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REMOVAL OF As(III) FROM WATER USING A NOVEL ORANGE PEEL BIOPOLYMER BASED MAGNETIC NANOCOMPOSITES

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Abstract. Arsenic pollution in groundwater is of high concern due to its impact to environment and human health. Numerous methods have been used to treat arsenic pollution. In this work, a practical application of biopolymer-based magnetic nanocomposites as a novel adsorbent for the arsenic pollutant was demonstrated. Magnetic nanocomposites were produced by incorporating cobalt superparamagnetic ($CoFe_2O_4$) nanoparticles into the biopolymer matrix which was extracted from orange peel. In which, the superparamagnetic nanoparticles were prepared by co-precipitation approach and the nanocomposites formation was carried out with the support of magnetic agitation. Various characterizations including Fourier transform infrared spectroscopy (FT-IR), X-ray powder diffraction (XRD), Scanning electron microscopy (SEM), and Vibrating sample magnetometry (VSM) were carried out to investigate the property of the obtained biopolymer magnetic nanocomposites. The materials was used as adsorbent, then applied to remove arsenic trioxide in the solution. The result indicated that 99.2% of arsenic trioxide (1.0 g/L feed concentration, 1.0 g/L dose of the material) could be removed by the adsorbent. In addition, the nanocomposites after treatment could be facilely separated from the aqueous mixture by simple magnetic decantation due to its superparamagnetism, making it easy to completely isolate them from water and exhibiting good reusability.

Keywords. nanocomposites, magnetic, orange peel, biopolymer, superparamagnetism, As (III), reusability.

1 INTRODUCTION

Arsenic (As), one of the common constituents of the earth's crust, is a contaminant in groundwater source. Groundwater arsenic pollution has been reported from numerous countries all over the world. A high concentration of arsenic is a big concern for drinking water and food safety. Long-term exposure to arsenic may cause negative effects on human health, even can lead to cancers [1]. Therefore, removal of arsenic from water is of high importance. Many different technologies such as precipitation, adsorption, ion exchange, membrane filtration, etc. have been used for arsenic removal from aqueous solution [2, 3]. Each method has its own advantages and disadvantages [4]. Among these methods, adsorption is one of the most efficient approaches which is cost-effective to remove arsenite(III) in groundwater. Various types of low cost adsorbent have been applied including oxides, soils and constituents, phosphates, agricultural products, industrial by-products as well as biosorbent [4]. Recently, biopolymer, which is biodegradable, hence environment-friendly, has demonstrated as a potential adsorbent to remove heavy metals in aqueous solution [5]. However, the separation of adsorbent from post-treatment water is still a drawback which inhibits its practical application. To overcome this challenge, polymer can be combined with magnetic nanoparticles, which can be easily isolated from water by applying a magnetic field [6]. Moreover, the adsorption capacity of such nanocomposites can be enhanced greatly since magnetic nanoparticles are also well-known as superior adsorbents [7]. In this work, we attempt to use waste orange peel as biopolymer source for preparing polymer-based magnetic nanocomposite as an adsorbent to remove As(III) in groundwater with enhanced collection ability.

2 MATERIALS AND METHODS

Materials: the reagents including cobalt (II) chloride ($CoCl_2.6H_2O$, 99%); iron (II) chloride ($FeCl_2.4H_2O$, 98%); sodium hydroxide (NaOH, 96%); n-hexane (95%); ethanol (C_2H_5OH, 96°); ammonium hydroxide (NH_4OH, 25-28%); arsenic trioxide (As_2O_3, 99%); chemical analysis filter paper (Newstar 101, filter hole diameter 20-25 μm) were supplied from China. While sodium dodecyl sulfate (SDS, >85%) was provided by Merck. All the reagents were used as received without any further purification. Orange peel was obtained from Go Vap market, HCM city, Vietnam.

2.1 Biopolymer isolation

The orange peel biopolymer was isolated following a modified procedure from previous publications [8, 9]. Firstly, 3.0 g of dried orange peel pulp was washed, chopped and blanched in hot water (60 °C). After adjusting pH to 2 using 0.1N HCl solution, the mixture was boiled for 180 minutes to remove enzymes. The mixture was then cooled to room temperature and adjusted to pH 7.0 by 0.01N NaOH solution. The first filtration was performed to get the filtrate and the precipitation of biopolymer was done by ethanol 96° overnight. The second filtration was performed to obtain biopolymer, then the biopolymer was washed several times with 96° ethanol. Finally, the biopolymer was dried at 60 °C prior to storage.

Biopolymer yield was calculated as follows:

$$Biopolymer\ (\%) = \frac{Weight\ (g)\ of\ dried\ biopolymer}{Weight\ (g)\ of\ dried\ orange\ peel} x100\%$$

2.2 Synthesis and hydroxylation of magnetic nanoparticles

The preparation of magnetic nanoparticles was followed a reported procedures [9, 10]. $CoFe_2O_4$ magnetic nanoparticles were obtained by coprecipitation using sodium dodecyl sulfate (SDS) as surfactant. Firstly, 250 ml aqueous solution of SDS (9.35 g; 27.75 mmol) was rapidly added into 250 ml aqueous solution of a mixture containing $CoCl_2.6H_2O$ (1.2 g; 5.00 mmol) and $FeCl_2.4H_2O$ (2.0 g; 10.00 mmol). The solution was then heated to (70 ± 5 °C) under stirring and maintained at these conditions for 30 mins. Successively, 500 ml of NaOH 0.75M was slowly poured into the reaction vessel and the solution was vigorously stirred in 5 hours. The fabricated magnetic nanoparticles were collected by a strong magnet, then washed with water, ethanol and n-hexane to remove the excess of surfactant and finally were left for drying overnight at ambient conditions [9, 10]. Then, the hydroxylation of obtained $CoFe_2O_4$ magnetic nanoparticles was carrying out by firstly dispersing them in 350 ml mixture of ethanol and water (1:1, vol/vol) under sonication for 30 mins. Then, 35 ml ammonium hydroxide was added and the suspension was vigorously stirred at 55-65 °C in 24 hours. Hydroxylated magnetic nanoparticles were recovered by a strong magnet, washed with excess of water, ethanol and left for drying overnight in air [9, 10].

2.3 Synthesis of nanocomposites based on the hydroxylated magnetic nanoparticles and orange peel biopolymer

The hydroxylated magnetic nanoparticles were added to 1.0 wt% biopolymer solution in a 500 mL beaker, weight ratio of hydroxylated magnetic nanoparticles/ biopolymer was 1/5 (g/g). The mixture was stirred and kept stable at 90 °C in 30 minutes. The formed nanocomposites were taken out using a strong magnet, washed with excess of water, ethanol, *n*-hexane and left for drying in air.

2.4 Characterization

The crystalline structure of the synthesized materials was investigated by X-ray diffraction (XRD) which patterns were recorded by a D8-Advance from Bruker using monochromatic Cu K_α radiation. The 2θ scanning ranges from 10-80° at a scanning rate of 2.25°/min. The presence of biopolymer in nanocomposite composition was studied by Fourier transform infrared (FT-IR) spectrometer (TENSOR 27- Bruker, Germany) in the wavenumber range of 400-4000 cm^{-1}. Scanning Electron Microscopy (SEM) (S-4800) was used to observe the morphology of the nanomaterials while vibrating sample magnetometer (VSM) was applied to assess magnetic properties via hystereris loop.

2.5 Removal of As(III) in aqueous solution via adsorption

In a typical experiment, 100 ml aqueous solution containing 0.1 g/L As(III) was added into a beaker. Then, a certain amount of adsorbent materials (either orange peel biopolymer or –OH enriched magnetic nanoparticles or biopolymer based nanocomposites) was added into each beaker. The mixture was agitated at 120 rpm for 5 h at ambient temperature. Finally, the adsorbent materials were simply collected by applying a magnet. As(III) in the obtained supernatant was precipitated by adjustment pH 5 by 0.1 M HCl, the filter residue was dried and weighed to determine the efficiency of the As(III) treatment. Each sample was duplicated and the average result was recorded. The amount of the arsenic adsorbed (mg) per unit mass of adsorbent (g), q_e (mg/g), was obtained by mass balance using following equation:

$$q_e = \frac{C_i - C_e}{C_a}$$

where C_i and C_e are initial and equilibrium concentrations of As(III) (mg/L), C_a is concentration of adsorbent (g/L).

Effect of adsorbents: effect of adsorbents on the percentage of As(III) adsorption by biopolymer, –OH enriched magnetic nanoparticles and nanocomposites were studied in the parameters: adsorbent dose 1.0 g/L, initial As(III) concentration 1.0 g/L, contact time 5 h, pH 12, agitation speed 120 rpm and volume 100 mL, were kept constant.

Effect of mass ratio of nanocomposites adsorbent to As(III): the effect of the weight ratio on the percentage of As(III) adsorption by nanocomposites adsorbent was studied by varying the adsorbent dose from 0.5 g/L to 2.5 g/L. Other parameters were kept constant, such as the initial As(III) concentration 0.5 g/L, contact time 5 h, pH 7.0, agitation speed 120 rpm and volume 100 mL.

Effect of pH: effect of pH on the percentage of As(III) adsorption by the nanocomposites adsorbent was studied in the pH range of 6 to 10. Other parameters, such as adsorbent dose 0.4 g/L, initial As(III) concentration 0.1 g/L, contact time 5 h, agitation speed 120 rpm and volume 100 mL, were kept constant. The pH of the solution was adjusted by adding 0.1 M HCl and 0.1 M NaOH. A pH of the solution was determined by using pH Tester, HANNA HI-98107, Romania.

Effect of contact time: the effect of contact time on the percentage of As(III) adsorption by nanocomposites adsorbent was studied at different contact time from 1.0 to 5.0 h. Other parameters were kept constant, such as the adsorbent dose 0.4 g/L, initial As(III) concentration 0.1 g/L, pH 6, agitation speed 120 rpm and volume 100 mL.

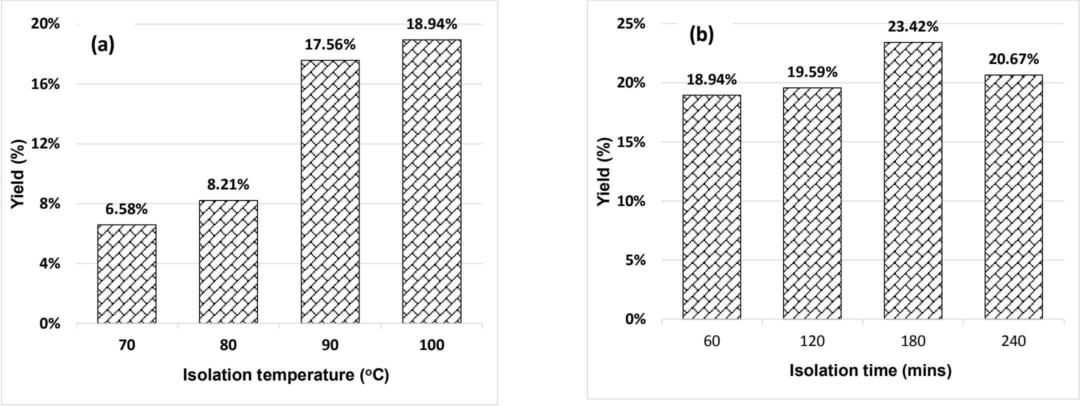
Desorption and reusability: desorption experiment was investigated using 0.1 M NaOH. Nanocomposites adsorbent was first loaded with As(III) by mixing 0.04 g adsorbent with 100 mL of 0.1 g/L As(III) solution under agitation for 5 h to reach equilibrium. The resultant suspension was magnetically separated and the remaining As(III) concentration in supernatant was determined. Subsequently, the solid residue was thoroughly washed with copious distilled water and mixed with 20 mL of 0.1 M NaOH at room temperature under agitation condition for 6 h. After desorption, the adsorbent was reused for removal As(III) for subsequent times with similar conditions.

Study of removal of As(III) in aqueous solution was performed in Jartest system (OVAN JT60E, Spain).

3 RESULTS

3.1 Orange peel biopolymer isolation

A fixed amount of 3.0 g orange peel was used for biopolymer isolation and the process was performed at the mass ratio of solvent/orange peel sample reached 30/1 (wt/wt). According to previous studies [11, 12], it was reported that the enzyme-reduction is not completed at the temperature below 60 ºC. Hence, the proper temperature for enzyme reduction was investigated. Except temperature, the biopolymer isolation of orange peel also depends on isolation time, pH [13, 14].



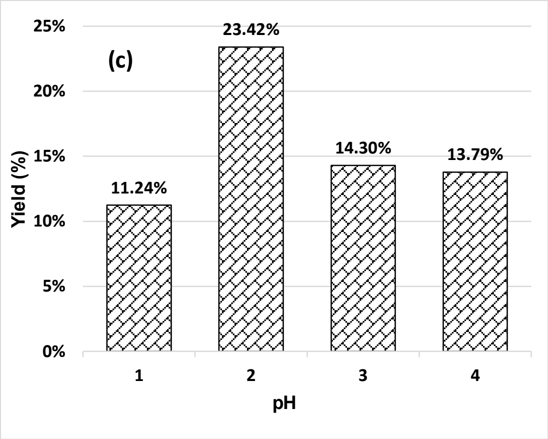


Figure 1. Effect of temperature (a), time (b) and pH (c) on biopolymer isolation in the following conditions: 1. Enzyme reduction: HCl 0.01N; 2. Ratio of solvent to sample: 30/1; 3. Weight of orange peel: 3.0 g

The effect of biopolymer isolation conditions show that the maximum amount of biopolymer was obtained when the temperature reached 100 ºC, in 180 minutes and pH 2. The effect of temperature was also studied in this work in which the highest biopolymer isolation yield of 18.94% was achieved at 100 ºC (Figure 1a), this finding was also reported in previous research [13, 14]. Additionally, obtained biopolymer amount depends on the isolation time where 23.42% was the highest yield when carrying out the isolation for 180 mins and prolong the time lead to the decrease of isolation yield (Figure 1b). Differently, when using HCl as reducing agent for the enzyme reduction, the yield of biopolymer isolation was lower when either increasing or reducing pH (Figure 1c). It is assumed that increasing the pH of the environment leads to the increase in solubility of the biopolymer, hence reduce their collected amount. Moreover, at pH higher than 2, diminished acidity cause a decrease in enzyme reducing capability of the environment which weaken the ability to convert the –$COOCH_3$ group with weak polarity into –COOH group with stronger polarization. As a result, the attained amount of biopolymer also decreased which is consistent with the previous findings [11, 12].

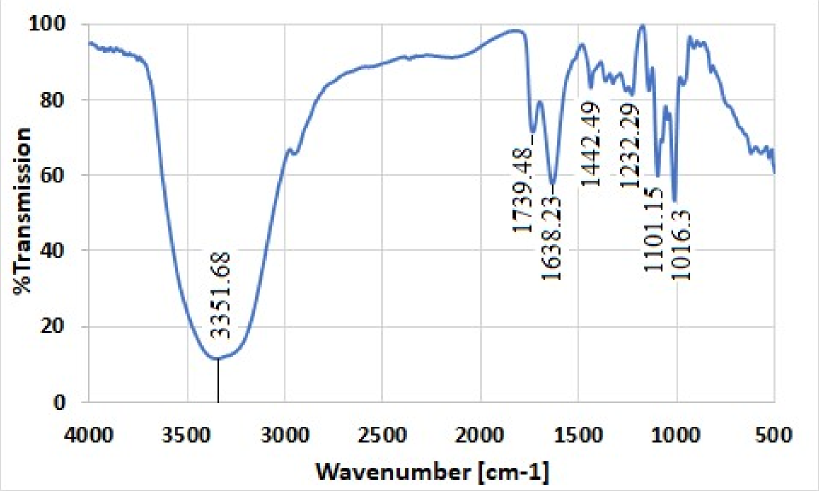


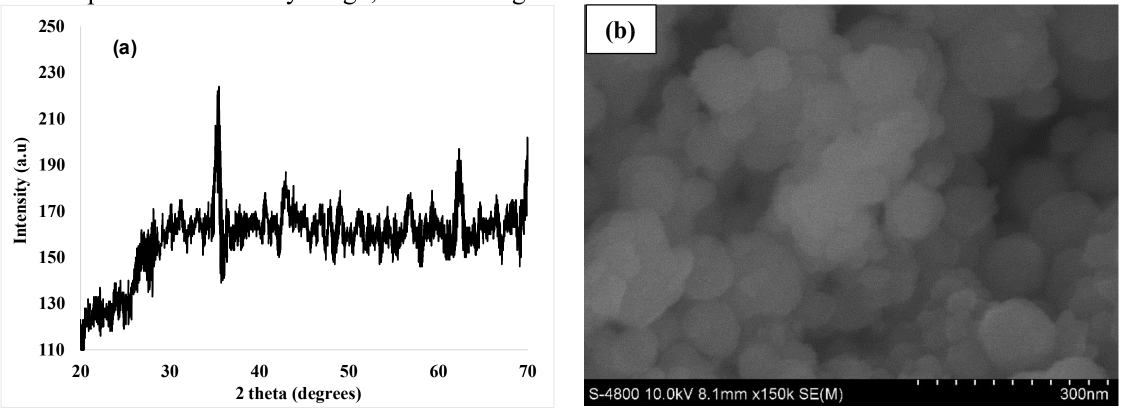
Figure 2. FT – IR spectrum of biopolymer

FT-IR results (Figure 2) show strong vibrational band centered at 3351.68 cm^{-1} which is typical oscillations for the –OH group. Further, the peak at 1739.48 cm^{-1} corresponds to the stretching vibrations of non-ionized C=O groups and the bands centered at 1638.23 cm^{-1} and 1442.49 cm^{-1} are characteristic for asymmetric and symmetric stretching vibrations of the COO^- group indicate low degree of esterification of the obtained biopolymer [15, 16]. Moreover, the peaks at the wavenumber of 1232.29 cm^{-1}, 1101.15 cm^{-1} and 1016.3 cm^{-1} represent characteristic vibrations of C-O in the C-O-H group of galactomannan [15, 17].

3.2 Characterization of the obtained magnetic nanoparticles and nanocomposites

The structure of the synthesized $CoFe_2O_4$ magnetic nanoparticles was investigated by X-ray diffraction method (XRD). The results (Figure 3c) show that the diffraction spectrum is completely consistent with the standard data (JCPDS card, No. 22-1086) and totally matches with previous studies on $CoFe_2O_4$ magnetic nanomaterials [10, 18]. In the XRD results, there were some peaks which represent impurities and amorphous structures. Moreover, it is observed in SEM image (Figure 3b) that the diameter of the $CoFe_2O_4$ magnetic nanoparticles varies in the range of 40-90 nm. The VSM results of $CoFe_2O_4$ nanoparticles and nanocomposites are shown in Figure 3d(I, II). Which exhibited the saturation of $CoFe_2O_4$ particles is 60.66 emu/g (magnetic resistance 4937.85 G) while the saturation of nanocomposites is 54.59 emu/g (magnetic resistance 4940.06 G). The materials with a saturation of 60.66 emu/g together with the particle size in nano range, the obtained nanomaterial is considered to possess superparamagnetic properties and therefore the material disperses well in solution and is easily recovered by external magnetic field when being used in As(III) treatment.

The presence of functional groups within $CoFe_2O_4$ magnetic nanoparticles as well as on their surface was determined by FT-IR spectrum. The result in Figure 3e.I shows the valence absorption band of Fe-O bond via peak centered at the wavenumber 551.54 cm^{-1}. Furthermore, OH bonds in hydroxyl group on the surface of magnetic nanoparticles were presented by valence oscillation in the vicinity of 3422.06 cm^{-1} and deformation oscillation at 1639.2 cm^{-1}. FT - IR analysis of nanocomposite (Figure 3e.IV) shows that there is still a peak corresponds to the vibration of Fe-O bond at 538.04 cm^{-1} which is a characteristic oscillation of $CoFe_2O_4$. The strong vibration peak at 3289.96 cm^{-1} is typical for -OH group and peaks centered at 1630.52 cm^{-1} and 1414.53 cm^{-1} are characteristic asymmetric and symmetric vibrations of C=O bonding in COO- group [15, 16]. It was reported that [19-25], the peaks centered at 1630.52 cm^{-1} và 1414.53 cm^{-1} could be attributed to the vibrations of asymmetric and symmetric metal-carboxylate bond (COO-Fe). Further, the difference (Δ) between ν_{asy} (COO-) and ν_{sym} (COO-) absorption band is indicative of the binding character of a carboxylate group with a metal ion. The Δ value of (1630.52 -1414.53 = 215.99 cm^{-1}) can be assigned to the bidentate bridge between COO- and Fe^{2+}, Fe^{3+} ions [24, 26-28]. SEM image of nanocomposites materials is displayed in Figure 3c. According to observation, the surface of the nanocomposites is relatively rough, clustered together.



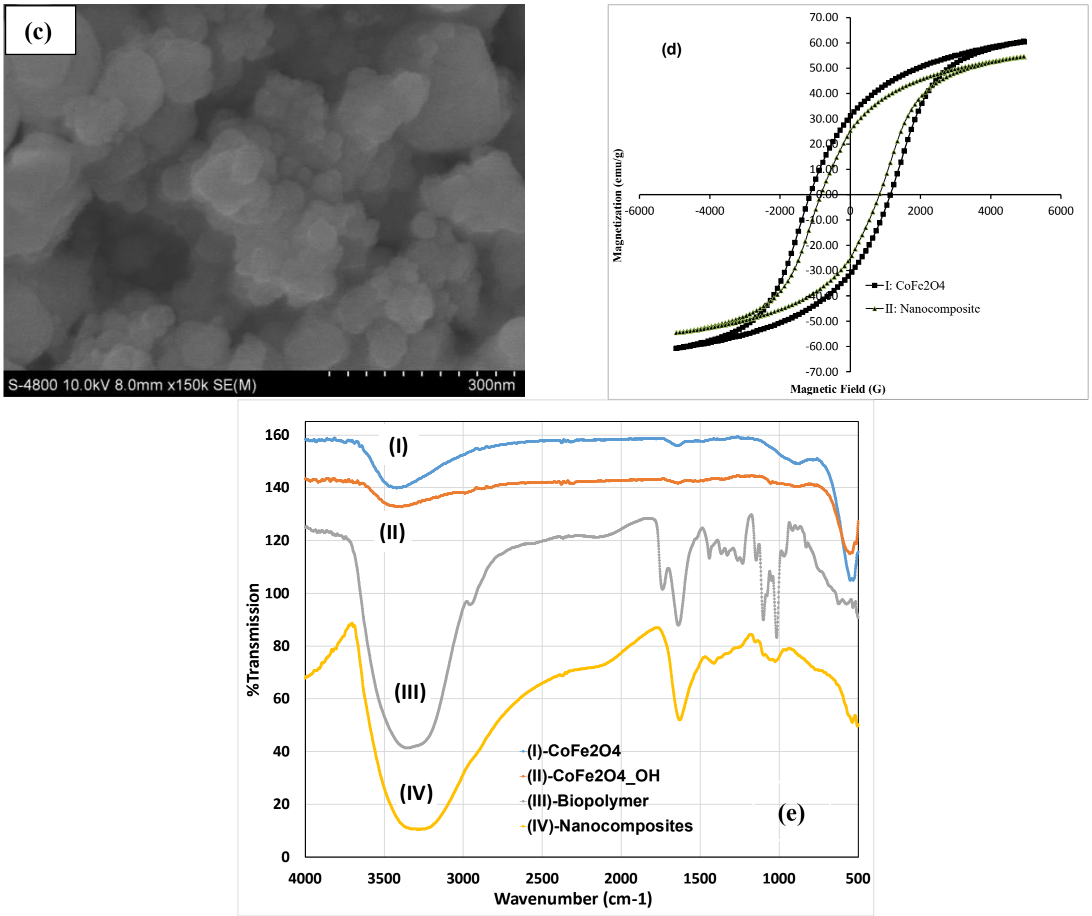


Figure 3. Characterization of magnetic nanoparticles and nanocomposites: (a) XRD results (CuKα- radiation) of $CoFe_2O_4$; (b) SEM image of $CoFe_2O_4$; (c) SEM image of nanocomposites; (d) Hysteresis curve; (e) FT – IR spectra of adsorbent materials

3.3 As(III) treatment efficiency via adsorption

The efficiency in treating As(III) of various materials was demonstrated in Figure 4a in which the adsorption yield was in the order Nanocomposites > Orange peel biopolymer > magnetic nanoparticles. Even though the magnetic nanoparticles have their own adsorption capability owing to the presence of hydroxyl groups, the ready aggregation of the particles reduces their available adsorption surface, resulting in their lowest adsorption capacity. Meanwhile, it is assumed that the mechanism of As(III) treatment using the polymer-based materials is mainly adsorption in which the As(III) adsorbs onto the polymer materials via interaction with their functional groups, mainly –OH groups [1]. Therefore, the combination between biopolymer and magnetic nanoparticles might result in the presence of more –OH groups which lead to higher adsorption capacity. Hence, it is straightforward that the As(III) treatment yield of biopolymer-based nanocomposites was highest due to the combined adsorption of biopolymer and well-dispersed magnetic nanoparticles. In addition, as mentioned previously, another importance role of magnetic nanoparticles in the composite is to easily collect the materials after treatment for reuse.

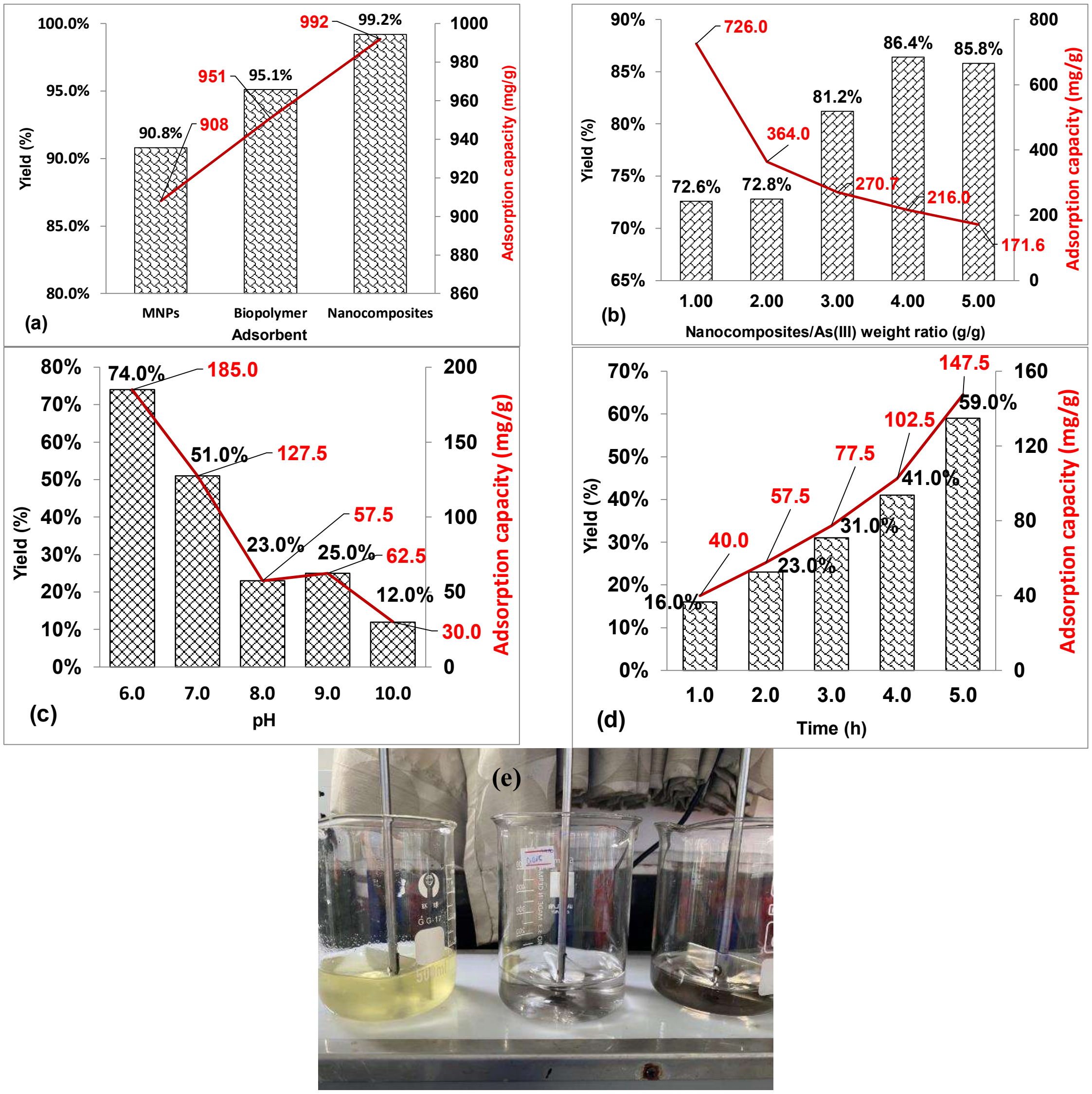


Figure 4. As(III) treatment efficiency: (a, e) Capability to treat As(III) of biopolymer, –OH enriched magnetic nanoparticles and nanocomposites; (b) Effect of nanocomposites/As(III) weight ratio; (c) Effect of pH; (d) Effect of contact time. Volume: 100 mL; absorbent dose: 0.5÷2.5 g/L; pH: 6-10; room temperature; contact time: 1÷5h; agitation speed: 120 rpm

In addition, results showed that when increasing the concentration of nanocomposites adsorbent from 0.5 to 2.5 g/L, with a fixed dose of As(III) of 0.5 g/L, the highest efficiency of As(III) treatment was obtained when the mass ratio of nanocomposites adsorbent/As(III) reached 4/1, corresponding to used dose of nanocomposites adsorbent of 2.0 g/L (Figure 4b). When the nanocomposites/As(III) mass ratio increased from 1.0 to 4.0, the As(III) treatment efficiency increased from 72.6% to the highest value of 86.4%. Usually, the As(III) treatment efficiency increased by increasing the dosage of the adsorbent. This is due to the increasing number of accessible active sites of the adsorbent for adsorption [29, 30]. Surprisingly, when increasing further the ratio to 5.0, the adsorption capacity slightly decreased. This could be attributed to the hindrance in approaching the adsorbent surface of the adsorbate if the density of the adsorbent is too high. It was also observed in this study that the Arsenic adsorption efficiency was highest, around 74%, at pH 6.0. When pH solution increased to 10, As(III) treatment efficiency dropped sharply to 12.0% (Figure 4c). This observation could be explained that the adsorption could not be performed at pH solution less than 6.0 because in acidic environment, Arsenic mainly exists in the form of neutral H_3AsO_3 reducing the ionic interaction with the adsorbent surface [30]. This result is consistent with the previous study which also used biopolymer based nanocomposites materials for arsenic removal [1]. Figure 4d displays the effect of contact time on As(III) removal efficiency in which the efficiency increased almost linearly with the augmentation of the contact time.

The advantage of using magnetic nanocomposites materials is to be easily collected by magnets after usage (Fig 5a), then be reused for treating As(III) for subsequent times with similar conditions. Results of the recovery and probability of reusing nanocomposites materials were presented in Figure 5b, showing that after the fourth cycle, As(III) treatment efficiency was significantly reduced. This might be assigned to the incomplete elimination of As(III) forming the complex with the nanocomposites during the recovery process or could be due to the dispersion of part of the biopolymer into the wash water.

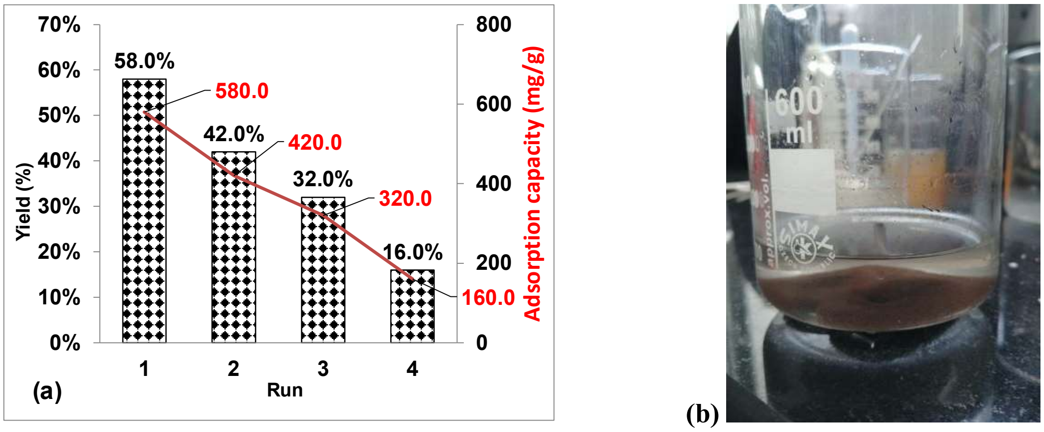


Figure 5. (a) Reusability of nanocomposites and (b) Treated sample after settling by magnet. Volume: 100 mL; absorbent dose: 0.4 g/L; initial As(III) concentration 0.1 g/L; pH 6; room temperature; contact time: 5h; agitation speed: 120 rpm

4 CONCLUSIONS

In conclusion, successfully synthetic magnetic nanomaterials based on Orange peel biopolymer and magnetic $CoFe_2O_4$ nanoparticles were found to be capable of treating As(III) in aqueous solutions. It was also found that the treatment efficiency varied with factors including the nanocomposites/As(III) weight ratio, pH, contact time. In fact, the As(III) adsorption was saturated at 0.4 g/L nanocomposites when carrying out the treatment for 5hrs under stirring at 120 rpm. At the same time, with saturation magnetisation of 54.59 emu/g and magnetic resistance of 4940.06 G, the biopolymer nanocomposites are considered as soft and superparamagnetic material. These properties facilitate the nanocomposites to work at ambient temperature as well as to be easily recovered by an external magnetic field. In this study, the materials could be reused for 4 times. With all these findings, Orange peel biopolymer based magnetic nanocomposite is a potential sustainable adsorbent to treat As(III) in polluted water.

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XỬ LÝ As(III) TRONG NƯỚC BẰNG VẬT LIỆU NANOCOMPOSITE TỪ TÍNH TRÊN NỀN POLYME SINH HỌC CHIẾT XUẤT TỪ VỎ CAM

Tóm tắt. Nước ngầm nhiễm Arsen đang là vấn đề được quan tâm hiện nay bởi tác động của nó đến môi trường và sức khỏe con người. Nhiều phương pháp đã được sử dụng để xử lý vấn đề này. Trong nghiên cứu này, vật liệu nanocomposite từ tính được sử dụng để xử lý As(III). Vật liệu nanocomposite từ tính được chế tạo bằng cách kết hợp các hạt nano coban siêu thuận từ ($CoFe_2O_4$) vào nền polyme sinh học được chiết xuất từ vỏ cam. Trong đó, các hạt nano từ tính được điều chế bằng phương pháp đồng kết tủa và sự hình thành nanocomposite được thực hiện với sự hỗ trợ của khuấy từ. Các phương pháp phân tích như: quang phổ hồng ngoại biến đổi Fourier (FT-IR), nhiễu xạ tia X (XRD), quét kính hiển vi điện tử (SEM) và từ kế mẫu rung (VSM) được sử dụng để kiểm tra đặc tính của vật liệu thu được. Vật liệu sau đó được sử dụng để xử lý As(III) trong nước sinh hoạt. Kết quả cho thấy, vật liệu nanocomposite có thể hấp phụ tới 99.2% As(III) (với nồng độ ban đầu của As(III) là 1.0 g/L, lượng vật liệu sử dụng 1.0 g/L). Sau quá trình xử lý, vật liệu nanocomposite dễ dàng được tách ra khỏi dung dịch bằng phương pháp gạn từ tính do đặc tính siêu thuận từ của vật liệu, quá trình xử lý và tái sử dụng vật liệu được thực hiện một cách thuận lợi.

Từ khóa. nanocomposite, từ tính, vỏ cam, polyme sinh học, siêu thuận từ, As(III), tái sử dụng.

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