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Synthesis of Activated Carbon from Sugarcane Bagasse and Application for Mercury Adsorption

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ABSTRACT: With the growth and development of chemical plants, the amount of mercury released in wastewater has increased. Mercury in wastewater contains harmful compounds which are hazardous to the human health and living organisms. Therefore, its removal from wastewater is significant. There are various techniques or methods available for removing mercury from aqueous solutions. This study focused upon the removal of mercury from aqueous solution with commercial activated carbon and activated carbon from sugarcane bagasse. Activated carbon produced from sugarcane bagasse was used as adsorbent. This adsorbent was used to remove mercury from aqueous solution. For this purpose, first, the optimal mercury solution pH for mercury removal was obtained. Effective parameters such as contact time, initial concentration of mercury, adsorbent dose and agitation speed were investigated. The mercury adsorption was increased when the mass of activated carbon was increased. Increasing the initial mercury concentration leads to decrease in mercury adsorption efficiency. The results of experiments indicated that the speed of the stirrer was not considered to be an effective factor in the mercury adsorption. Experiments were also carried out on a commercial activated carbon. Adsorption results obtained for sugarcane bagasse activated carbon were compared with commercial activated carbon. The adsorption efficiency was increased as the contact time was increased. Finally, the experiment was carried out on water samples released from South Pars platforms. In addition to the mercury removal, other heavy metals removal such as lead and cadmium were also carried out.

Keywords: Activated carbon, Sugarcane Bagasse, Mercury, Adsorption efficiency, South Pars.

INTRODUCTION

As industry develops, discharge of high-concentration metal ion-containing waste from the battery, dye, petrochemical, and mining industries is steadily rising. In recent years, much attention has been drawn to heavy metals contamination, because these substances have a detrimental effect on health (Yao et al., 2016). The presence of heavy metals can cause neurological disorders, cellular aging and carcinogenesis. Unfortunately, heavy metals were seen in water, soil and wastewater in different parts of Iran. Arsenic was found in some parts of Iran's Kurdistan (Nasrabadi & Shirani Bidabadi, 2013). Significant amounts of As, Cd and V were observed in the soil of Ahvaz oilfield (Afkhami et al., 2013). Mean concentration of As, Sb, Hg and Zn was analyzed to be 234.9, 19.6, 24.2 and 476.7 ppm in soil samples of Zarshuran gold

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mine (Karbassi et al., 2014). Nasrabadi et al. (2016) investigated the correlations amongst total suspended solids concentration and turbidity with total pollutant concentration to evaluate the toxic metals (Ni, Pb, Cd, Cu, Zn, Co, As, Sr) in Haraz basin (Iran). Arsenic, Cadmium and Zinc were detected in the soil sample from the vicinity of National Iranian Lead and Zinc Company in Zanjan Province (Karbassi et al., 2016).

Mercury and its compounds have been shown to have an effect on the central nervous system and the liver. One of the largest sources of mercury pollution on earth is mercury-dependent artisanal, small-scale gold mining (Esdaile & Chalker, 2018). Mercury metals are produced by many industries such as coal combustion, oil refining, gas refining, chloralkali plant and waste incineration (Worthington et al., 2017). Therefore, in order to protect the environment and human health, the removal of heavy metal ions from water is very important (Saad et al., 2016). In the EU, standard mercury dosage in drinking water and wastewater has been reported 1μg/L and 10μg/L, respectively.

A variety of methods such as ion exchange, chemical treatment, reverse osmosis technology and membrane filtering have been cited for mercury removal (Yao et al., 2016). These methods include problems such as high operating costs, lack of full access to heat, high energy, high levels of chemicals, and toxic sludge production (Hadi et al., 2015). One of the oldest methods for the removal of mercury is the application of sulfide from industrial effluents. This method is based on the fact that all metals have shown extremely low solubility in the form of a metal sulfide (Wu et al., 2011).

The application of nanoparticles is another method of removing mercury from aqueous solution (Zhang et al., 2013; Zho et al., 2017). Adsorption is the transfer of compounds from the liquid phase onto the surface of solid phase. In recent years, special emphasis has been placed on the preparation of the activated carbon from agricultural byproducts and waste material. Agricultural wastes are among the most abundant, accessible and renewable resources available to produce activated carbon. The activated carbon obtained from coconut shell (Zhu & Kolar, 2014), almond nutshell (Bouaziz et al., 2017), olive stone (Alslaibi et al., 2013), palm stone (Alhamed, 2009), walnut nutshell (Nazeri et al., 2015), pistachio (Siddiqi & Ahmad, 2017), hard skin of apricot stone (Djilani et al., 2015), rice bran (Ogataa et al., 2015), sugarcane bagasse (Esfandiari et al., 2014; Khoramzadeh et al., 2013), grape pomace, licorice residue, corn wood, peel of plants such as cocoa, coffee, cinnamon, palm, oak were produced and used for removal of heavy metals (Dawlet et al., 2013; Abu Ismaiel et al., 2013; Wahby et al., 2011).

Activated carbon derived from pine cones was chemically optimized using H_3PO_4 and 2,6-Diaminopyridine. A rise in temperature caused a decrease in mercury adsorption and an increase in the time and initial concentration of mercury increased the mercury adsorption. The maximum mercury adsorption capacity from this activated carbon was obtained at pH 7 (Mokhtari & Faghihian, 2016).

Activated charcoal derived from corn was used as a biologically modified activated carbon. Charcoal and modified activated carbon showed a higher percentage of adsorption (Tan et al., 2016). The adsorption of mercury from aqueous solutions was investigated using sugarcane bagasse. The effective factors such as pH, contact time and temperature were investigated. The maximum adsorption capacity was 35.71 mg/g. The mercury removal rate at pH 4 was obtained 94.584 percent (Khoramzadeh et al., 2013).

Nayak et al. (2017) studied the sawdust activated carbon. Dawlet et al. (2013)

evaluated the mercury adsorption using activated carbon from sheep bone. To remove cadmium ions, mercury and zinc from aqueous solution, activated carbon produced from wild bamboo with a specific surface area of 608 m^2/g were studied (Gonzalez, 2014). Hadi et al. (2015) reviewed the studies on the adsorption of mercury using activated carbon. Activated carbon from Fox nutshell was produced for mercury adsorption.

South Pars gas field is located 100 km from the Coast of Asaluyeh in Bushehr Province. On these platforms, water is separated from the hydrocarbon mixture and released into the sea. Regarding the increasing number of operating platforms, the necessity of investigating the released water into the sea is of high significance in terms of environmental pollution. According to the available standards, the maximum amount of mercury available in the water released into the sea should be below 0.2 ppm. The amount produced increases as the reservoir lifetime and the removal of heavy metals becomes more significant.

In this research, activated carbon from sugarcane bagasse was produced. Phosphoric acid was used as an activation agent in the production of activated carbon. Chemical structure, particle shape and special surface of activated carbon were analyzed. Experiments were carried out to adsorb mercury with activated carbon. Finally, the mercury was separated from the drained water of south Fars gas field by activated carbon.

MATERIAL AND METHODS

Mercuric chloride salt (purity 99.5%), 1 N sodium hydroxide solution, 1 N chloride acid, phosphoric acid 85% and commercial active carbon were purchased from Merck Company.

Perkin Elmer FT-IR was used to investigate the structure of activated carbon. To measure the specific surface area of activated carbon, the Gemini 2375 porosimeter manufactured by Micromeritics Inc. was used. The SEM analysis was performed with Phenom ProX scanning electron microscope (SEM) from Netherlands. Varian Atomic Absorption Spectrometer was used to measure the concentration of mercury.

Sugarcane bagasse as a raw material was used to produce activated carbon. Firstly, the sugarcane bagasse was washed with urban water and then distilled water. Sugarcane bagasse was exposed to sunlight for a few days until it was dried completely. The samples were then milled and sieved. Screened particles between 20 and 30 meshes were chosen. 500 g of raw material was mixed with 1000 mL of phosphoric acid solution 85%. The mixture was poured for 5 h in an oven at 100 °C. Then, the impregnated mixture was placed inside the compression-molded fittings (the elbows used in the plumbing fittings). Afterwards, the open elbow paths were blocked by special caps and placed inside the industrial furnace cylinder and sealed inside it. The air was sucked out from inside the cylinder. The furnace temperature was set to 600 °C for 90 min. Then the cylinder was removed from the furnace and cooled at ambient temperature for 8 to 9 h. The adsorbent was removed from the elbows and washed several times in hot distilled water at a temperature close to boiling point until pH 7 was reached. The adsorbent was placed in the oven at 105 °C for 24 h until it was dried.

1.347 g of mercuric chloride salt was dissolved in 1 L distilled water. Mercury solution (1000 ppm) was prepared for experiments (0.52, 5, 10, 15 and 20 ppm) by dilution with the required concentrations of mercury.

After the mercury solution was prepared at a specified concentration, 50 ml of each solution with a specific concentration was put into a 100 mL Erlenmeyer flask. The pH of the solution was adjusted. A specific amount of sugarcane bagasse

activated carbon was put into each flask. The material inside the Erlenmeyer was mixed at a certain speed. After a specified amount of time had passed, mixing was stopped. The material inside the Erlenmeyer was allowed to sediment for 30 min. The solution was then passed through a filter paper. The concentration of the remaining mercury in the filtered solutions was measured by cold-vapor atomic absorption technique.

RESULTS AND DISCUSSION

The parameters were changed in different experiments and the effect of changed parameters on the amount of mercury adsorption was investigated. In this study, the effect of contact time (10, 20, 30, 45, 60, 90, 120, 180 min), pH (2, 3, 4, 5, 6, 7, and 8), agitation speed (220, 320, 420 rpm), activated carbon mass (0.2, 0.3, 0.5, 0.7 g) and the initial concentration of mercury (0.52, 5, 10, 15, 20 ppm) on the mercury adsorption were studied.

Special surface area is one of the parameters determining adsorption capability in adsorbent. Gemini 2375 porosimeter and Accupyc 1330 pycnometer were used to measure the specific surface area and volume of produced adsorbent porosity. The results of these analyses have been presented in Table 1. According to the results, the adsorbent has a specific surface area. An increase in the level of specificity is indicative of a rise in the presence of activated carbon sites.

FT-IR analysis of activated carbon produced from sugarcane bagasse was shown in Figure 1. In the FT-IR analysis, a spectrum at a wavelength of 3373.41 can be seen which is linked to the OH bond from phenolic and carboxylic groups. The spectrum at a wavelength of 2873.62 indicates the C-H bond in the –CH2 group. The spectrum at a wavelength of 2142.22 indicates a C≡N- bond that belongs to aliphatic isonitriles. The spectrum at a wavelength of 1660.62 indicates a NO2 bond that belongs to nitrate. Spectrometers with a wavelength of 1300 to 1458.74 belong to the P-C, P=O and C-O-H bond. These spectra are related to carboxylic compounds. The spectrum with a wavelength between 1100 and 1300 belongs to anhydrides P-O. The wavelength of 1070.15 can be attributed to the C-N aliphatic bond. The wavelength of 935.75 belongs to the N-O oxime bonds. The wavelengths from 700 to 1050 can be attributed to the P-F and P=S bonds of the aromatic group (Stuart, 2004).

Figure 2 and 3 refer to SEM images of raw sugarcane bagasse and activated carbon produced from sugarcane, respectively. Comparison of the SEM image of raw materials and activated carbon shows that the raw material contains very small pores. Although the activated carbon contains pores, gaps and channels that are much larger than the original material show the effect of activation and carbonization precisely. The high porosity increases the surface of active carbon. Surface area is a very important factor in the adsorption process.

Table 1. Adsorbents properties

Properties	Sugarcane bagasse activated carbon	Commercial activated carbon
Average pore diameter (nm)	31.562	29.5236
Total pore volume (cm^3/g)	0.07358	0.111408
Macropore Volume (cm^3/g)	0.0193	0.0287
Mesopore Volume (cm^3/g)	0.049	0.0789
Micropore Volume (cm^3/g)	0.00521	0.0039
Specific area (m^2/g)	1023	1205

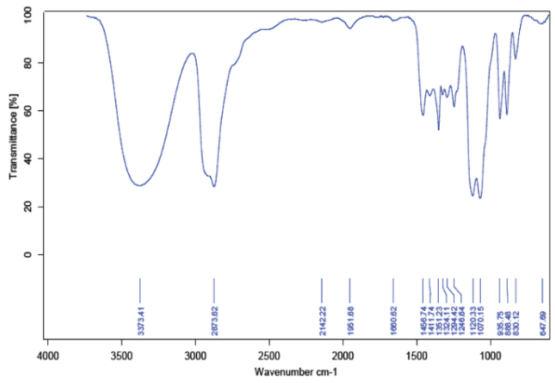

Fig. 1. FTIR spectra of activated carbon prepared by sugarcane bagasse

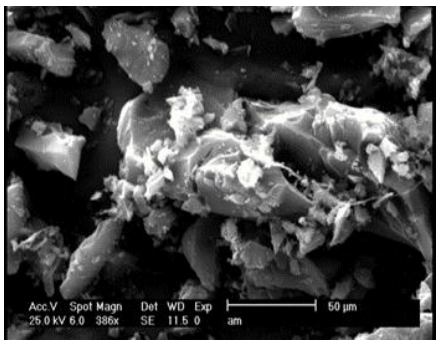

Fig. 2. SEM photograph of crude sugarcane bagasse

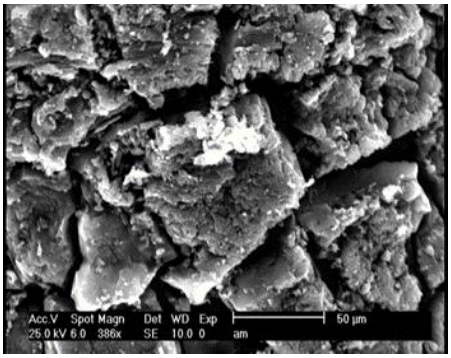

Fig. 3. SEM photograph of activated carbon prepared by sugarcane bagasse.

To determine optimum pH, seven samples with different pH were prepared (2 - 8). To prepare each sample, 50 mL of mercury solution at a concentration of 5 ppm was firstly poured into each Erlenmeyer flask (100 mL). Sodium hydroxide (1 N) and chloride acid (1 N) were used to adjust the pH of mercury solution inside the flask. After adjusting pH, 0.2 gr of activated carbon was poured into the Erlenmeyer and it was placed inside a shaker and the shaker speed was set 320 rpm and 180 min. The shaker was stopped after this preset time period. After 30 min to allow for the sedimentation of the adsorbent particles, the solution was filtered using filter paper. To determine the residual mercury concentration, the removed solution was analyzed. The experiment was repeated with the same experimental conditions for commercial activated carbon. The adsorption capacity (q_e) and adsorption rate (adsorption efficiency) were calculated by Eqs. (1) and (2) (Kobya et al., 2005):

$$q_e = \frac{C_0 - C_e}{m_c} V \quad (1)$$

$$Adsorption\% = \frac{C_0 - C_e}{C_0} \quad (2)$$

where C_o is the initial concentration of mercury, C_e is the concentration of mercury after reaching the equilibrium time, mc is the mass of activated carbon, and V is the volume of the mercury solution.

As shown in the Figure 4, adsorption of mercury has been found to increase with increasing pH value. The amount of adsorption onto the natural adsorbents was increased as the system shifted from an acidic pH environment to a neutral one. This process can be attributed to the impact of the release of OH^- ion and charged carbon which is associated with chemical adsorption with functional groups such as -COOH and the formation of a superficial complex.

Commercial activated carbon showed higher mercury adsorption, because the specific area of commercial activated carbon was higher than sugarcane bagasse activated carbon (Table 1). The optimum pH of sugarcane bagasse was 8. Some researchers reported that loading surface is regarded as an important factor in the adsorption of metals. With increasing the number of OH^- functional groups, competition between mercuric ions and this group of agent's starts and the active sites of the adsorbent are occupied and thus fewer mercury ions are adsorbed. Furthermore, the presence of Cl^- ions and functional groups containing oxygen such as carboxylic acids, phenols and the like lead to an increase in mercury adsorption (Yardim et al., 2003).

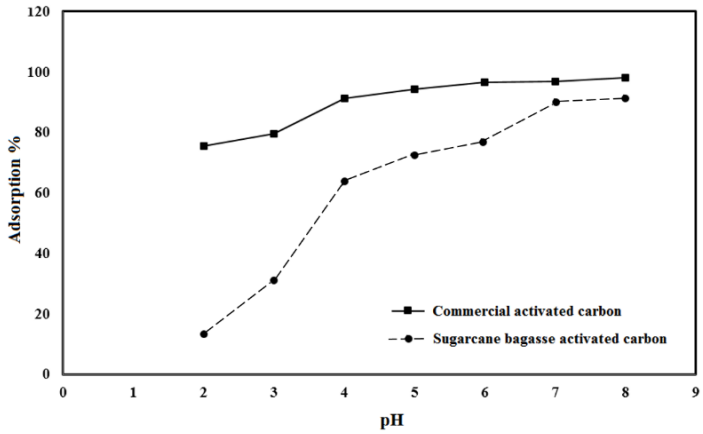

Fig. 4. The effect of pH on adsorption efficiency via commercial and sugarcane bagasse activated carbon at experimental condition; 0.2 g activated carbon, 320 rpm, 180 min.

In accordance with the results of many studies, when the pH was increased the mercury adsorption efficiency was increased (Abu Ismaeil et al., 2013; Tan et al., 2016; Zhang et al., 2005; Kaghazchi & Shamsijazeyi, 2011).

Figure 5 shows the percentage of mercury adsorption by contact time (10, 20, 30, 45, 60, 90, 120 and 180 min). In these experiments, activated carbon used was 0.2 g. The initial concentration of mercury in the solution was considered to be 5 ppm, solutions with pH of 8, and agitation speed 320 rpm. Mercury concentration and mercury adsorption by time with activated carbon produced from sugarcane and commercial activated carbon were summarized in Table 2.

As indicated in Figure 5, the adsorption efficiency of mercury was increased when the contact time was increased until ultimately, equilibrium was reached. The commercial activated carbon reached equilibrium after 45 min. The adsorption capacity of mercury remains virtually unchanged after this time. The equilibrium

time of sugarcane bagasse was 160 min. The reason for this can be attributed to the size distribution of commercial and sugarcane bagasse activated carbon. The size distribution of sugarcane bagasse activated carbon was chosen between mesh 30 and 35. The size of commercial activated carbon was greater than mesh 40. The size of activated carbon was one of the key factors of adsorption (Anoop Krishman & Anirudhan, 2002; Demiral & Gungor, 2016).

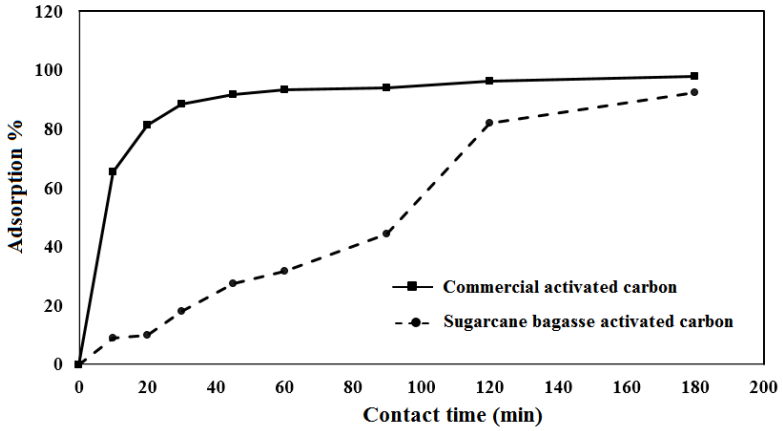

Fig. 5. The effect of contact time on adsorption efficiency via commercial and sugarcane bagasse activated carbon at experimental condition; 0.2 g activated carbon, 320 rpm, pH=8 and C_0=5 ppm.

Table 2. The effect of contact time on mercury removal via commercial and sugarcane bagasse activated carbon at (0.2 g activated carbon, pH=8 and 320 rpm)

Contact time (min)	Sugarcane bagasse activated carbon		Commercial activated carbon	
	q_e (mg/g)	C_e (mg/L)	q_e (mg/g)	C_e (mg/L)
0	0	5	0	5
10	0.1123	4.5508	0.8175	1.73
20	0.12356	4.5057	1.0175	0.93
30	0.2256	4.0976	1.1075	0.57
45	0.3415	3.634	1.145	0.42
60	0.3985	3.409	1.1675	0.33
90	0.5523	2.7908	1.175	0.3
120	1.0236	0.9052	1.2025	0.19
180	1.1547	0.3812	1.225	0.1

Arias et al. (2017) observed a mercury concentration decrease in the first 60 min. 70% of mercury was adsorbed during this time. Khoramzadeh et al. (2013) also reported similar trends in the mercury adsorption. As the time increased in the first 120 min, the adsorption capacity of mercury was also increased and then reached equilibrium (Alslaibi et al., 2013). The adsorption capacity of mercury by walnut activated carbon after 30 min has been fixed (Zabihi et al., 2009). Mercury adsorption on activated carbon with various properties was investigated. The adsorption was very high in the first 120 min, and the adsorption capacity was fixed after 500 min and the system reached equilibrium (Lu et al., 2014). The equilibrium time of mercury with activated carbon produced and improved by 2,6-diaminopyridine was obtained 20 min (Moktari & Faghihian, 2016).

The amount of heavy metals removed from activated carbon produced by Bambusa vulgaris striata was increased as the contact time was increased (Gonzalez, 2014). This trend was also observed in a study by Asuquo et al. on the adsorption of

cadmium and palladium ions by commercial activated carbon (Asuquo et al., 2017). The rate of manganese adsorption by activated carbon was extremely high within the first 25 min. The major portion of manganese was adsorbed at this time range. The system reached equilibrium after 120 min (Esfandiari et al., 2014). The major portion of palladium was removed in the first 20 min and the system reached equilibrium after 180 min (Khoramzadeh et al., 2013). Yang et al. (2015) evaluated the adsorption of chromium with activated carbon produced from Longan seed. The chromium adsorption system reached equilibrium after 30 min.

The effect of sugarcane bagasse activated carbon mass (0.2, 0.3, 0.5 and 0.7 g) on mercury adsorption was studied. The experiments were done at initial mercury concentrations 5 ppm, pH 8, and agitation speed 320 rpm. The results of the experiments are shown in Figure 6. As indicated in Figure 6, the percentage of mercury adsorption was increased when the mass of activated carbon was increased.

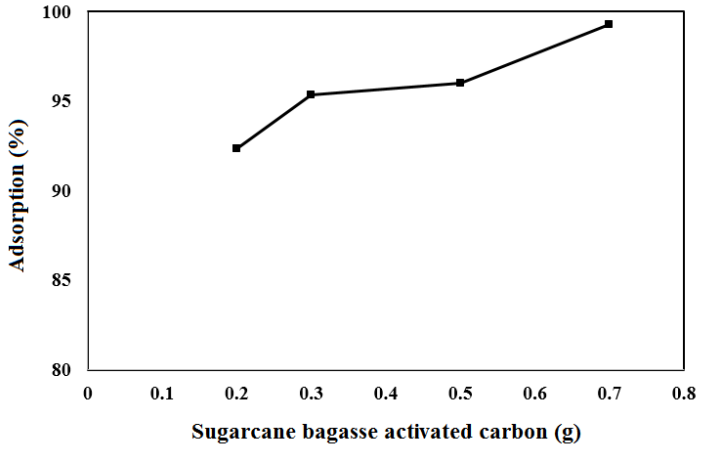

Fig. 6. The effect of sugarcane bagasse activated carbon dose on adsorption efficiency at experimental condition; 320 rpm, pH=8 and C_0=5 ppm.

A rise in activated carbon produced from Bambusa vulgaris striata led to an increase in the percentage of heavy metal adsorption (Gonzalez, 2014). The adsorption rate of mercury was increased with a rise in adsorbance for mercury (Kaghazchi & Shamsijazeyi, 2011). Esfandiari et al. (2014) and Guo et al. (2017) found the same trend for the adsorption of mercury by activated carbon.

Experiments of mercury adsorption were carried out for activated carbon produced from sugarcane bagasse in different agitation speed (220, 320 and 420 rpm). The results of these experiments indicated that the speed of the stirrer was not considered to be an effective factor in the mercury adsorption.

Some experiments have been carried out for activated carbon produced from sugarcane bagasse at different initial concentrations of 0.52, 5, 10 and 20, at pH 8, 180 min of contact time, 320 rpm mixer speed and adsorbent mass 0.2 g. The experimental results indicated that increasing the initial mercury concentration leads to an increase in mercury adsorption capacity and decrease in mercury adsorption efficiency.

This phenomenon can be explained by the fact that in lower concentrations, mercury level was found to be less than on the surface of the adsorbent; however, an increase in the soluble concentration results in a significant reduction of the available sites on the adsorbent compared to mercury molecules, resulting in reduced adsorption. In studies on mercury adsorption, it is

usually observed that the adsorption capacity increased in an approximately linear manner with a rise in initial concentration. The reason may be more related to the possibility of collision between ions or active carbon particles. Changes in adsorption capacity may be related to the fact that all active sites on the activated carbon level were empty and gradient concentration of metal ion was relatively high. Accordingly, an increase in ion adsorption decreases with increasing contact (Zahabi et al., 2009). Kaghazchi and Shamsijazeyi (2011), Gonzalez (2014), Arias Arias et al. (2017) found a similar trend in the adsorption of mercury by activated carbon.

After ensuring the performance of producing activated carbon in the removal of mercury in synthesis solution, the effluent water which was released from the South Pars offshore platforms to the sea after the separation of gas and natural-gas condensate, was tested. The amount of activated carbon was 0.2 g, the amount of solution required per test was 50 ml, the temperature was equal to the ambient temperature and pH was considered 8. The result of this separation is presented in Table 3.

As indicated in Table 3, sugarcane bagasse activated carbon has good performance in metal removal (mercury, cadmium, lead) in effluent water of the platforms. The amount of mercury removal (82.88%) is lower than that used for the aqueous solution made from mercuric chloride. The reason for this is other metal contaminants removal through producing absorbents and reduction of the number of active sites to remove mercury metal. According to the atomic radius of mercury (1.51 Å), lead (1.75 Å) and cadmium (1.51 Å), these heavy metals were removed through sugarcane bagasse activated carbon, since the average radius of cavities in producing activated carbon was much higher than the atomic radius of heavy metals.

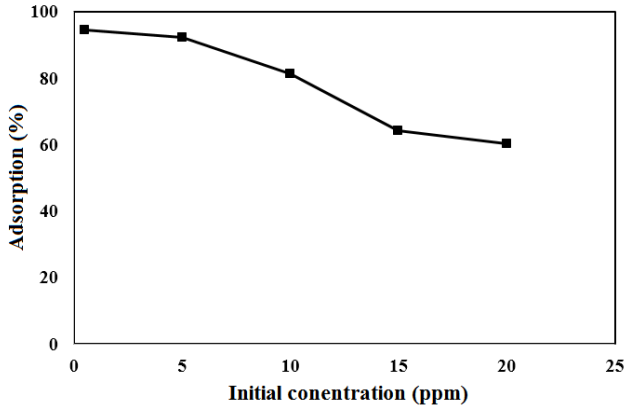

Fig. 7. The effect of mercury initial concentration on adsorption efficiency at experimental condition; 320 rpm, 180 min and 0.2 g activated carbon.

Table 3. Heavy metal removal from the released water into the sea in south pars platforms

Metal	Metal Concentration in effluent water of the platforms (ppb)	sugarcane bagasse activated carbon		
		C_e (ppb)	q_e (µg/g)	Adsorption (%)
Mercury	520	89	107.75	82.88
Lead	2.5	0	0.625	100
Cadmium	12.2	2.5	2.425	79.5

CONCLUSION

Although the price of activated carbon is not high, the activated carbon produced from sugarcane bagasse is valuable because its primary ingredient is the low-cost residue of sugar industry. The results of the experiments indicated that pH 8 is the best pH for removing the highest amount of mercury from aqueous solution for activated carbon produced from sugarcane bagasse. Activated carbon produced from sugarcane bagasse has a lower tendency to adsorb mercury with an acidic pH. The rate of adsorption was increased with increasing pH. The mercury adsorption efficiency was increased with contact time until the system reached equilibrium. Commercial activated carbon and activated carbon produced from sugarcane bagasse reached equilibrium after 60 and 160 min, respectively. Increasing activated carbon caused a rise in the mercury adsorption efficiency. The agitator had no effect on the amount of mercury removal. An increase in the concentration of mercury in aqueous solution led to a reduction in the percentage of mercury removal. The amount of mercury removal from effluent water of the platforms (82.88%) is lower than that used for the aqueous solution made from mercuric chloride. In this study, the mercury removal was 61% at the initial concentration of 20 ppm. Wahly et al. (2011) observed 65% removal at the same concentration with activated carbon from olive stones. Gonzalez et al. (2014) indicated a removal of 78.35% of mercury at initial concentration 10 ppm using activated carbon from Bambusa vugaris striata. The result of experiment in this research shows a removal of 81.43% at the same concentration.

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