



Arsenic removal from aqueous system using modified chestnut shell

Şerif Targan, V. Nüket Tirtom*

Faculty of Science and Arts, Chemistry Department, Celal Bayar University, 45140 Muradiye-Manisa, Turkey,
Tel. +902362013172; Fax: +902362013040; email: nuket.tirtom@cbu.edu.tr (V.N. Tirtom), Tel. +902362013166;
Fax: +902362013040; email: serif.targan@cbu.edu.tr (Ş. Targan)

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ABSTRACT

Natural adsorbent chestnut shell treated with FeCl_3 was prepared and employed by means of batch method for selective adsorption and removal of arsenic from aqueous system. The influences of different parameters, such as contact time, pH, temperature, and concentration of adsorbate, on adsorption performance of chestnut shell treated with FeCl_3 was studied in order to optimize the adsorption conditions. Batch adsorption studies have shown that removal capacity of chestnut shell can be increased by FeCl_3 treatment. The maximum adsorption capacity was found to be 0.885 mg g^{-1} As(III) of chestnut shell, which was treated with FeCl_3 . The adsorption data obtained follow a first-order rate expression and fit the Freundlich isotherm that has been used to obtain the thermodynamic parameters. In addition, the thermodynamic parameters, such as standard free energy (ΔG°), standard enthalpy (ΔH°), and standard entropy (ΔS°), of the adsorption process were calculated. It was found that the adsorption of arsenic on chestnut shell was exothermic. The adsorbents were characterized by scanning electron microscopy.

Keywords: Arsenic; Adsorption; Chestnut shell

1. Introduction

Arsenic pollution in aquatic environments is a worldwide concern due to its toxicity and chronic effects on human health [1]. Permanent arsenic intake leads to chronic intoxication, and prolonged arsenic exposure can damage the central nervous system, liver, and skin, and results in the appearance of diverse types of cancer such as hyperkeratosis, lung and skin cancer [2]. WHO had recommended a maximum permissible contaminant level of $10 \mu\text{g L}^{-1}$ of arsenic in drinking water standard considering the epidemiological evidence of arsenic carcinogenicity [3–6]. Removal of arsenic is one of the most important

areas of wastewater treatment. Various treatment methods, such as precipitation [7], electrolysis [8], reverse osmosis [9,10], ion exchange [11], and adsorption, have been so far proposed and adopted for the removal of arsenic from aqueous media. As it is easy to remove the adsorbent from aqueous media after treatment, adsorption technique is generally considered to be a promising method and has been studied for arsenic removal as well. Advantages of adsorption processes over other techniques are: its low running cost, the absence of electrical supply, and its low maintenance requirements. In recent years, many low-cost sorbents including agricultural waste and byproducts have also been tested in batch and fixed-bed sorption systems by a number of authors. For

*Corresponding author.

example, Coconut shell [12], orange waste [13], pine wood char, oak wood char, oak bark char, pine bark char [14], and *Escherichia coli* [15,16] were used to remove of arsenic ions from wastewaters. In this study, the effectiveness of a low-cost arsenic adsorbent, a chestnut shell modified with FeCl_3 , is presented. Iron, aluminum, and copper have the greater affinities towards arsenic, because they form different compounds with it [17–20]. Ferrate(VI) is one of the most promising candidates for advanced technologies of arsenic treatment [21]. Recently, magnetic nanoparticle materials have gained special attention in water treatment, based on their advantages such as easy separation, simple manipulation process, kind operation conditions, and easy specifically functional modifications [22,23]. The objective of the present work is to assess the function of pH, contact time, temperature, and initial arsenic concentration on the As(III) sorption onto chestnut shell modified with FeCl_3 .

2. Materials and methods

2.1. Reagent

All the chemicals were of analytical grade. As_2O_3 , NaOH, HCl, CH_3COOH , $\text{NaCH}_2\text{COO}\cdot 3\text{H}_2\text{O}$, KH_2PO_4 , Na_2CO_3 , and NaHCO_3 were purchased from Merck. Chestnut shell was mined at Türkiye. Ultrapure deionized water used in all the study was purified by Milipore ultrapure water purification system.

2.2. Instruments

Arsenic concentration measurements were carried out using Varian 220 FS AAS. Atomic absorption spectrophotometer equipped with a arsenic hollow cathode lamp at the wavelength 253.7 nm was used for the determination of arsenic in sample solutions. A Varian vapor generation accessory model VGA-77 with a quartz cell was used for the hybrid generation of arsine vapor. The pH of solution was measured with a Hanna P211 microprocessor pH-meter using a combined glass electrode. The shaking experiments were carried out in a thermostated electronic shaker Labart SH-5 and drying was carried out in a Nüve EN 400 drying oven.

2.3. Preparation and modification of adsorbent

Chestnut shell was washed with distilled water and dried at room temperature. They were sieved with a grinded mill to obtain 100 and 200 μm particle size. Treatment method was used for modification of

the adsorbent. Twenty grams of dry adsorbent was transferred into a beaker containing 25.0 mL of 0.5 M FeCl_3 solutions and the suspensions were stirred for 12 h. The treated adsorbents were separated from the solution by decantation and washed with distilled water to remove the soluble compounds. They were dried at 105°C in an oven and used in the adsorption studies.

2.4. Method

Stock solutions ($1,000 \text{ mg L}^{-1}$) of As(III) were prepared by dissolving $0.6602 \pm 0.0002 \text{ g}$ As_2O_3 in 10% NaOH and the solution was acidified by adding concentrated HCl and diluted to 500 mL with ultrapure deionized water. As_2O_3 does not dissolve completely in water; therefore, As_2O_3 should be dissolved in NaOH and the basic solution could be neutralized by an acid solution such as HCl. Standard solutions of arsenic at required concentrations were prepared by dilution of appropriate volume of stock solution. As reducing agent, 0.5 g NaBH_4 and 0.25 g NaOH was prepared daily diluting to 50 mL with distilled water.

2.5. Batch adsorption experiments

Adsorption experiments were carried out using batch technique. The value $0.10 \pm 0.02 \text{ g}$ of each adsorbent (chestnut shell) was put into beaker containing 25 mL of 1 mg L^{-1} As(III) solution and the suspension was stirred. After decantation and dilution, the concentration of As(III) was analyzed by atomic absorption spectrometer at 253.7 nm by hydride generation system. The effect of contact time was studied in 0.5–24 h. The effect of As(III) adsorption was studied in the pH range of 3.0–10.0 using 0.1 M CH_3COOH and 0.1 M NaCH_2COO , $\text{Na}_2\text{HPO}_4\cdot 2\text{H}_2\text{O}$ - $\text{NaH}_2\text{PO}_4\cdot 2\text{H}_2\text{O}$, and $\text{NH}_3\text{-NH}_4\text{Cl}$ buffer systems. The effect of temperature was studied at 25–40°C. Isotherm studies were conducted with a constant modified chestnut shell weight ($0.10 \pm 0.02 \text{ g}$) and varying initial concentrations of As(III) in the range of 5–100 mg L^{-1} . All the experiments were carried out twice.

The percentage adsorption of arsenic on adsorbent from aqueous solution was computed as follows:

$$\text{Adsorption (\%)} = \frac{C_{\text{int}} - C_{\text{fin}}}{C_{\text{int}}} \times 100 \quad (1)$$

where C_{int} is the initial As (III) concentration (mg L^{-1}) and C_{fin} is the final As(III) concentration (mg L^{-1}).

2.6. Characterization of modified chestnut shell

Fourier transform infrared (FTIR) analysis was accomplished on a Perkin Elmer spectrum BX scanning from 4,000 to 400 cm^{-1} at room temperature. Chestnut shell, arsenic adsorbed chestnut shell, chestnut shell treated with FeCl_3 , and arsenic adsorbed chestnut shell treated with FeCl_3 were mixed with KBr and pressed to plates for measurements. Surface morphology of samples was determined by scanning electron microscope (SEM) (XL30-SFEG, FEI/Philips).

3. Results and discussion

3.1. Effect of contact time on sorption of Arsenic(III)

To determine the effect of contact time on arsenic adsorption, adsorbents were prepared as similar way given under batch adsorption experiments section. The adsorbent was treated with arsenic solution for a time of 0.5, 1.0, 2.0, 4.0, 6.0, 18.0, and 24.0 h at room temperature. The effect of treated time on the adsorption capacity of the adsorbents is shown in Fig. 1. The initial concentration of the As(III) was 1 mg L^{-1} . According to the obtained data, the highest value of adsorbed As(III) ions on chestnut shell treated with FeCl_3 was reached at 24.0-h treatment. The adsorption percentage was 44% for chestnut shell treated with FeCl_3 .

3.2. Effect of pH on sorption of Arsenic(III)

pH is one of the most important parameters affecting the adsorption of metal ions. To study the influence of pH on the adsorption capacity of modified chestnut shell for As(III), experiments were performed using an initial concentration of 1 mg L^{-1} over the pH range 3.0–10.0 and the results are presented in Fig. 2. The maximum adsorption percentage of As(III) was

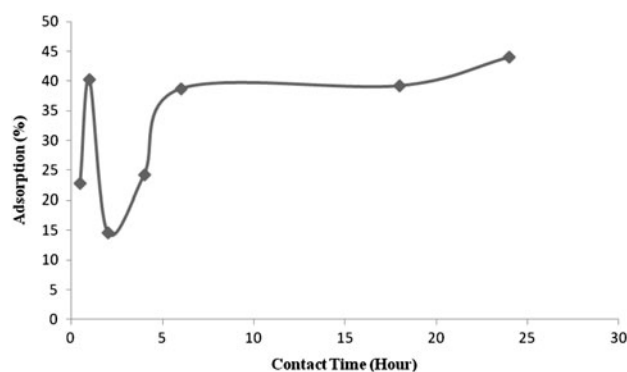


Fig. 1. Effect of contact time on sorption of As(III) ($0.10 \pm 0.02 \text{ g}$ adsorbent, 25 mL of 1 mg L^{-1} As(III) solution).

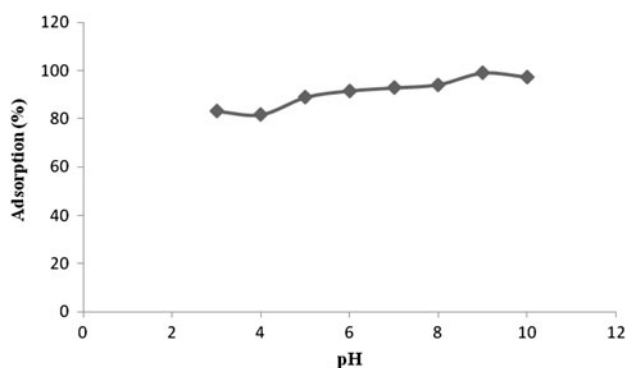


Fig. 2. Effect of pH on sorption of As(III). ($0.10 \pm 0.02 \text{ g}$ adsorbent, 25 mL of 1 mg L^{-1} As(III) solution, 24 hour).

observed at the pH 9.0. Oxygen, in the form of negatively charged anions, was stable type of arsenic in water. Trivalent arsenite (AsO_3^{3-}) such as HAsO_3^{2-} , H_2AsO_3^- , H_3AsO_3 types are available. There are anionic H_3AsO_3 and H_2AsO_3^- types in water in the pH range (6.0–9.0). The pH value of the water was 6.0, when fully H_3AsO_3 species, H_2AsO_3^- type as well as the pH begins to emerge. There is 70% of the H_3AsO_3 and 30% H_2AsO_3^- at pH 9.0. High pH of maximum adsorption youth +3' as observed at pH reputation of the anionic forms (HAsO_3^{2-} and H_2AsO_3^-) due to the adsorption of these structures can be explained by voluntary.

3.3. Effect of temperature on sorption of Arsenic(III)

The temperature of the adsorption could be important for energy-dependent mechanisms in metal adsorption by adsorbents. The adsorption experiments were performed in the temperature range of 25–40°C and the results are shown in Fig. 3. The optimum temperature was selected as 35°C for further adsorption experiments.

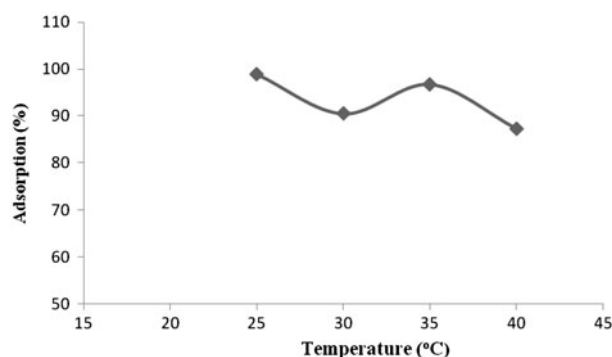


Fig. 3. Effect of temperature on sorption of As(III) ($0.10 \pm 0.02 \text{ g}$ adsorbent, 25 mL of 1 mg L^{-1} As(III) solution, 24 hour, pH = 5.0).

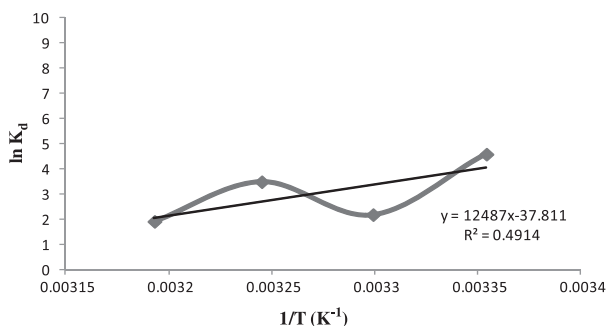


Fig. 4. Plot of $\ln K_d$ vs. $1/T$ As(III) (0.10 ± 0.02 g adsorbent, 25 mL of 1 mg L^{-1} As(III) solution, 24 hour, pH = 5.0).

3.4. Thermodynamics of adsorption

To investigate the controlling mechanism of the adsorption processes, temperature-dependent distribution coefficient was computed as follows:

$$K_d = \frac{C_{Ad}}{C_e} \quad (2)$$

where K_d is the equilibrium constant, and C_{Ad} and C_e are the equilibrium concentrations (mg L^{-1}) of As(III) on the modified chestnut shell in the solution. The adsorption process was also assessed at different temperatures between 25 and 40°C. Temperature effects on adsorption were shown in Fig. 3. As shown in Fig. 3, adsorption was increased with temperature for As(III) ions. Thus, the As(III) adsorption-modified chestnut shell is endothermic in nature. Thermodynamic parameters, such as free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°), were calculated by using the following equation (3). The change of enthalpy (ΔH°) and entropy (ΔS°) can be obtained from the intercept and slope of Van't Hoff equation of ΔG° vs. T :

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (3)$$

where ΔG° is standard Gibbs free energy change (kJ/mol), R is universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$),

and T is absolute temperature (K). The values of ΔH° and ΔS° were calculated from the slope and intercept of Van't Hoff plot of $\log K_d$ vs. $1/T$ as presented in Fig. 4. Values of the standard Gibbs free energy change for the adsorption process obtained from Eq. (3) were listed in Table 1. The negative value of ΔH° ($-103.816 \text{ kJ mol}^{-1}$) and the negative value of ΔS° ($-314.36 \text{ J K}^{-1} \text{ mol}^{-1}$) indicates that the adsorption processes are exothermic. The negative value of ΔG° ($-6792.80 \text{ J mol}^{-1}$) shows an adsorption process proceeds spontaneously.

3.5. Adsorption isotherms

Adsorption isotherm models describe the relationship between adsorbents and adsorbents at equilibrium [17]. The Langmuir and the Freundlich are the most known isotherm models. In this study, equilibrium isotherms were used to determine the adsorption mechanism of modified chestnut shell for As(III) ions. The adsorbent was mixed with fixed volumes (25 mL) of metal ion solutions varying the initial concentrations (25, 50, 75, 100, 125, 150, 175, 200, 400, 600, 800, and $1,000 \mu\text{g L}^{-1}$) in 24 h for As(III) ions (Fig. 5).

Freundlich isotherm is an empirical equation describing adsorption on to a heterogeneous surface and reversible adsorption, and not restricted to the

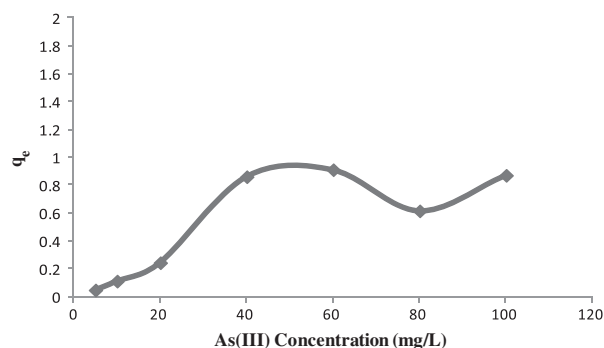


Fig. 5. Effect of As(III) ions concentration on the adsorption (%) by modified chestnut shell (0.10 ± 0.02 g adsorbent, 25 mL of 1 mg L^{-1} As(III) solution, 24 hour, pH = 5.0, 35°C).

Table 1

Isotherm constants and thermodynamic parameters for adsorption of As(III) ions on modified chestnut shell

Adsorbent	Freundlich isotherm constants As(III)			Thermodynamic parameters As(III)		
	R^2	KF (mg g^{-1})	n	ΔG° (J mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° ($\text{J mol}^{-1} \text{ K}$)
Modified chestnut shell	0.8295	0.0178	1.1566	-6792.80	-103.816	-314.36

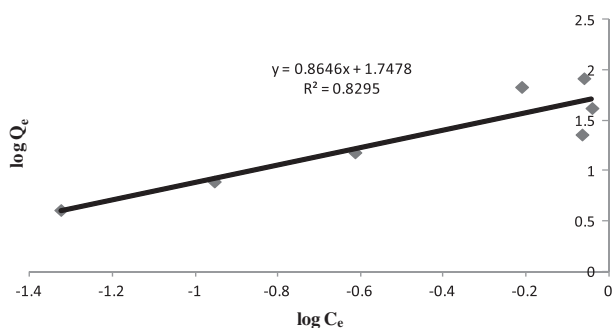


Fig. 6. Freundlich plot for As(III) adsorption on modified chestnut shell at equilibrium.

monolayer formation [24,25]. The Freundlich isotherm equation is the most important multi-site adsorption isotherm for heterogeneous surfaces. The Freundlich isotherm is commonly presented as [26]:

$$q_e = KF C_e \frac{1}{n} \tag{4}$$

where KF and *n* are the Freundlich constants related to the adsorption capacity and adsorption intensity of the sorbent, respectively.

Eq. (4) can be linearized by taking logarithms:

$$\ln q_e = \ln KF + \frac{1}{n} C_e \tag{5}$$

The Freundlich parameters for the adsorption of As (III) were also given in Table 1. The slope of line expressed by Eq. (5) gives the value 1/*n* and intercept of the line gives the value log KF. The Freundlich constants for As(III) ions sorption and correlation coefficient were calculated. According to the results, Freundlich model was found to describe adsorption

successfully than Langmuir model isotherm in respect to linearity coefficients obtained for both models (*R*² = 0.8295) for As(III) ions (Fig. 6). The Freundlich type adsorption isotherm is an indication of surface heterogeneity of the adsorbent. Comparative adsorption of As(III) and As(V) ions onto various types of adsorbents are shown in Table 2.

3.6. Surface morphology

The scanning electron microscopy of modified chestnut shell and As(III)-loaded modified chestnut shell are shown in Fig. 7. The surface change in the SEM micrographs indicated the structural changes in the sorbent before and after As(III) treatment. The general morphology of the modified chestnut shell before adsorption could be characterized as rough and folded. It was found that the surfaces of the As(III)-loaded modified chestnut shell was noted to have much asperity. It has seen that surface is more uneven after adsorption.

FTIR spectra, depicted in Fig. 8, shows the changes observed in chestnut shell, arsenic adsorbed chestnut shell, chestnut shell treated with FeCl₃, and arsenic adsorbed chestnut shell treated with FeCl₃. The IR spectrum of chestnut shell exhibits an adsorption band 3,640 and 3,200 cm⁻¹ due to -OH stretching vibration; at around 3,100 and 2,900 cm⁻¹, assigned to -CH stretching. An adsorption band at 1,730 cm⁻¹ is assigned to -C=O stretching vibrations in esters. An adsorption band at 1,590 cm⁻¹ is assigned to -C-C stretching (in ring) vibrations in aromatics. An adsorption band at 1,300 and 1,000 cm⁻¹ is formed due to -C-O stretching vibrations. In spectrum of chestnut shell treated with FeCl₃, difference can be observed. For instance, in spectrum of arsenic-adsorbed chestnut shell treated with FeCl₃, the pick at around 2,300 cm⁻¹ is due to C (triple bound)N-stretch bending vibrations in nitriles.

Table 2

Comparative adsorption of As(III) and As(V) ions onto various types of adsorbents

Type of adsorbent	As(III) <i>q_m</i> (mg g ⁻¹)	As(V) <i>q_m</i> (mg g ⁻¹)
Iron hydroxide coated Alumina [27]	7.64	36.64
Iron oxide coated sand IOCS [28]	0.136	–
Ferrihydrite (FH) [29]	–	0.25
Iron oxide uncoated sand [30]	0.006	–
Al ₂ O ₃ /Fe(OH) ₃ [31]	–	0.09
Waste Fe(III)/Cr(III) [32]	–	11.02
GAC-Fe-H ₂ O ₂ [33]	3.94	–
Modified iron oxide-coated sand [34]	0.14	–
<i>Escherichia coli</i> cell [15]	0.035	–
Chestnut Shell treated with FeCl ₃ (This study)	0.88	–

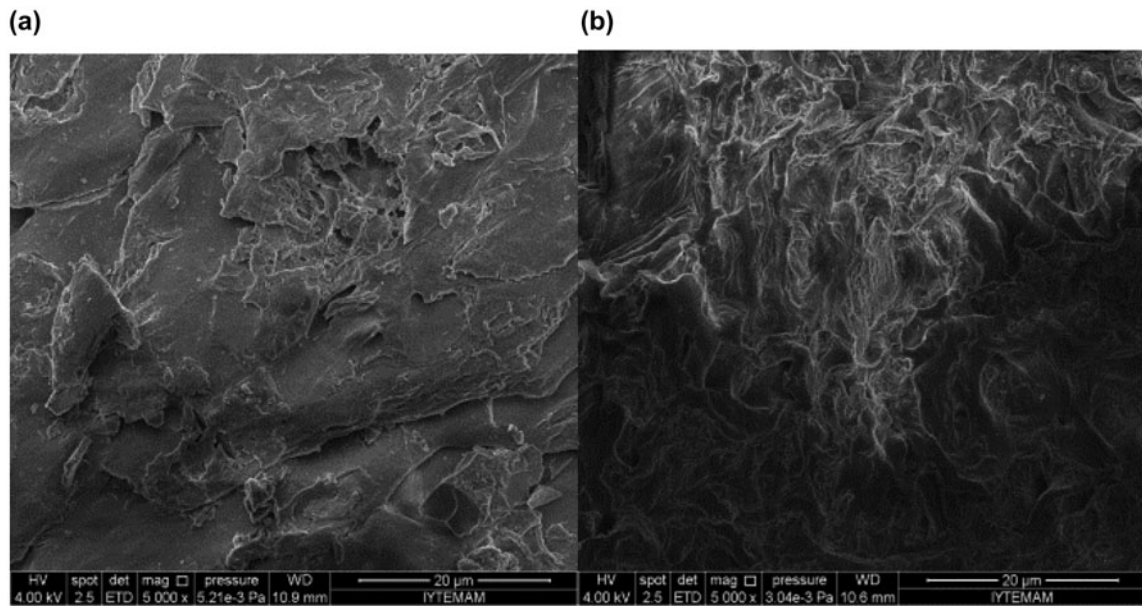


Fig. 7. The SEM analyses of modified chestnut shell and modified chestnut shell with As(III). (a) Modified Chestnut Shell (before adsorption). (b) Modified Chestnut Shell (after adsorption).

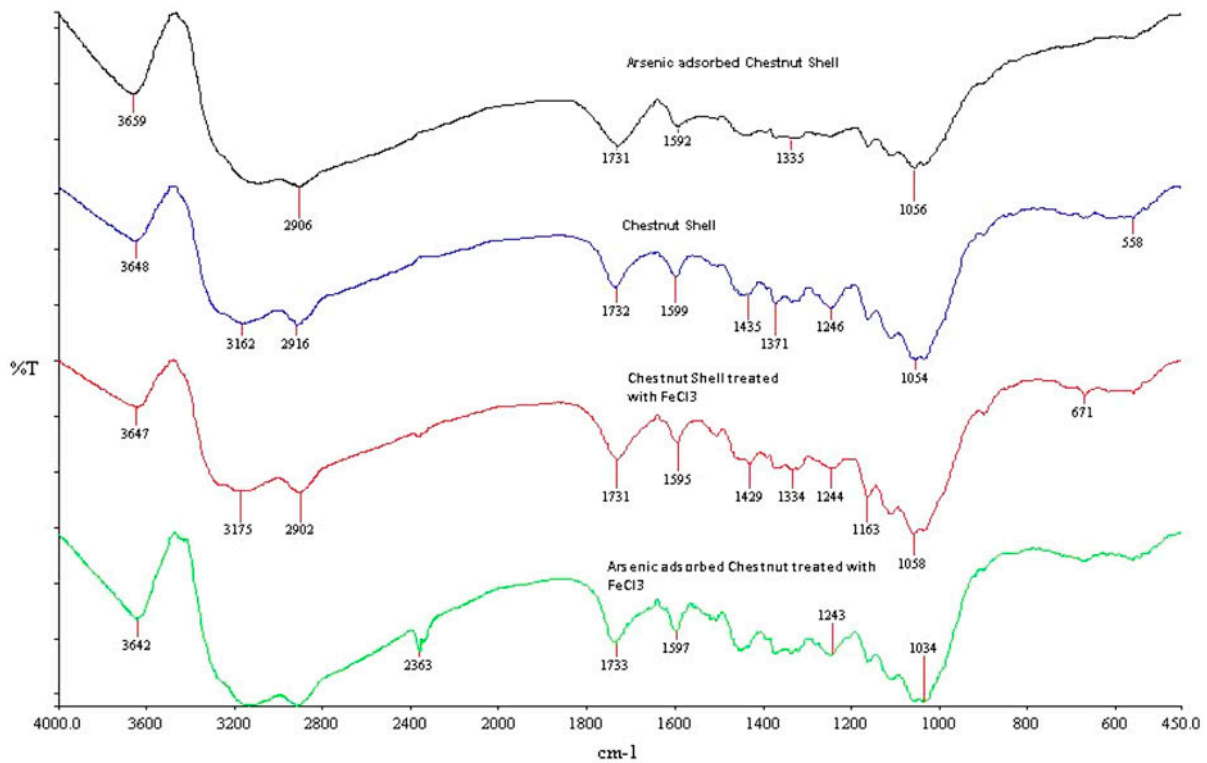


Fig. 8. FTIR spectrum chestnut shell, arsenic adsorbed chestnut shell, chestnut shell treated with FeCl₃ and arsenic adsorbed chestnut shell treated with FeCl₃.

4. Conclusion

In this study, modified chestnut shell was prepared as adsorbent to remove As(III) ions from aqueous solution. The prepared adsorbent was characterized by SEM analyses. Adsorption study showed that modified chestnut shell could be effectively used to remove As(III) ions. The pH 9.0 showed to have the highest adsorption for As(III) ions. The adsorption process was exothermic ($\Delta H^{\circ}_{\text{ads}} < 0$) for As(III) ions. Maximum adsorption capacity was 0.885 mg g^{-1} for As(III) ions. Many adsorbents are available but few are economical for removal of heavy metals. Modified chestnut shell is low price and effective material to remove As(III) ions from water. In conclusion, the results suggested that the adsorbent could be used in processes of As(III) ion uptake from aqueous solutions.

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