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Synthesis and characterization of activated carbon from biowaste-walnut shell and application to removal of uranium from waste

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ABSTRACT: The aim of this study is to synthesize and characterize an economical and environmentally-friendly adsorbent with high adsorption capacity. For this purpose, the walnut shells (*Juglans regia* L.) were chemically modified using sulfuric and citric acids, separately. After pyrolysis and synthesis of activated carbon (AC), the optimization of conditions at the preconcentration/removal step was performed using parameters such as pH and contact time for uranium in the model solutions. The measurements were carried out by inductively coupled plasma-mass spectrometry (ICP-MS). From the shapes of the BET isotherms, it may be stated that activated carbon exhibit type I. It was found that the surface area and total pore volume of the activated carbon were 696.6 m2/g and 0.35 mL/g, respectively. The adsorption capacity was found to be 220 mg/g. It was found that the optimum pH is 6.0 for preconcentration/removal using AC obtained by sulfuric acid as chemically-modifier. The optimized method was applied to determination of U at ng/mL levels in the model solutions.

Keywords: Preconcentration; adsorbent; toxic element; biomass.

INTRODUCTION

The problem of uranium pollution continues to increase due to the increased interest to nuclear energy. Especially, the incomplete removal of uranium from soil and water has led to a significant threat for human health because of its toxicity, accumulation, and persistence in nature. Therefore, it is necessary to develop a rapid and sensitive method for accurate determination of uranium. Because of the limitations in sensitivities of ICP-AES and flame AAS, preconcentration methods are commonly used (O'Connell et al., 2008; Bicim et al., 2016). Unlike some trace metals such as copper, lead, cadmium, and nickel (Yaman and Bakirdere, 2003; Yaman, 2001), the sensitivity of uranium by FAAS is particularly too poor for trace uranium determinations because of formation refractory oxides in the flame. In state of the use of high temperatures and a rich nitrous oxide–acetylene flame, poor sensitivity is achieved for uranium, approximately 50 mg/L for 1% absorbance. A typical example, the nitrous oxide–acetylene flame provides an absorbance reading of about 0.1 for 1000 mg/L uranium (Santos, et al., 2010). If the adsorption capacity of any adsorbent is large enough and non-expensive, the preconcentration material can be applying as also removal mediator (Liu, et al., (2017). Many different techniques including chemical precipitation, membrane separation, ion exchange, and adsorption strategies have been used for

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this purpose (Feng et al., 2011; Das, et al., 2016a; Das, et al., 2016b; Das, et al., (2016c). Among those methods, the adsorption approaches is regarded as the most promising one. A large number of materials have been used as adsorbents, and the criteria for easily accessible, low-price, effective and ecofriendly properties have high importance in their selections (Metilda et. al., 2004; Metilda et. al., 2007). Activated carbon (AC) as adsorbent has been widely used in the preconcentration and removal processes due to its high surface area, large porosity, well-developed internal pore structure, and a wide spectrum of functional groups on the surface (Yaman et al., 2015; Lu et. al. 2017; Ma, et al., 2020). In order to reduce the cost of AC preparation, AC can be prepared from agricultural biowastes (Anastopoulos, et al., 2019). Among the other agricultural biosorbents, walnut shell can be examined because its food crops are widely grown in different countries and consumed worldwide (Jahanban-Esfahlan, et. al., 2020). Compared to physical activations, the chemical activation process for preparation of AC takes place at a relatively lower temperature between 500 to 800 °C and producing with higher carbon yields (Hashemi et al., 2019). The properties of surface functional groups on AC may be modified by suitable thermal or pre- and post-chemical treatments to improve its adsorption performance (Ozdemir et al., 2014; Mattson et al., 1969). Acidic treatment has some advantages including cost effectiveness, ease of operation, and energy saving. So, sulfuric, citric, phosphoric and similar non-oxidizing acids were generally used (Ma, et al., 2020). Briefly, the chemically modified biowastes are candidate as they offer the highest attainable enrichment factors with relatively rapid separation and low cost (Feng et al., 2011).

In this study, a high adsorption capacity of AC was firstly achieved using walnut shell as a raw material after modifying with sulfuric and citric acids, separately, and pyrolysis at 500 °C. After characterization, the AC obtained was successfully applied to preconcentration/removal and determination of uranium in the model solutions, at ng/mL levels.

MATERIAL AND METHODS

The walnut (*Juglans regia* L.) shells were provided from a local market in Elazig city, Turkey. The samples were washed thoroughly with distilled water and then dried at 100 °C in an oven for 12 h. Then, samples were ground using grinding equipment and sieved by sieve of 325 meshes (lower than 52 micron). All chemicals used were analytical reagent grade. A stock solution of uranyl ion was prepared by dissolving uranyl nitrate [$UO_2(NO_3)_2.6H_2O$] purchased from Merck (Germany) in sulfuric acid and diluting to 50 mL with distilled water. This solution was diluted daily to preparing the model U solutions (between 10-50 ng/mL). Other chemicals including HNO_3, H_2SO_4, Citric acid, HCl, NaOH and buffer solutions of CH_3COOH/CH_3COONa were prepared from their ultrapure reagents (Merck). For all the experiments, ultrapure water was obtained using Millipore Q UV3.

The infrared spectra were performed to characterize the molecular structure of the AC obtained. The spectra were obtained by transmittance using KBr pellets and analyzed using a Fourier Transform-Infrared Spectrophotometer-FTIR (Perkin Elmer FT-IR-USA) in the region between 400 and 4000 cm^{-1}. An electron scanning microscope (SEM) (Zeiss EVO MA10) was used to evaluate the morphology of the adsorbents. The surface area, total pore volume, and pore size distribution analyses were measured by Micromeritics Asap 2020 Surface Area and Porosity Analyzer (the Brunauer–Emmett– Teller (BET)) method. For this purpose, 90 mg of material were heated to 300 °C under vacuum for approximately 3 h. Subsequently, the process of adsorption and desorption of N2 was performed. The surface area and pore volume were calculated using the standard Brunauer, Emmett and Teller (BET) and a pore size was obtained using the method of Barrett–Joyner-Halenda (BJH). A Digital pH meter 100 (Cyberscan) was used for pH measurements. An Inductively Coupled Plasma-Mass Spectrometry (Perkin Elmer Elan9000) was used for determination of uranium.

A 5.0 grams of dried walnut (*Juglans regia* L.) shell powders were chemically modified using H_2SO_4 (10%, w/v) and citric acid (1 M), separately, as described in Figure 1. After washing the modified sample by distilled water up to pH=6, the pyrolysis procedure was followed by heating up to 500 °C at a rate of 10 °C min^{-1} and maintaining at this temperature for 1 h, under N_2 flow. It has been established by several authors that the temperatures around 500 ^{o}C are most suitable to obtain optimum properties of the activated carbons in case of acid treatment of agricultural materials (Ozdemir et al., 2014; Mattson et al., 1969). Raising the activation temperature to 500 ^{o}C has a positive influence on the raw material with a generation of developed porosity. After activating by HCl of 15%(w/v) for 1 h, the sample was washed to neutralize (about pH=6). Afterthat, the AC obtained were dried and used for adsorption of uranyl cation.

A 0.05 g of the AC obtained was added to the model uranium solutions of 150 mL and the pH of these solutions were adjusted to the required values (between 3.0-6.0) by addition of diluted acid and base solutions and then 15 mL of buffer (pH=6) solution (10 mL for 100 mL)) were added. The solutions were stirred in ranges of 15-60 min. After filtration, the uranyl ions remaining in filtrate solutions were determined. The detailed scheme steps were given in Figure 1.

RESULTS AND DISCUSSION

Before and after modifications, FT-IR spectra of the synthesized AC were obtained (Figure2). In the spectrum of raw walnut shell, characteristic bands are observed at 3432, 2928, 1733.2, 1629.0, 1503, 1458.9, 1383.2, 1323, 1244.5, 1156.2, 1052 and 781 cm^{-1}. The broad band about 3400cm^{-1} associated with –OH stretching vibration of hydroxyl functional groups. The bands at 1600 cm^{-1} are usually assigned to C=C aromatic ring stretching vibration as well as C=O stretching vibrations of ketones, aldehydes, lactones or carboxyl groups. There are significant changes in the spectra from raw walnut.

Shell to activated carbon product. Unlike the spectra for the raw walnut shell, the prepared activated carbon demonstrates smooth spectra with less absorption peaks. The spectra proved the carbonization and activation process have eliminated most of the functional groups and created an aromatic ring or C=C stretching vibration that reveals around 1400 cm^{-1}. In the literature, it was reported on the structure of activated carbon, the predominant functional groups containing oxygen are ketone carbonyl groups (Mattson et al., 1969; Garten et al., 1957; Qian et al., 2015). The bands at 1156 cm^{-1} correspond to the presence of sulphonate group introduced by the high temperature (500 ^{o}C) activation with sulfuric acid. Further, it was seen that conjugation was obtained, and the U can be adsorbed on those groups. The increase in the peak around 1400 reveals the conjugation. When it acts as an adsorbent, there are some adsorption interaction including hydrogen bonding and electrostatic attraction between these functional groups and adsorbate, which imply toward wider adsorbate and larger adsorption capacity.

Synthesis of Activated Carbon

Biomass, walnut shell
+
Washing, drying and sieving
+
1-Mixing by H_2SO_4 of 10%(w/v) for 8 h/
2- Mixing by citric acid of 1M for 1 h and followed NaOH of 0.1M for 1 h
+
Washing with distilled water to neutralize (about pH=6)
+
Pyrolysis up to 500 ^{o}C at inert N_2 gas
+
Activating using HCl of 15%(w/v) for 1 h
+
Washing with distilled water to neutralize (about pH=6)
+
drying
+
Grinding and sieving

Preconcentration/Removal Procedure

0.05 g of Activated Carbon
+
The model Uranium solution (150-300 mL)
+
Buffer solution (10 mL for 100 mL solution)
↓
Stirring for 50 min
↓
Centrifugation
↓
Measure the filtrate

Fig. 1. Schema of the synthesis of activated carbon and preconcentration/removal processes

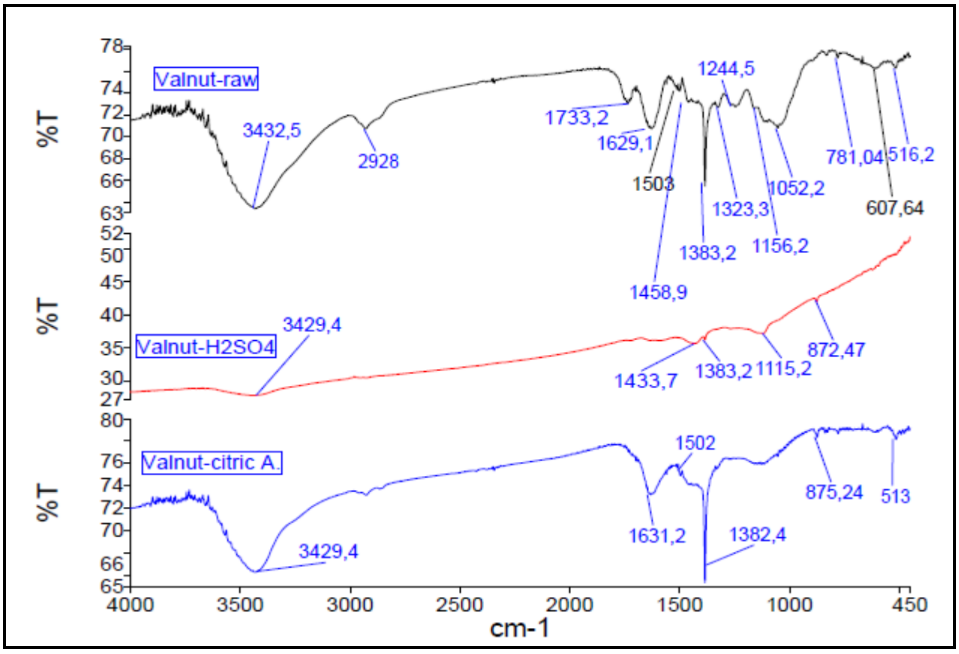

Fig. 2. FTIR spectra of walnut for its raw and the ACs using chemically modifier.

The surface morphologies of the obtained AC from walnut shell were investigated by scanning electron microscope (SEM). The SEM photographs of the samples are given in Figure 3. Figure 3a shows that the carbonized walnut shell possesses a discontinuous bulk structure. The images from Fig. 3b to Fig.3e show that a discontinuous, rough, and irregular surface originates from large cavities on the surface. The rough surfaces and large cavities denote a large specific surface area for adsorption. Figure 3f shows that large number of cavities was filled by adsorption of uranium metal on activated carbon. From the SEM results, it was found that there are holes and cave type openings on the surface. As a result, the surface area has definitely increased that are available for the adsorption.

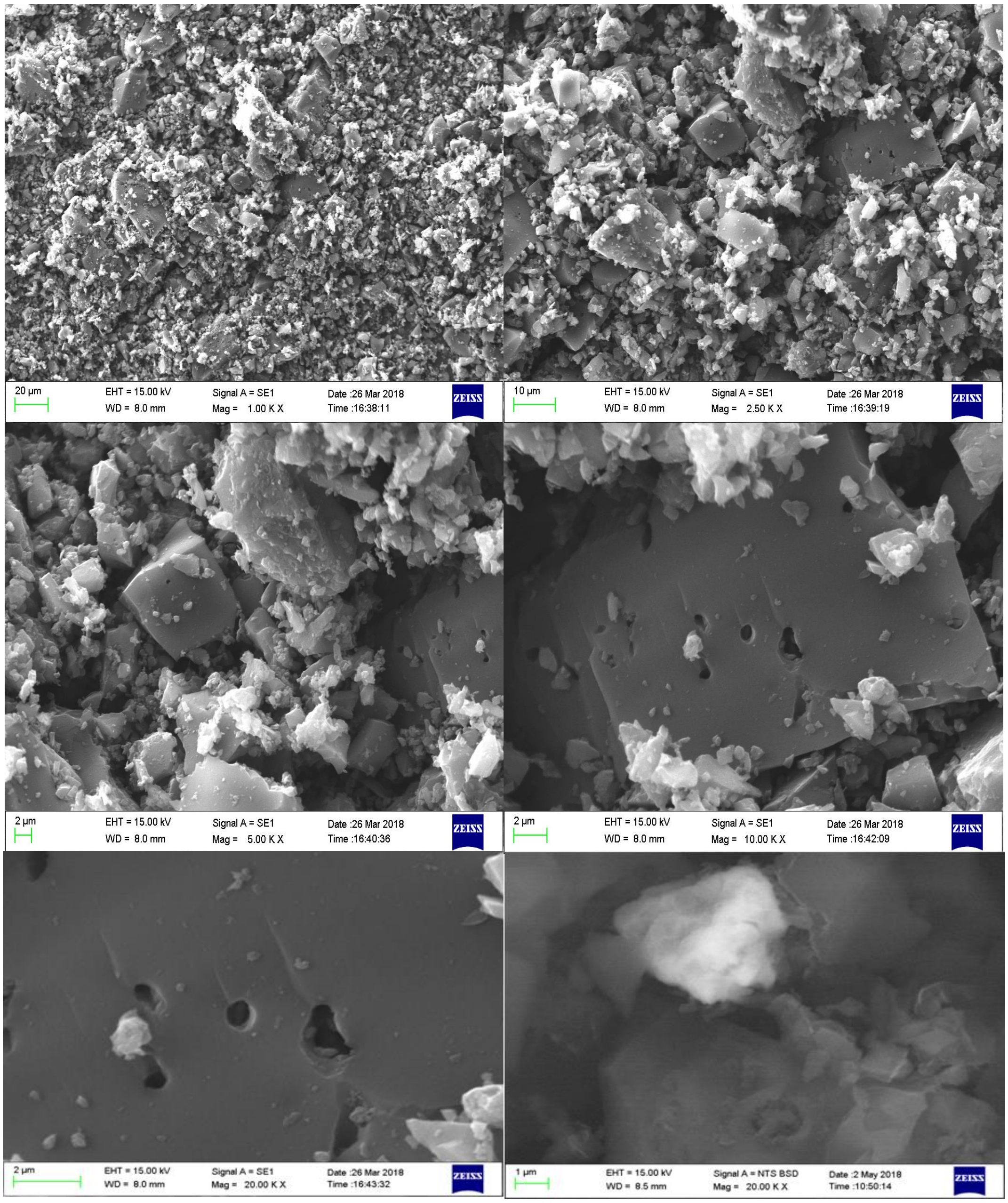

Fig. 3. Investigation of surface structure of Walnut Shell + H_2SO_4 modification by SEM (a) Before Uranium adsorption at 1000X zoom, (b) Before Uranium adsorption at 25000X zoom, (c) Before Uranium adsorption at 5000X zoom, (d) Before Uranium adsorption at 10000X zoom, (e) Before Uranium adsorption at 20000X zoom, (f) After Uranium adsorption at 20000X zoom.

The adsorptive capacity of activated carbon is related to its surface area and pore volume. Surface area and pore volume are enhanced during the activation processes. The shape of the adsorption isotherm can provide qualitative information about the adsorption process and the extent of the surface area available to the adsorbate. The BET isotherm of the activated carbon developed at different temperatures and impregnation ratios are shown in in Figure 4. From the shape of the isotherm, it may be stated that activated carbon exhibits type I and that graph depicts monolayer adsorption. At low relative pressures, a rapid increase is observed in the adsorption–desorption isotherm, which is followed by a nearly horizontal plateau at higher relative pressures, indicating highly microporous solids. The absence of hysteresis loop indicates the absence of mesoporosity, and suggests that the AC obtained contains mostly micropores with only a small contribution of mesopore (Qian et al., 2015; Ezra et al., 2019). It was found that the surface area, total pore volume and pore size of the AC obtained were 696.6 m^2/g, 0.35 mL/g, and 10.07 A^o, respectively.

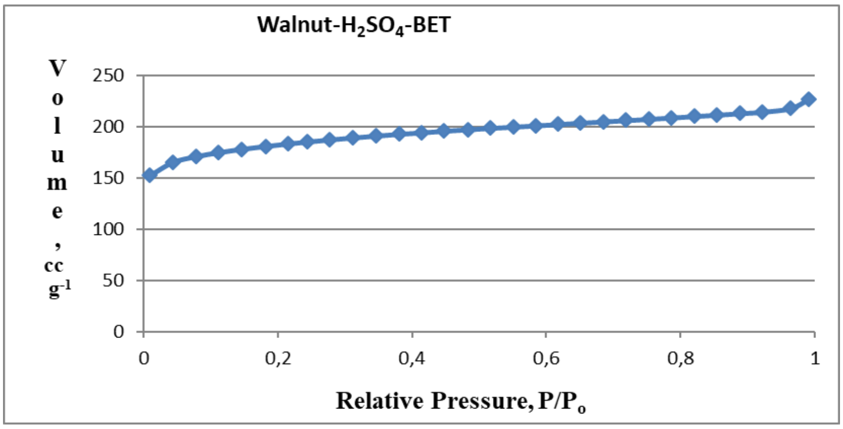

Fig. 4. The N_2 adsorption isotherm of the activated carbon.

The pH of solution plays an important role in adsorption step because the pH affects the surface charge of adsorbent and degree of ionization of the adsorbate. Figure 5 shows that the optimum pH value in adsorption step 6.0 for the AC obtained by H_2SO_4 as chemical modifier and the stirring time of 50 min at room temperature. At the studied conditions, the relatively lower recovery (lower than 40%) of U was obtained for citric acid as chemical modifier-walnut shell-AC. So, the studies were only examined for H_2SO_4-modifier-walnut shell-AC. On the other hand, the pH of the solution determines the different ionic forms of the U. The U is a cationic form around the optimum pH=6.0 which belongs to the positively charged species such as UO_2OH^+ and UO_2^{2+} in solution. The dominant adsorption interaction mechanism is electrostatic interactions between metal ions and the AC having amphoteric character. Thus, depending on the pH surface of the AC might be positively or negatively charged. At higher pH, the negatively charged surfaces are benefit for cationic ion adsorption by electrostatic interactions. As a result, the lower recoveries of U at acidic pH are probably due to the presence of H^+ competing with the cationic species of U for adsorption sites. When the pH value increases, the surface of the activated carbon becomes negatively. Thus, the adsorption capacity of the U reaches maximum at pH 6.0 and it was used in the subsequent studies as 6.0±0.2.

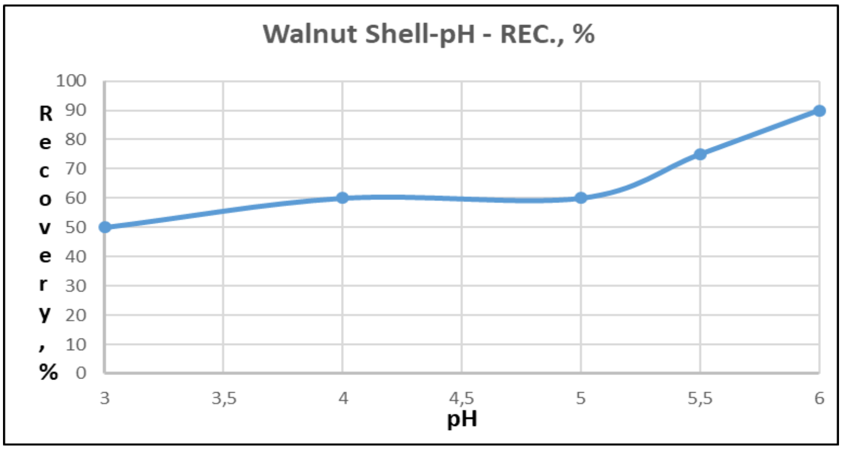

Fig. 5. Effect of pH on the recovery of U.

The effect of stirring time on the preconcentration/removal of uranium at pH=6.0±0.2 using H_2SO_4-modified AC shows that 45 min is enough (Fig. 6). Hence, 50±5 min for stirring time was employed in subsequent studies. On the other hands, the increase of the initial volume of solution up to 300 mL did not affect the quantitative recovery of uranium. So, calibration curve was also obtained for 300 mL of initial solutions.

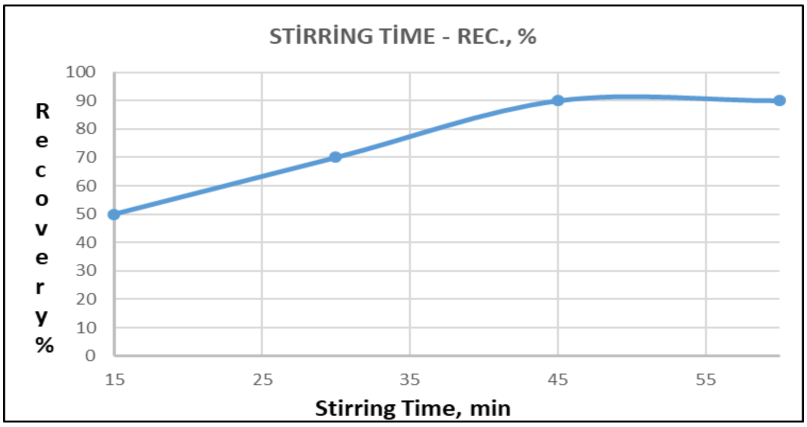

Fig. 6. Effect of stirring time on the recovery of U.

All the results are based on the average of triplicate readings for each standard solution. A recovery test was performed by determining the spiked concentration in the samples in which 10 µg/L of uranium was added. The recoveries of uranium from the model solutions were 90±3% which could make the procedure a reliable method. Related with the interference studies (the values in parenthesis are concentrations as mg/L), Fe (5), Ca(50), Mg(15), Al(2) and Zn(2) metal ions were added to the model U solutions before preconcentration/removal step. The results showed that those ions do not affect quantitative determination of uranyl ions from solutions. To determine adsorption capacity, 300 mL solutions of 50.0 mg U/L were preconcentrated using 0.05 g of the AC. It was found that the adsorption capacity was 220 mg/g. Under the optimum conditions described above, the calibration curve was linear over the concentration range 1.0–100 µg/L of uranium using 300 mL of the model solutions that can be used in real environmental analysis. The LOD and LOQ were found to be 0,030 and 0,090 µg/L^{-}.

The applicability of the method was

tested for the model U solutions. The WHO's, U.S and EU-member states' Health Drinking Water Guidelines fixed the maximum uranium concentration in drinking waters as <10 to 30 μg/L (World Health Organization 2011; EU 98/83/ECD, European Commission Directive 1998; U.S. Environmental Protection Agency 2009). Because the sensitivity achieved are lower than the permitted U levels in natural waters, the ground, river and lake water samples can be subjected to uranium analysis by employing the developed preconcentration method. Yaman et al. (2011) have reported U concentrations in ranges of 37-110 μg/L in Lake Van Water, 0.09-0.18 μg/L in water of Dicle river, and 2.0-4.0 μg/L in seawater from Black sea and Mediterranean sea, using ICP-MS (Yaman et al., 2011). Metilda et al. (2014) determined 1.59 μg/L U in seawater from southwestern coast of India using ion-imprinted polymer and Arsenazo III spectrophotometric method (Metilda et al., 2014). In another study, they obtained the lowest concentration that could be determined by their method as 5.0 μg/L U after enrichment factor of 200 times (Metilda et al., 2007). On the other hand, Kolpakova (2014) reported U concentrations up to 3000 μg/L in water of West Mongolian Lakes, China (Kolpakova, 2014). Singh et al. (2009) found 2.3 μg/L U in tap water using the octadecyl-silica membrane disks and spectrophotometry (Singh et al., 2009). Lemos and Gama (2010) synthesized UO_2^{+2}-ion imprinted polymer and achieved to determine LOD of 5.0 μg/L U (Lemos and Gama, 2010). Their calibration curve was linear over the concentration range 20–100 μg/L of U using Amberlite XAD-4 resin functionalized with β-nitroso-α-naphthol and Arsenazo III spectrophotometric method. Bicim and Yaman (2016) reported the LOD and LOQ for U as 0.021 and 0.070, respectively, using ion imprinted polymer-ICP-MS (Bicim and Yaman, 2016). Compared to the other studies, the developed method in this study is more advantaged than the others taking into consider LOD=0.03 μg/L except one.

Although walnut shells were used to remove some toxic elements from water matrices, their usage for uranium is limited (Jahanban-Esfahlan et. al., 2020). The adsorption capacity of the developed adsorbent (220 mg U/g) was found to be much higher in comparison with the most of other adsorbents (Jahanban-Esfahlan et. al., 2020). For example, the adsorption capacities were reported for banana peels-nanosorbent as 27-34 mg/g (Oyewo et. al., 2016), for polyethylene fiber adsorbents named the AF series as 3.9 mg/g after 56 days contact time (Das et. al., 2016), for polyethylene fiber adsorbents named the AI series as 3.35 mg/g after 56 days of exposure in seawater (Das et. al., 2016), for porous aromatic framework-based adsorbents named the PAF as 4.81 mg/g after 42 days contact with U-spiked seawater (Yue et. al., 2016), for poly(imide dioxime) sorbents as 4.48 mg/g after 56 days of seawater exposure (Das et. al., 2016), and for amidoxime-grafted activated carbon as 191.6 mg/g after 48 h exposure to U (Liu et al., 2017). Liu et al. (2017) found adsorption capacity as 71.85 mg U/g using biochar derived from poplar catkins (Liu et al., 2017). Ma et. al. (2020) studied adsorption of uranium on phosphoric acid-activated peanut shells and found 120.9 mg/g (Ma et al., 2020). Briefly, it can be concluded that the obtained adsorption capacity in this study is higher than the most of other studies published in literature (Anastopoulos et al. 2019) except one using peanut shell-AC (387.6 mg/g) (Yi et. al., 2018). This may be attributed to their usage very high U concentration up to 250 mg/L for the determination of adsorption capacity.

CONCLUSION

In this study, the activated carbon with high performance to removal of U was prepared from walnut shell modified with both sulfuric acid activation. It was found that the surface area and total pore volume of the activated carbon were found up to 696.588 m^2/g and 0.35 mL/g, respectively. The synthesized activated carbon was characterized by FTIR, SEM and BET techniques. The developed method was applied for the determine and removal of U (UO_2^{2+} ions) from the model aqueous solutions by using the optimized conditions. The LOQ was found to be 0.09 µg/L. The adsorption capacity was found to be 220 mg/g. Consequently, uranium levels in the river and ground waters can be determine by this method because the sensitivity achieved are lower than the permitted U levels in natural waters in addition to its ability for removal of U from waste.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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