectively confirms the binding of metal ions with the sorbent.

3.2 XRD,

X-ray diffraction helps in the determination of the nature of sorbent whether it is crystalline or amorphous. The XRD patterns of the sorbent before and after modification is shown in Fig. 3. From the figure, the peaks represent the amorphous nature of the sorbent. Fig 3a shows the diffraction peaks of SBA-15-Cl and Fig 3b shows the diffraction peaks of SBA-15-ATT. From the diffraction patterns it was clear that, the reflection peaks for both SBA-15-Cl and SBA-15-ATT was appeared at 2θ of (100) (110) and (200) which shows that the hexagonal structures of SBA-15-Cl was retained in SBA-15-ATT after modification. The peak intensity of SBA-15-ATT was decreased when compared with SBA-15-Cl is due to the fact that, 5-Amino-1,3,4-Thiadiazole-2-Thiol group anchored on to the mesoporous sorbent which leads to the decrease of scattering power of the mesoporous material.

SEM, TEM

Scanning electron microscopy and Transmission electron microscopy was used to determine the morphological features of the sorbent. The SEM images of SBA-15-Cl and SBA-15-ATT was depicted in Fig. 4. The images in Fig. 4 shows the well distributed 2D-hexagonal structures arranged in the form of rope-like sheets which confirms that the hexagonal structures of SBA-15-Cl was retained in SBA-15-ATT after modification. Fig 5 shows the TEM images of unmodified and modified mesoporous material in which it was observed that the presence of ordered hexagonal patterns, cylindrical and nano tubular pore structures. The TEM studies of SBA-15-Cl and SBA-15-ATT clearly indicates that the pore structures are not disrupted after anchoring of functional groups on to the sorbent which is an advantageous property of the sorbent in the sorption of Fe (III) ions from the aqueous solution [11-13].

Nitrogen adsorption-desorption isotherms Analysis

The nitrogen adsorption-desorption isotherms of SBA-15 having the average ranges of surface area, pore size and pore volume are $630\text{--}1040\text{ m}^2/\text{g}$, $47\text{--}100\text{ \AA}$ and $0.56\text{--}1.26\text{ cm}^3/\text{g}$ respectively [14]. The nitrogen adsorption-desorption isotherms of SBA-15-Cl and SBA-15-ATT was shown in Fig. 6. It is an evident from the Fig.6 that the mesoporous structures were not disturbed even after surface modification and displayed characteristic type IV isotherms (hysteresis). The surface area, pore size and pore volume have been calculated by BET and BJH methods and are summarized in Table 1.

From the results which are presented in Table 1, the surface area, pore size and pore volumes of SBA-15-Cl and SBA-15-ATT were decreased on surface modification of SBA-15.

3.3 Effect of pH on the sorbent

pH of the solution plays a key role in binding of metal ions on to the mesoporous sorbent. The effect of pH on the recovery of Fe(III) ions by SBA-15-ATT was investigated by taking different pH values ranges from 4.0 to 12.0 with an initial concentration of 10 mg/L of Fe(III) aqueous solution and it was shown in Fig.7. From the Figure the percentage recovery of Fe(III) was increased with increase in solution pH from 4.0 to 9.0. The maximum recovery of Fe(III) on to the sorbent SBA-15-ATT was found at 9.0. The reason behind in the increase of percentage recovery of Fe(III) by increase in the pH is due to the presence of more number of H^+ ions at low pH values. At low pH values, H^+ ions can occupy the ligand sites which creates a competition between the H^+ ions and the incoming metal ions and hence the sorption capacity of sorbent is low at low pH values. By changing the pH of the solution from 4.0 to 9.0 there is a chance in the decrease of competition between H^+ and incoming metal ions which can help in the anchoring of metal ions to the binding sites of the sorbent [15-16]. Hence the percentage recovery of Fe(III) ions increases from low pH values and reaches to the maximum level at pH 9.0. This point is said to be the optimum level. Further increase in the pH of the solution after the optimum level leads to the decrease in the percentage removal of Fe (III). This is due to the formation of metal hydroxides at high pH values.

3.5 Sorption mechanism

Fe(III) is a hard acid that can bind with the hard bases like amine ligands. So from the IR spectra, there is a slight shift in the $-NH_2$ stretching peaks which is due to the chelation of Fe(III) with SBA-15-ATT at pH 9.

3.6 Sample flow rate effect on sorption of Fe(III) onto the SBA-15-ATT

Column technique is one of the common method for the extraction of metal ions from huge water samples. In this technique, retention of metal ions from packed column is depend on the interaction of metal ions with binding sites of sorbent. The flow rate was examined by passing aqueous solution through column with the help of peristaltic pump. The flow rates of aqueous solution varied between 0.5 mL min^{-1} to 4 mL min^{-1} . The effect of flow rates with respect to the recovery of Fe(III) is shown in Table 2. From the table, the highest recovery of Fe(III) (>96%) was achieved at the flow rate of 3.5 mL min^{-1} . So the flow rate of 3.5 mL min^{-1} has taken as an optimum level and used the same in the further experiments to get better results. So the optimum flow rate confirms a good interaction of Fe(III) with binding sites of SBA-15-ATT. 10 mL of 1M HNO_3 solution is good enough to show good stability and elution towards the Fe(III) chelated SBA-15-ATT complex.

3.7 Interaction of SBA-15-ATT with potentially interfering ions.

Many ions such as Li^+ , Na^+ , K^+ , Mg^{+2} , Ca^{+2} , Al^{+3} , Zn^{+2} , Mn^{+2} , Ni^{+2} , Cl^- , PO_4^{-2} , CO_3^{-2} , SO_4^{-2} and CH_3COO^- are responsible to interfere in the determination of Fe(III) ions in water samples. For this preconcentration of Fe(III) under optimized conditions have been studied. In Table 5, the sorption of Fe(III) in various interfering species and their tolerance limits were presented and the error was not more than the 5%. The binding capacity of Fe(III) with respect to the sorbent was increased by modifying the SBA-15-Cl. In order to determine maximum adsorption capacity (N_{fmax}), it is necessary to calculate how much sorbent used to recover a specific trace metal ion from aqueous samples. From the results, the maximum adsorption capacity (N_{fmax}) of Fe(III) onto the SBA-15-ATT was calculated as $N_{\text{fmax}} = 0.82 \pm 0.01 \text{ mmol g}^{-1}$.

3.8 Stripping of Fe(III) from SBA-15-ATT

The volume and type of eluent for stripping of metal ions can decide the preconcentration factor. The chelated SBA-15-ATT bound metal ions were stripped off by using different acids like dilute HCl, dilute CH_3COOH and dilute HNO_3 . The results are presented in the Table 3. From the table it is clear that, 10 mL of 1M HNO_3 can recover more than the 96% of Fe(III) ions. So HCl was used as eluent for stripping of Fe(III) from SBA-15-ATT. The stability and regeneration of SBA-15-ATT was investigated by exchange capacity of the Fe(III) ions and it was determined after several cycles of sorption and desorption process. For the first, second, third and fourth cycles the recovery percentage of Fe(III) was found to be more than or equal to 96%, For the fifth and so on found to be 92%. Results indicated that up to 4 cycles SBA-15-ATT show good response and decreases slowly with multiple cycles.

4. CONCLUSION

The analytical applications of a new hybrid sorbent 5-Amino-1,3,4-Thiadiazole-2-Thiol functionalized SBA-15 has showed effective separation and preconcentration of Fe(III) from environmental samples. This method gives the preconcentration factor of 50 and showed low limit of detection as compared with other procedures as listed in the table 7. The effect of matrix on recovery and determination of Fe(III) from environmental samples with proposed method was negligible. Sorption and desorption studies revealed that SBA-15-ATT can be regenerated by treatment with low volume of HNO_3 and can be reused up to for 5 cycles.

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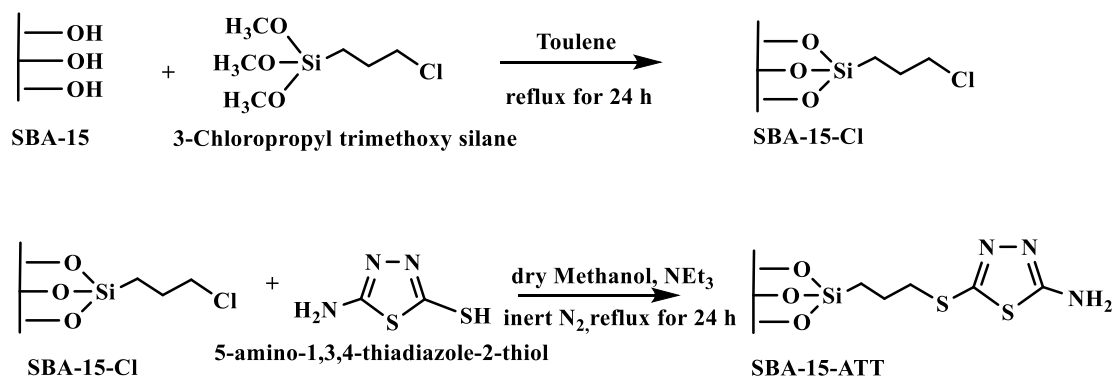


Fig.1 The schematic mechanism of preparation of SBA-15-ATT

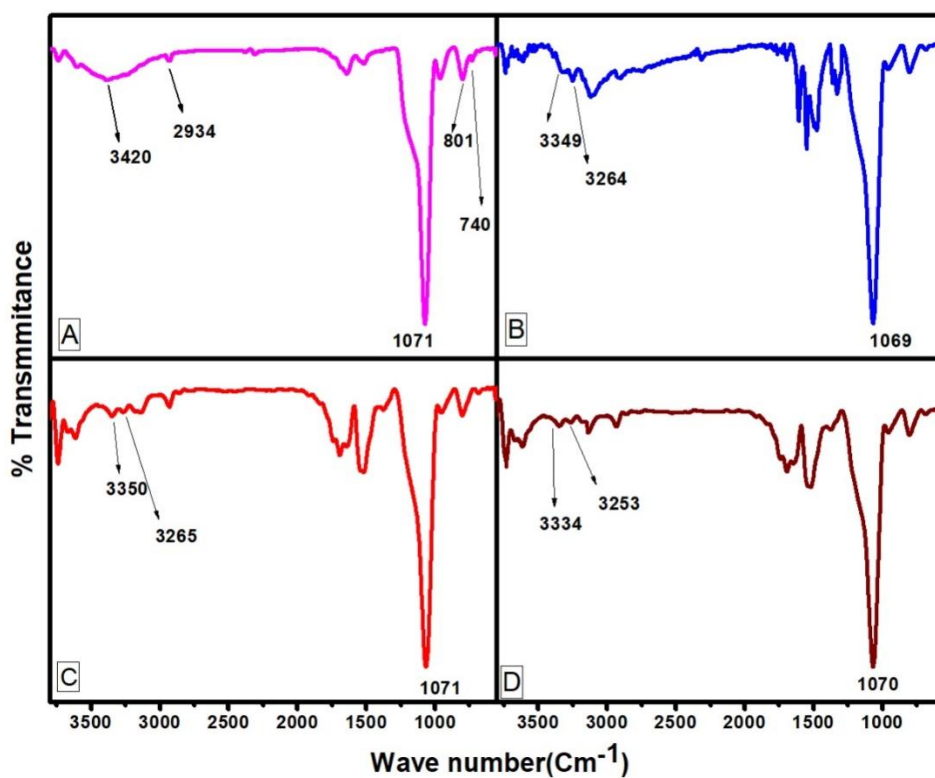


Fig. 2A) SBA-15-Cl, 2B) SBA-15-ATT, 2C) SBA-15-ATT- 2D) SBA-15-ATT-Fe

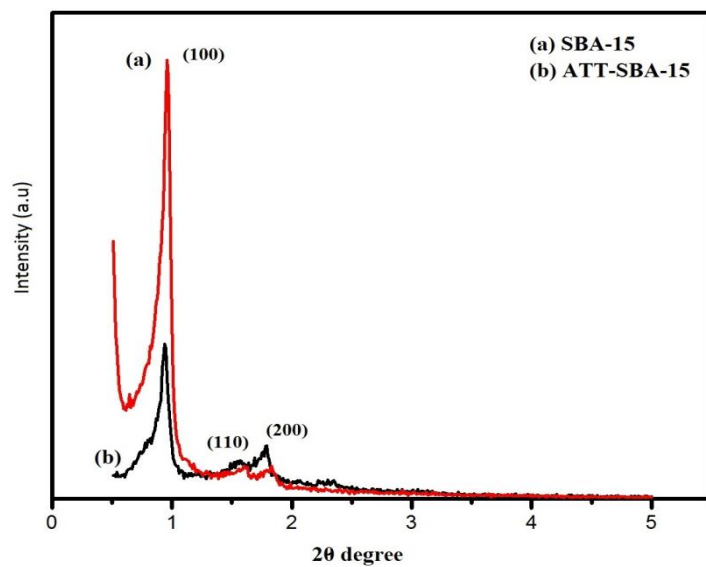


Fig. 3 a) SBA-15, 3b) SBA-15-ATT

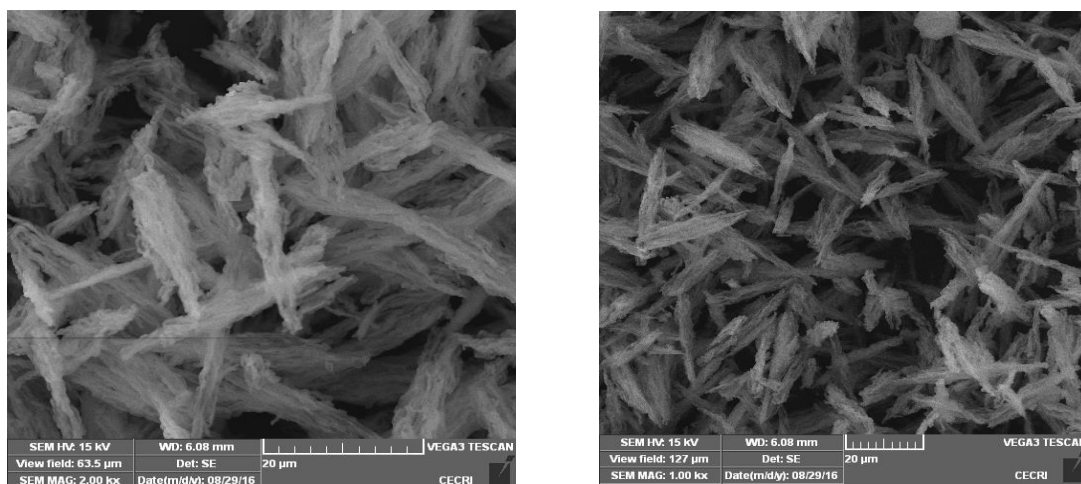


Fig. 4 a) sem image of SBA-15-Cl 4b) sem image of SBA-15-ATT

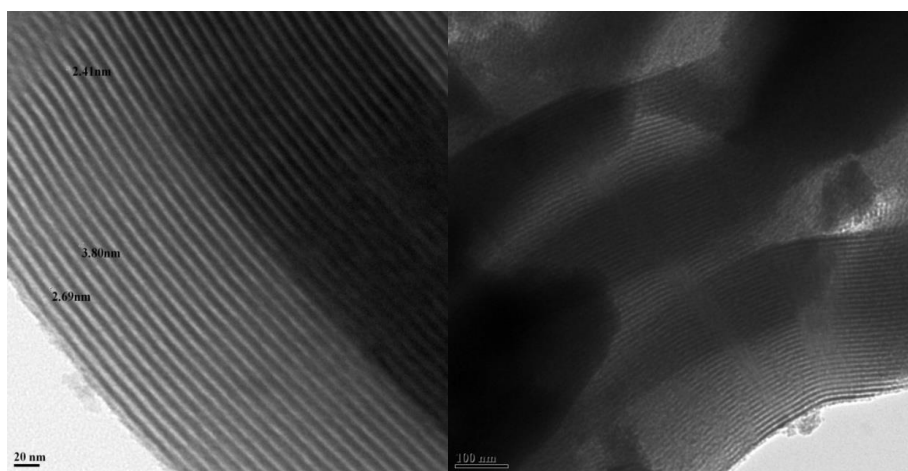


Fig. 5 a) TEM images of SBA-15-Cl, 5b) TEM image of SBA-15-ATT

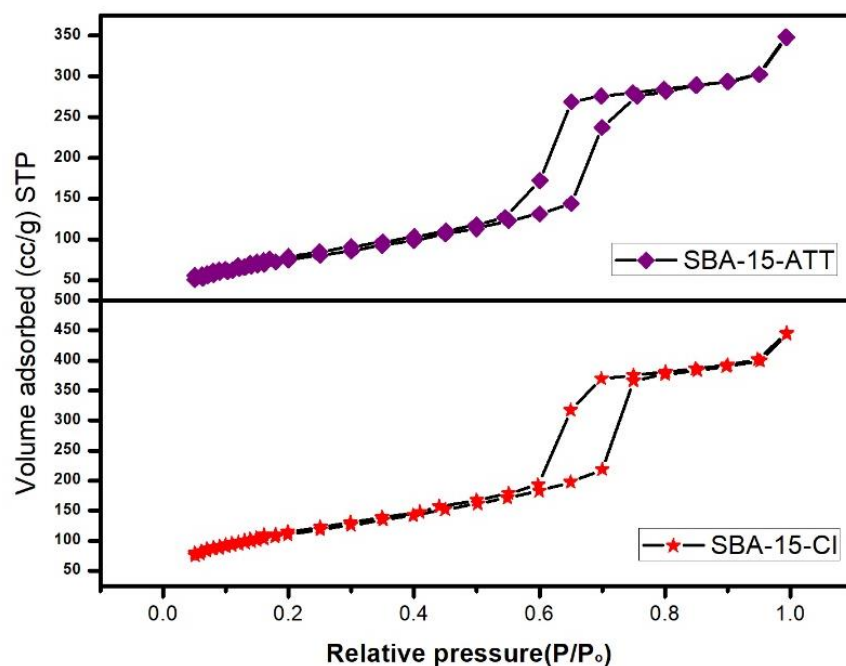


Fig.6a) Nitrogen adsorption-desorption isotherm images of SBA-15-Cl, 6b) Nitrogen adsorption-desorption isotherm image of SBA-15-ATT

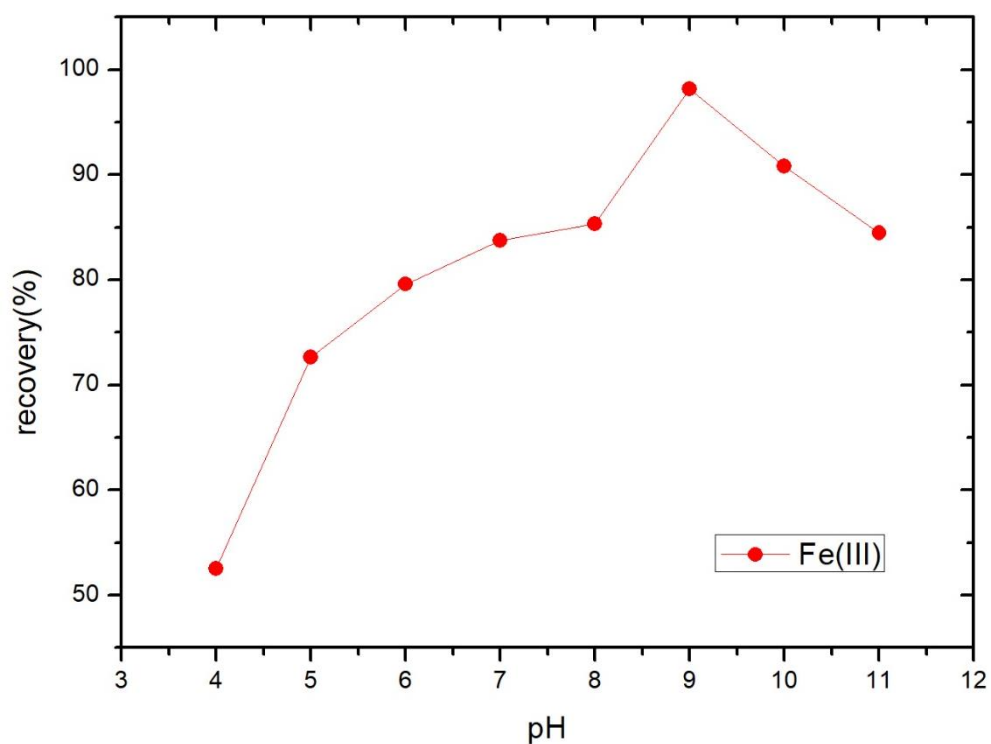


Fig.7 effect of pH on Fe(III) recovery

Table 1 Surface area, Pore volume and Pore radius of SBA-15-Cl and SBA-15-ATT.

Materials	Surface area	Pore volume	Pore radius
SBA-15-Cl	598.758 m ² /g	1.063 cc/g	31.534 Å
SBA-15-ATT	393.132 m ² /g	0.829 cc/g	27.152 Å

Table 2. Effect of the flow rates in the Fe(III) recovery.

Flow rate (mL/min)	% Recovery of Fe(III) ^a
0.5	93.2±1.40
1.0	90.9±0.96
1.5	92.7±1.95
2.0	92.2±1.60
2.5	92.6±0.91
3.0	94.1±0.64
3.5	92.8±0.97
4.0	91.1±1.03

^aAverage of four determinations±standard deviation.

Table 3 stripping of Fe(III) by eluent

Type of eluent (1M)	Volume(mL)	% Recovery of Fe(III)
HCl	5	63.5(1.0)
HCl	10	68.4(0.6)
HCl	15	71.5(1.0)
HCl	20	72.5(0.7)
HNO ₃	5	92.9(0.6)
HNO ₃	10	96.3(0.8)
HNO ₃	15	96.4(0.4)
HNO ₃	20	95.5(0.8)
CH ₃ COOH	5	64.5(1.0)
CH ₃ COOH	10	69.8(0.5)
CH ₃ COOH	15	72.3(0.4)
CH ₃ COOH	20	72.1(0.7)

^a % RSD based on three repetitions

Table 4 Effect of eluent flow rate

Eluent flow rate (mL/min)	%Recovery of Fe(III)
0.2	93.1±0.5
0.4	94.2±0.3
0.6	96.2±0.9
0.8	92.8±1.0
1.0	91.6±1.5

Table 5 Effect of interfering ions (ions that are present other than the sample) on recovery of Fe(III) by the proposed method (n=4)

Matrix ion	Tolerance limit (μgL^{-1})	Recovery (%)
Na^+	1700	96.19±1.31
Li^+	1300	94.12±0.86
K^+	1900	95.66±0.12
Ca^{+2}	1200	95.43±0.18
Mg^{+2}	1200	95.37±0.53
Cl^-	700	96.90±0.31
NO_3^-	500	96.53±0.12
CH_3COO^-	200	95.27±1.05
PO_4^{-2}	300	93.71±0.27
SO_4^{-2}	700	94.12±2.05

Table 6 Estimation of Fe(III) in water samples

Environmental water samples	Spiked Fe(III)	Found	Recovery
	$\mu\text{g/L}$	$\mu\text{g/L}$	%
Sample(1)	0.0	0.63 \pm 0.5	-
	5.0	5.45 \pm 0.4	96.80
	10.0	10.56 \pm 1.0	99.35
	15.0	15.46 \pm 0.9	98.92
Sample(2)	0.0	0.85 \pm 1.3	-
	5.0	5.75 \pm 0.4	98.30
	10.0	10.76 \pm 1.2	99.17
	15.0	15.79 \pm 0.7	99.62

Table 7: Comparison of present method with reported methods

Proposed method	limit of Detection ($\mu\text{g L}^{-1}$)	reference
	Fe(III)	
SBA-15-ATT	0.32	this work
Diphenylcarbazide(AmberliteXAD-2000)	0.32	17
Dowex optipore L-493	0.82	18
Amberlite XAD-7	3.07	19