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Adsorptive removal of Cu (II) from aqueous solution using succinylated *Artemisia vulgaris* seed hydrogel

Sajid Khan
Faculty of Sciences, Superior University Lahore, Lahore 54000, Pakistan
sajidinshal@gmail.com
Aiman Ishfaq
Faculty of Sciences, Superior University Lahore, Lahore 54000, Pakistan
aiman.ishfaq.sgd@superior.edu.pk
Rashid Mahmood
Faculty of Sciences, Superior University Lahore, Lahore 54000, Pakistan
rashid.mahmood.sgd@superior.edu.pk

Abstract

Hydrogels are the three-dimensional and hydrophilic biomaterials. This is because of their ability to be chemically modified due to the presence of chemically modifiable groups like carboxylic (--COOH) and hydroxyl (-OH). In the current research, an acetylated hydrogel of the seeds of *Artemisia vulgaris* (AVH) was extracted, succinylated, and saponified to obtain an eco-friendly and a cost effective polysaccharide-based sorbent (Na-AVH) medium for Cu(II) removal from polluted water. The effect of the five parameters such as sorbent dose (10-90 mg/100 mL), initial Ni(II) ion concentration (50-290 mg L^{-1}), contact time duration (5-120 min), temperature (298-338 K), and pH (1-10) on the adsorption capacity were examined. Cu(II) was eliminated by a large percentage using the Na-SH adsorbent. The highest Ni(II) uptake was observed at sorbent 30 mg/100 mL, initial concentration of Cu(II) ions = 130 $^{-1}$, contact time = 30 min, temperature = 298K, and pH =6. Therefore, these conditions were chosen as the most suitable one for the Cu(II) removal from polluted water.

Keywords: Hydrogel, esterification, copper removal, optimization, adsorption

1. Introduction

This is because of the many human activities and unregulated industrialization, which has caused a constant leakage of heavy metals into the water. Consequently, heavy metal ions present long term dangers of water contamination besides damaging the stability of the world as a whole [1, 2]. Copper is among the most hazardous and common heavy metals pollutants of fresh water [3]. Due to the common use of copper batteries and pigment, it (Cu(II)) can thus be commonly found in domestic and industrial waste [4]. Moreover, the mining activities, burning fossil fuels, leather tanneries, metallurgical activities, etc. are exceeding the recommended limits of Cu(II) in drinking water as recommended by the World Health Organization, leading to the different life threatening diseases [5]. Purification of water is therefore seen as another solution to the world drinking water crisis.

In the last 20 years, a number of conventional physical and chemical methods have been reported to remove Cu(II) both in surface and ground water. Because of economic advantages, speed, efficiency and regenerability selectivity, easy manipulation and the broad adsorbent material variety; adsorption preceded by ion exchange seemed most likely methodology [6-10]. They have widely been used as drug delivery vehicles, water purification sorbent due to their high water absorbancy, non-toxicity nature of the polysaccharide based hydrogels [11-26]. The *Artemisia vulgaris* (AVH) has been acetylated and polymerized [14, 15] which also exhibits some thermal stability and is also called a promising candidate in controlled delivery of drugs. AVH can therefore even be saponified after being esterified with succinic anhydride. It is expected that this product (Na-AVH) will be an ion-exchange sorbent of its own ions to the polluted water heavy metal ions.

This paper describes the synthesis, purification, and characterization of Na-AVH as a Cu(II) ions sorbent using a polluted water. Current work aims to investigate and compare the

effect of pH, the sorbent dose, the initial Cu(II) concentration, the contact time and temperature on the uptake capacity of Na-AVH. The goal is to achieve the maximum adsorption condition to extend this material to strip off toxic heavy metal ions and dyes in personal care and water setting, to save live.

2. Materials and Methods

2.1. Materials

A. vulgaris seeds were acquired in the local market where they were collected in the District, Sargodha in Pakistan. The complete chemicals and reagent used in this study were of analytical grade (> 99% pure) and in undetermined quantities. All reagents and chemicals employed in this work were bought in Riedel-de Haen, Germany and Sigma-Aldrich, USA.

2.2. *A. vulgaris* Hydrogel Isolation of Seeds

After post sieving and screening, which follows to remove unwanted substances, the *A. vulgaris* seeds were soaked at 50℃ in DW using a muslin cloth and a spatula to remove the excretion of mucilage on the seed (AVH). DW was used to wash the as-obtained AVH to get rid of polar impurities, followed by triple washing with *n*-hexane to get rid of the non-polar substances. AVH was obtained and then used as a starting material to get AVH through centrifugation (4000 rpm) and drying in vacuum oven at 60℃ and then screen grinded at no. 60 mesh and then stored in air-tight container under vacuum desiccator [19].

2.3. Synthesis of Adsorbent

Sodium salt of AVH adsorbent was made through the process put in place by [27].

2.4. Studies on Sorption

In order to manage the best experimental conditions to allow Na-AVH to adsorb as much as possible Cu(II) in polluted water, a series of batch sorption measurements were performed at

room temperature. It was observed that sorption with Na-AVH depended on various operating conditions, including sorbent dose (10-90 mg/100 mL), initial Cu(II) ion concentration (50-290 mg L^{-1}), contact time duration (5-120 min), temperature (298-338 K), and pH (1-10).

A 1000 ppm (1000 mg L^{-1}) stock solution of Cu(II) salt was prepared by first dissolving the salt in deionized water using a 1-L volumetric Erlenmeyer flask. This solution was mixed thoroughly to make sure that it dissolved completely. Based on this stock, working solutions were prepared through suitable dilution and their pH was adjusted through 0.1 M HCl or 0.5 M NaOH with constant monitoring through the use of a calibrated pH meter. After that, 100 mL of 130 mg L^{-1} Cu(II) solution was added to 250-mL Erlenmeyer flasks and 30 mg of the optimized Na-AVH sorbent was added. The flasks which were sealed were left in a shaking thermostat of 200 rpm to ensure that sufficient time was provided to facilitate the contact of the Cu(II) ions and the sorbent. The suspensions were then filtered and the concentration of Cu(II) that remained in the filtrate was analyzed using FAAS. The adsorption capacity at equilibrium (q) was determined by using given below equations.

$$q_e = \frac{C_i - C_e}{m} \times V$$

$$Percentage\ uptake = \frac{C_i - C_e}{C_i} \times 100$$

3. Results and Discussion

3.1. pH Impact

Sorption experiments were performed between pH 1-10 at optimum sorbent dosage, initial concentration of metal ions, contact time and temperature in order to observe the influence of variation in pH of Cu(ll) removal by Na-AVH in contaminated water. As illustrated in Figure 1, the shift of Cu(II) is not observable at pH 3.0 of less in Na-AVH. This could be because at

low pH, -COOH functional groups of Na-AVH are protonated and transformed to acidic form of sorbent (S-AVH) with ineffective ion-exchange capacity as well as lower sorption capacity. At low PH, the competition of H(I) ions and Cu(II) to overtake the sorption sites Na-AVH is observed. The sorption of H(I) is more preferable than that of Cu(II), which reduces the adsorption capacity of Na-AVH. Interestingly, the Na-AVH surface is negatively charged in high pH region because it is due to de protonation of Na-AVH that prefer the sorption of Cu(II). This is possibly why the rate of Cu(II) species sorption by the Na-AVH provided by crude water is speedier. The adsorption of Cu(II) was reduced smoothly with the pH above 6, and the outcome was similar to other metal ions that had been previously reported which explained the outcome as a result of colloidal precipitation of $Cu(OH)_2$ in a basic environment [28, 29]. Optimal sorption was achieved at pH 6. Thus, pH 6 was chosen as the best pH in future tests.

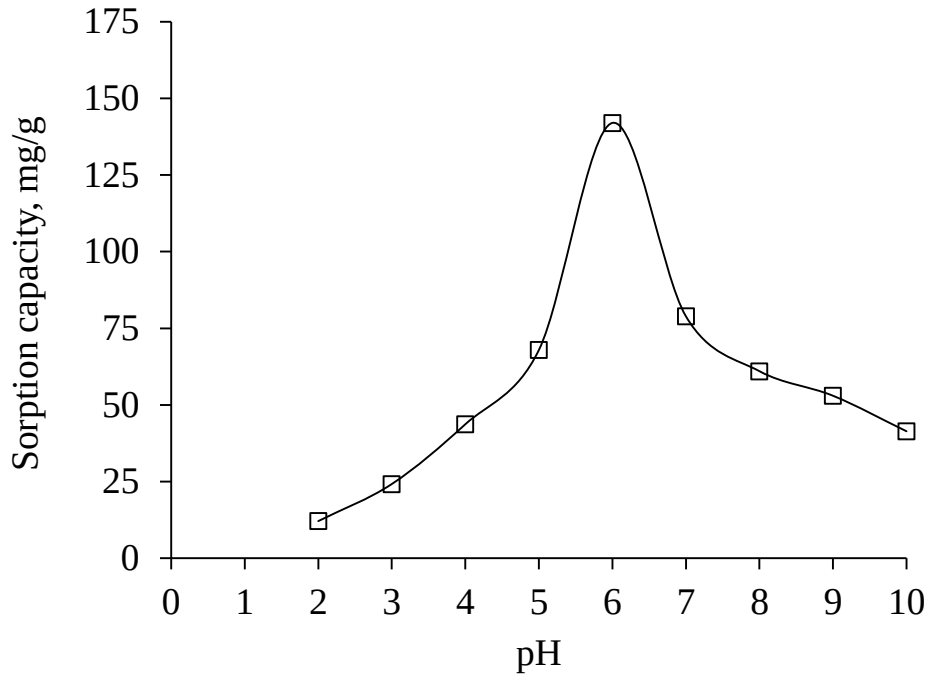

Figure 1. Effect of pH

3.2. Sorbent Dose Effect

Since excessive amounts of sorbents can have a strong impact on the ability of holding heavy metal ion, this is attributed to the fact that the ability of holding heavy metal ions by sorbents can significantly be affected. It is hence, worthwhile to establish optimum sorbent concentration at which it can be capable of extracting maximum quantity of metal ions of their polluted water. The impact of Na-AVH dose on the sorption behavior of Cu(II) in the polluted water was determined. It was observed that the effect of dose 10-90 mg (Na-AVH) with optimum initial Cu(II) concentration (130 mg L^{-1}/100 mL) at optimum pH = 6 and temperature = 298 K, and time = 30 min is used to check whether Na-AVH had any influence on the removal of Cu(II) in the contaminated water. This may be because of the increase in the surface sites of sorption with increased quantity of Na-AVH from 10 mg to 50 mg and resultant increased percentage uptake of Cu(II). Nonetheless, the sorption capacity levels off following an embarkation of optimal dosage of Na-AVH (50 mg; i.e., 50-90 mg) (Figure 2). It may be proposed because of aggregation of Na-AVH leaving its surface sites unsaturated as the sorption process proceeded and did not have an active sorption site to get Cu(II). The competition among the active sites of Na-AVH to the Cu(II) sorption is on the increase as the concentration of Na-AVH increases; It has been reported that the rise in the concentration of Na-AVH could also be attributed to the reduction in the adsorption of Cu(II) [30, 31]. Consequently, it is suggested that 50 mg Na-AVH can be employed in using the current material in industrial usage and all subsequent Cu(II) sorption experiments.

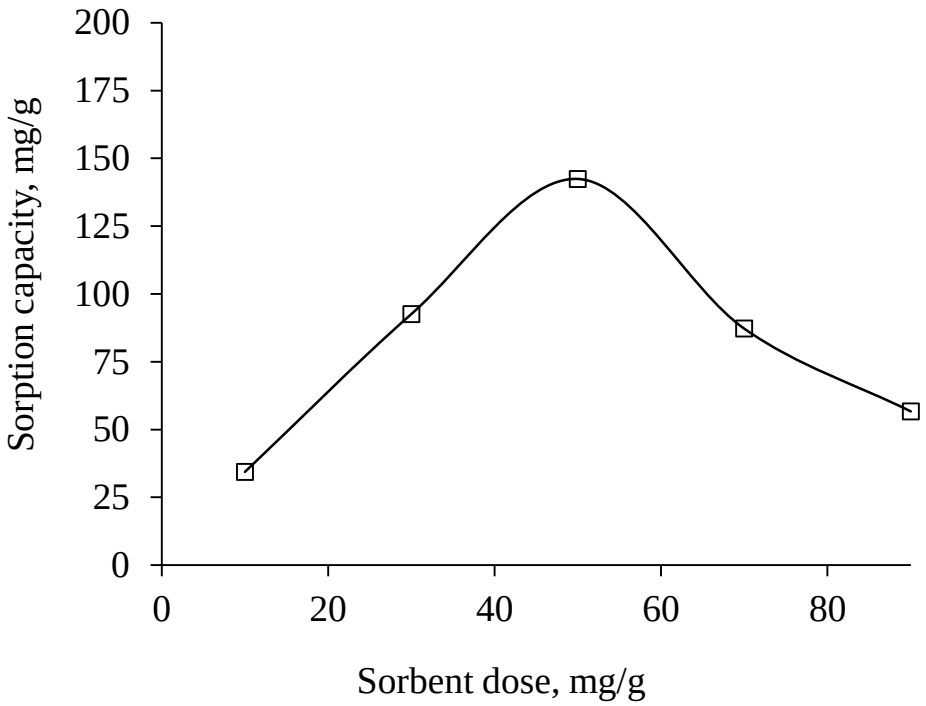

Figure 2. Effect of sorbent dose

3.3. The Effects of the Initial Concentration of Metal Ions

Na-AVH sorption capacity was determined at a concentration of 50-290 mg L^{-1} Cu(II) to clarify the mechanism involved in sorption. A rise of the sorption capacity was recorded with the increase in the initial concentration of Cu(II) brought about by the increase in its concentration to 130 mg L^{-1}. The optimum concentration of 130 mg L^{-1} was considered as the maximum level, where the most Cu(II) ions were removed. This is possibly due to the increase in the mass-transfer driving power at the increased concentration of Cu(II), this facilitates the more productive interaction of the sorbate and the active sites of the sorbent. At this stage further increase in sorption is probably restricted by saturation of available binding sites. This encouraged the diffusion of Cu(II) of the bulk solution to Na-AVH surface which lead to improved sorption. Nevertheless, the capacity of sorption will be constant at an optimum level of (130 mg L^{-1}) as depicted in Figure 3 where the remaining vacant sites are not available to accept Cu(II). This could be explained by the fact that the majority of the Cu(II) ions formed

in the course of the sorbate are in reaction with Na(I) of the Na-AVH, which leads to an increase of the Cu(II) concentration. But in the completely saturated phase more exchangeable Na^+ of Na-AVH will be substituted by Cu(II) of sorbate and Cu(II) will be on Na-AVH surface. It comes to be realized that the sorption capacity finally assumed a stabilization position [32, 33].

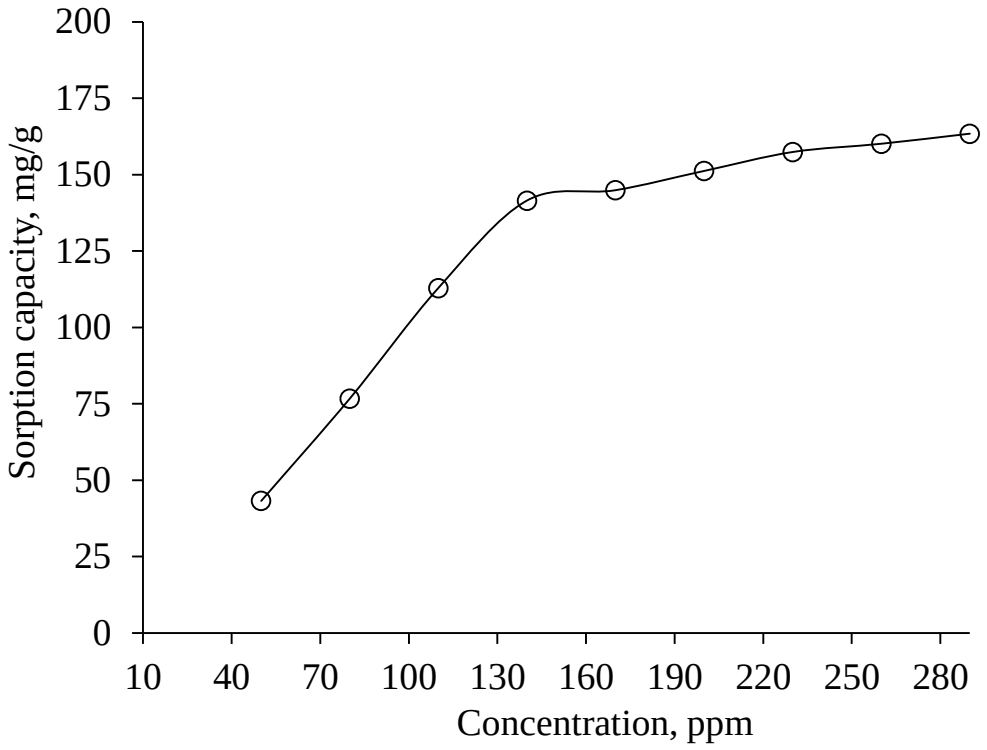

Figure 3. Effect of initial concentration of metal ions

3.4. Contact Time Impact

Effect of contact time between sorbent and sorbate was investigated to derive the sorption activity of Na-AVH, as well as to derive sorption kinetics to learn the sorption mechanism. Initially, the sorption rate of Cu(II) on Na-AVH was relatively high (Figure 4) and the sorption capacity of polluted water was rising or reaching maximum values after 30 min only. The optimal time to reach the maximum equilibrium sorption capacity was 30 min and it means that the sorption equilibrium with Cu(II) is possible within 30 min. That is, the sorption rate became concentrated on saturation after 30 min. This is likely due to the comparatively high

degree of concentration of Cu(II) at the onset of the sorption process and a big portion of these adsorbate hydrated would have readily become at slightly low free energy as it came in contact with the surface of Na-AVH containing many active sites could be loaded. However, as the amount of sorption of Cu (II) increased, the concentration of Cu (II) dropped, and thus a high repulsive force was developed between Cu(II) ions and aqueous solution. Therefore, Na-AVH active sites were decreased, and no Cu (II) left to be absorbed. Finally, fixed and retarded sorption rate fixed [8-10].

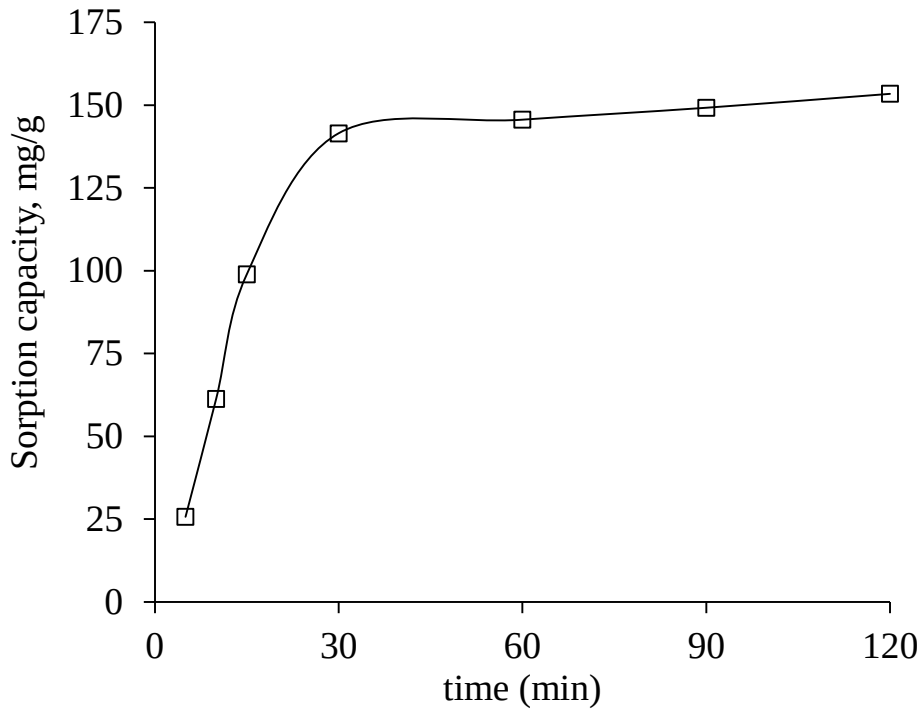

Figure 4. Effect of time

3.5. The Impact of Temperature

To gain further insight into the thermodynamic behavior of the adsorption process, the temperature impact on the sorption capacity of the Na-AVH in the removal of Cu(II) in contaminated water was therefore, studied systematically. As illustrated in Figure 5, the adsorption equilibrium of Cu(II) reduced slowly as temperature varied between 298 K and 338 K that is, a low temperature is good in adsorbing Cu(II) on Na-AVH. Such a change in

temperature of the sorption process indicates that the adsorption process is exothermic in nature. The Cu(II) ions does not have kinetic energy and high opportunity to form stronger interaction with the functional groups and active binding sites on the surface of the Na-AVH at a lower temperature. The reduction in the molecular mobility would enhance the stability of surface complexes either through an enhanced attraction in response to an electrostatic or ion-exchange reaction, depending on the dominant mechanism. However, when the temperature increases, there are a number of factors that interact, in a co-operative fashion, to decrease sorbate-sorbent interaction. They proposed that the faster and faster kinetic energy brought about by the rising temperature, enhances the mobility and rate of diffusion of the Cu(II) ions in the solution and consequently decreases the time that each ion stays on the sorbent and, as a result, reduces the formation of complexes. Moreover, high temperature is able to partially disrupt or activate active sites of binding of Cu(II) that has likely some effect on the overall attraction of Na-AVH to the metal ions. In this way, it is possible to consider adsorption process to be exothermic in nature and it goes hand in hand with common tendencies of thermodynamics that the more the temperature, the less preferable is heat-absorbing sorption. The thermodynamic parameters ($\Delta G°$, ΔH^o, and, ΔS^o) also justify this concerned the fact that a combination of them proved that Cu(II) sorption onto Na-AVH was spontaneous and exothermic [33, 34].

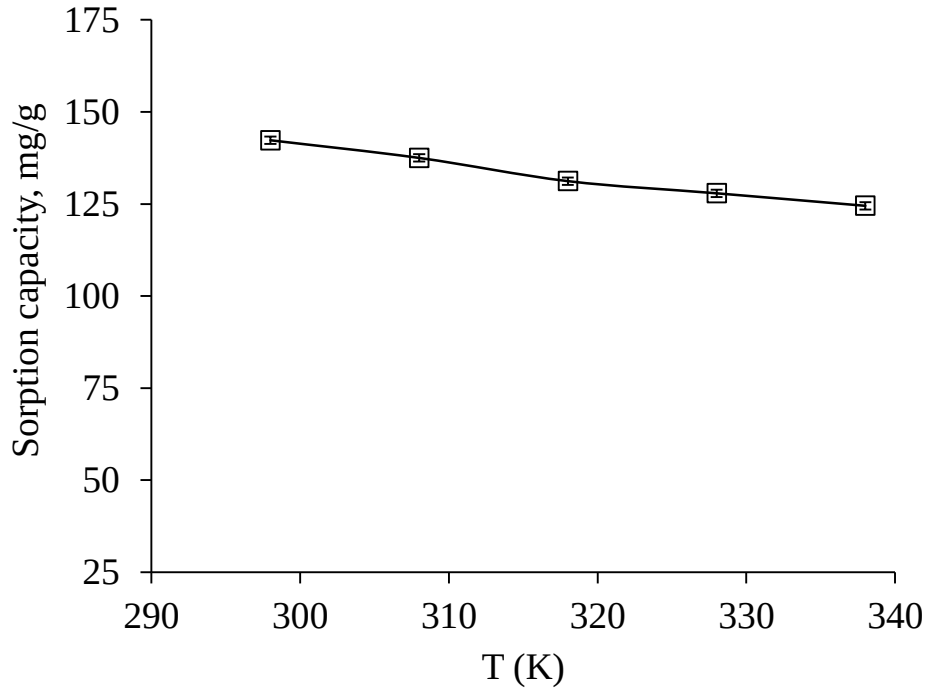

Figure 5. Effect of temperature

4. Conclusion

Upon esterification of the *A. vulgaris* seed extracted hydrogel (AVH) into succinylated S-AVH, Na-AVH was obtained and examined to take up Cu(II) in the polluted water. After 30 min, the equilibrium of the Cu(II) sorption was attained. At 50 mg of Na-AVH adsorbent, contact time of 30 min, 298 K, and 6 pH, the maximum Cu(II) uptake was achieved using Na-AVH adsorbent. More so, since the AVH has a moiety of Na-succinate, thus, it can be concluded that Na-AVH is an ideal ion-exchanger in the remediation of Cu(II) from polluted water. To sum up, the sorbent Na-AVH is a promising super-sorbent in the uptake of heavy metals, therefore, such a material can be utilized in the commercial sector to make water pure as well.

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