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Brick sand particles as an adsorbent in column chromatography to remove heavy metals from solution

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ABSTRACT

The column chromatography method was used with brick sand particles as an adsorbent for the removal of heavy metals (Cr, Fe, and Pb) from the aqueous solution. These metals can pollute water, and soil and also come in contact with the food chain, causing life-threatening health issues in the human body. The pore volume and the specific surface area of the brick sand particles were determined using the standard method which was 44.41% and 29.4 m^2/g respectively. The different concentrations of toxic metals in the water solution were considered to determine the adsorption capacity of the adsorbent. The adsorption methods were able to remove about 94% of Cr, 24% Pb, and 69% Fe from the aqueous solution. From the investigation results, we may conclude that raw brick sand particles can play a vital role as an adsorbent in the treatment of heavy metal-contaminated wastewater. Moreover, the saturated adsorbent can be used as an element of construction materials to prevent further pollution.

Keywords: Heavy metals; Brick sand particles; Adsorbent; Pore volume; Specific surface area

Introduction

Water pollution is a global issue. Due to rapid industrialization, water pollution is a common problem in Bangladesh. Cu, Ag, Cd, Au, Hg, Pb, Cr, Ni, Sn, As, Se, Mo, Mn, and Al are known as heavy metals in environmental pollution when the density is more than the WHO recommended such as for As it is 0.1 µg/l by ICP-MS; 2 µg/l by hydride generation AAS or flame AAS (Musa *et al.* 2013; WHO 2011; Egorova *et al.* 2017; Jarup 2003). Among the metals, some are essential for our body (Duffs 2002)), but become toxic in excess amounts (over the standard level). The metals cadmium, mercury, and lead are highly poisonous to the human body (Emslay 2011). Among the toxic metals, Cr, Pb, and Fe are the most important elements that are polluting the groundwater in Bangladesh.

In Bangladesh, Cr is one of the toxic metals that is polluting our water sources and life treating human health. The tannery industries are one of the main sources of Cr metal pollution in water. In the leather tanning process, 40-30% of unused Cr is ejected as an effluent in water like rivers, lakes, or ponds. As a foreign exchange earner industry, the leather industries are contributing 4% of total exports or 0.5 % of the GDP of Bangladesh (Jothilingam *et al.* 2020). Chromium is also widely used in electroplating, metallurgy, paints, preservatives, pigments, pulp and papers, fire bricks materials, and metal ceramics industries in Bangladesh. Cr (III) and Cr (VI) are the most stable forms of Cr metal in the environment. In water, chromium mainly occurs in two oxidation states Cr (III) and Cr (VI), and related ion forms depending on pH values, redox potential, and the presence of natural reducing agents which are highly soluble, can easily pollute water. Because of teratogenic and carcinogenic properties (Kousalya1 *et al.* 2010), it can create serious life-threatening diseases in human life. The standard level of Cr (VI) is 0.05 mg/L recommended by WHO (WHO Guidelines, 1996). Depending on the pH of the aquatic environment Cr (VI) is generated from monovalent chromate, dichromate, or divalent chromate and it is an oxyanion species (Sankararamakrishnan *et al.* 2006).

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Pb contamination can usually occur by aerosol and inhaling lead-contaminated dust particles. But it can also come from contaminated water and food as well. Pb is a possible carcinogenic substance for the human body, it can also damage the liver, kidneys, brain, nervous system, heart, etc. (Kinuthia *et al.* 2020). The Environmental Protection Agency (EPA) has set an action level for lead in public drinking water at 15 µg/L (micrograms per liter). According to the Department of Public Health Engineering, Bangladesh, the water quality parameters standards for lead and WHO guidelines are 0.05 and 0.01 ppm respectively (Chiroma *et al.* 2014; Ayeni 2014, Kinuthia *et al.* 2020).

Iron in water does not usually present a health risk. Our body needs iron to transport oxygen in the blood. Most iron comes from food since the body cannot easily absorb iron from water directly. Iron may present some concern if harmful bacteria have entered as well. Some bacteria need iron to grow and survive, so they can easily enter our body through iron-contaminated water. If there is iron in the water, it may be harder to get rid of harmful bacteria. Note that one treatment cannot prevent or cure all iron-bacteria-related problems. The recommended iron level in water is usually below 10 mg/L.

In the literature, the researcher reported on many different methods to remove toxic metals from aqueous solution like evaporation, reverse osmosis, zeolites, membrane separation, ion exchange, etc. (Kinuthia *et al.* 2020; Devaprasath *et al.* 2007). Due to the cost, and not complete removal of metals, and toxic sludge as well as some operational complications, many methods have not become popular or successfully used as unique methods (Kinuthia *et al.* 2020; Rahaman *et al.* 2016). To remember the Socioeconomic condition of Bangladesh, this study was targeted on two points: one is cost and the other is a simple operational method that will be economically viable and encourage industrial people to remove toxic metals from industrial effluent.

The adsorption method is the most economical, effective, and easy operational technique the all others (Kinuthia *et al.* 2020; Karim 2024a, Karim *et al.* 2024b). Many types of low-cost and efficient adsorbents have been reported and used for removal of heavy metals like fly ash, brick kilns (Rai *et al.* 1999), peanut hull (Brown *et al.* 2000), teak leaves (Ajamal *et al.* 2001), neem leaves (Sharma *et al.* 2005), Wallastonite (Sharma 2001), bidi leaves (Shrivastava *et al.* 2001), amla dust (Anbalagan *et al.* 2004), Chinese reed (Namasivayam *et al.* 2004), rice husk (Bishoi *et al.* 2004), bagasse (Gupta *et al.* 2004), wheat straw (Chun *et al.* 2004), sawdust (Yu *et al.* 2003). Therefore, if we can ensure the availability of low-cost adsorbent and simply operational-oriented adsorption methods could be more viable to the Socioeconomic status of our country to treat industrial effluent.

Our research goal is to find a highly effective, cost-effective adsorbent and method that will be affordable to use for industrial people for the removal of toxic metals from industrial effluent. In our previous work, we treated brick sand particles with di-0-tolyl phosphoric acid to remove Cr (III) from the solution (Islam *et al.* 2004, Karim *et al.* 2024a). In this research, we take third-class bricks, the bricks were then crushed into small particle sizes and the brick particles were used without any pretreatment like our previous work (Karim *et al.* 2024a). The untreated brick particles were used for the removal of heavy metals like Chromium, Lead, and Iron from aqueous solution by column chromatography method. The performance of brick sand particles to remove heavy metals from the aqueous solution was analyzed by Atomic absorption spectroscopy, Scanning Electron Microscope, and Energy Dispersive respectively.

Materials and method

Bricks particles (from grade 3 brick collected locally), Potassium dichromate (Sigma-Aldrich, ≥99.5%), Iron (II) sulfate heptahydrate (Sigma-Aldrich, ≥99.0%), Lead (II) nitrate(Sigma-Aldrich, ≥99.0%), Double distilled water.

Preparation of hexavalent Chromium solution: Two types of hexavalent Chromium solution (200 ppm and 300 ppm) were prepared using dilution from the standard stock of metal ions. Similarly, Fe and Pb solutions for experimental purposes (100 ppm and 200 ppm for each metal) were prepared for this experimental process.

Adsorbent preparation: Class 3 or 3-grade bricks were collected from a brick field near the University Rajshahi campus, Bangladesh. First, bricks were cleaned by an air blower and then crushed into small sizes by using a locally available crusher machine. The desired brick particles were obtained by using a screening analysis process. To remove moisture from the brick particles, it was kept in the oven for 24 hrs. at 100^{o}C temperature. After removing moisture, the adsorbent was ready for the experiment.

Investigational System: The column chromatography method was using with brick particles as adsorbent as packing materials. The glass wool was used at the bottom of the packing column (as shown in Fig. 1). The two concentrated solutions of Cr (VI) (200 ppm and 300 ppm), Pb (100 ppm and 200 ppm), and Fe (100 ppm and 200 ppm) were used in this experiment. The aqueous solution of the heavy metals was passed through the column carefully which was packed with brick particles (as shown in Fig. 1).

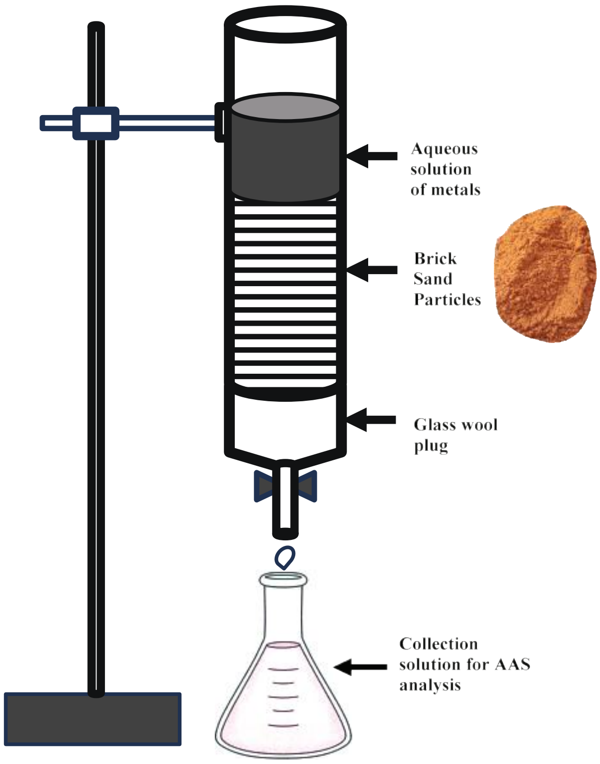

Fig. 1. Schematic diagram of column chromatography in experiment

Adsorbent Characterizations: After passing metal aqueous solution through the column, column filtrate solution was collected from the bottom of the column (as shown in Fig. 1). The collected solution is now ready to analyze AAS to see the percentage of removal of metals. The brick particles which were saturated with metal ions analyzed by SEM to see the surface morphology, EDX for elemental analysis to confirm the removal of metals.

Pore volume: The pore volume of the adsorbent brick sand particles was measured. It is an important parameter of adsorbent. From the effective pore volume, the efficiency of the adsorbent can be assumed (Obey *et al.* 2022).

The effective pore volume was measured by the equation:

Effective pore volume percentage $V_{pore}(\frac{v}{w}\%) = \frac{(\frac{1}{\rho})(W_w - W_d)}{W_w} \times 100$ ………………………………. ....(1)

Here, ρ= the density of water, W_w= wet weight in g, W_d = dry weight in g of brick sand particles.

Specific Surface Area: We determine the specific area of the adsorbent brick sand particles according to the literature (Ahmad *et al.* 2013; Jawad *et al.* 2019). The specific surface area of the sample was obtained using the following formula:

Specific surface area $(\frac{m^2}{g}) = 32v - 25$ ..………………….... (2)

Here V = the volume of 0.1 M NaOH required to raise the pH from 2.0 to 12.0. The pH point of zero charge (pHpzc) of the adsorbent was estimated according to (Karim *et al.* 2024a) with slight modification.

Point zero charge: In a 250 mL conical flask, we take 1.5 g brick sand particles with 30 g NaCl and dissolve them with 100 mL distilled water while stirring for 5 minutes. The solution pH was adjusted to 2 and titrated the solution by 0.1M NaOH until the solution pH reached 12. The required volumes of NaOH change the pH from 2 to 12 (Karim *et al.* 2024).

Results and discussions

Our experimental schematic diagram is shown in Fig. 1 for the removal of heavy metals Fe, Cr, and Pb from aqueous solution using the column chromatography method. From the column chromatography experiments, the fraction collected from column chromatography was passed through brick sand particles packed column. This liquid was used for analysis by AAS and the percentage of metals was removed.

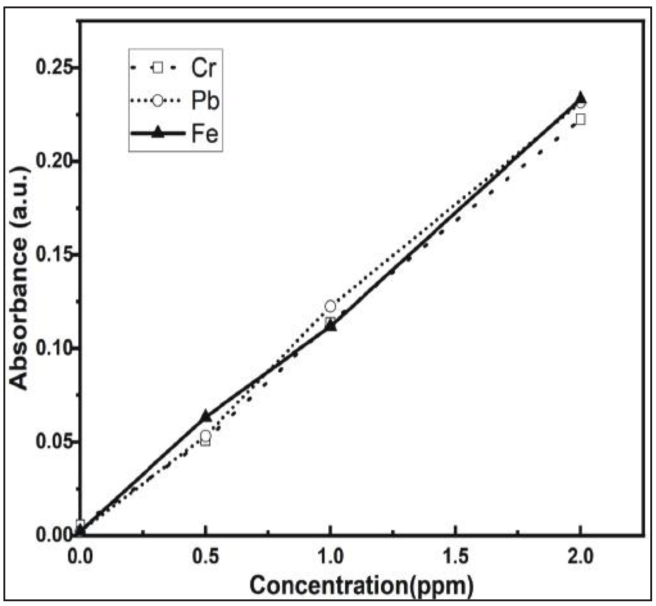

Fig. 2. Atomic absorption analysis of after adsorption of Cr, Pb, and Fe solution onto brick particles surface

AAS study: Fig. 2 represents the AAS analysis of the removal of heavy metals. The results indicate that the highest removal of Cr metal from 200 ppm concentrated solution was about 94% (actually 93.64%). In the case of 300 ppm concentrated Cr solution, the column chromatographic adsorption method was able to remove about 92% (actually 92.38%). Similarly, 24% of Pb and 69% of Fe were removed from aqueous solution. The column chromatography adsorption results and brick particles adsorbent properties are summarized in Table I.

SEM analysis

The brick particle surface morphological study was investigated by SEM as represented in Fig. 4. The SEM images give information on the adsorbent surface status, indicating the changes that occurred due to the column chromatographical adsorption process. Cavities were also observed in the images suggesting a high surface area (Al-Ghouti *et al.* 2020). The SEM images showed the presence of many micropores in brick particles in Fig. 5 before (a) and after (b) adsorption. We also calculated the pore volume using equation (1) which was 44.41%. To understand the adsorption process, it is also important to know the specific surface area. For that, we also determined the specific surface area of brick particles using equation (2), which was 29.4 m^2/g. Raw brick particles image in Fig. 4(a), Fig. 4 (b) surface morphology of brick particles after adsorption of Cr solution, 4 (c) surface morphology of brick particles after adsorption of Pd solution and 4 (d) morphology of brick particles after adsorption of Fe solution. In Fig. 4(a), the micropores are more visible. The surface was so rough and had an irregular distribution of clumps in SEM images after and before the adsorp-

Table I. Summary of the column chromatography process for removal of Fe, Pb and Cr from aqueous solution and adsorbent properties

% Fe removed	% Pb removed	% Cr removed	Pore volume of Brick Sand Particles	Specific surface area of Brick Sand Particles
69.0	24.0	94.0	44.41%	29.4 m^2/g

The % removal of metals was calculated using the following equation

$$C_r = \frac{(C_i - C_f)}{C_i} \times 100 \quad \text{.......... (3)}$$

Here, Ci represents the initial concentration.; C_f represents the final concentration.

Removal of Pb in percentage:

$$\% \text{ Removal} = C_r = \frac{(C_i - C_f)}{C_i} \times 100 \quad \text{.......... (4)}$$

Here, Ci represents the initial Concentration of Pb and C_f represents the final concentration of Pb.

XRD for analysis: The particles size of the brick particles was calculated from XRD ((Bruker D8 Advance) analysis data by Scherrer equation as follows:

$$D = \frac{K\lambda}{\beta\, Cos\theta} \quad \text{.......... (5)}$$

Here, D is referred to Crystalline size in nm, K is Scherrer Constant (0.9), β refers to FWHM -Radians, θ is the position of the peak in Radians respectively.

By using origin software, the position of the peak is FWHM radians. The brick particles' average sizes were 204.7 nanometers. The XRD spectrum is shown in Fig. 3.

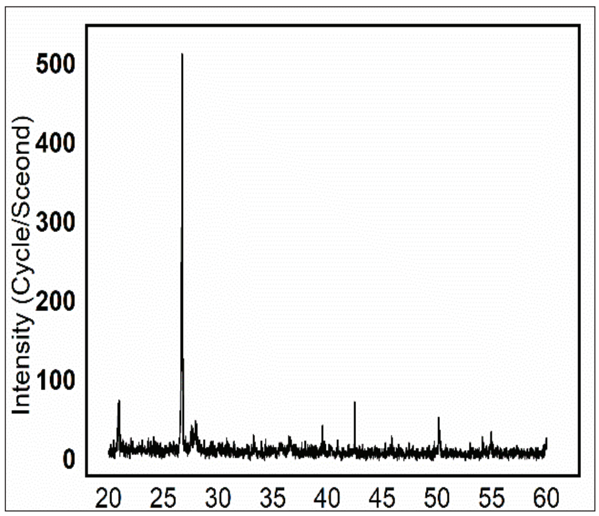

Fig. 3. XRD of brick particles

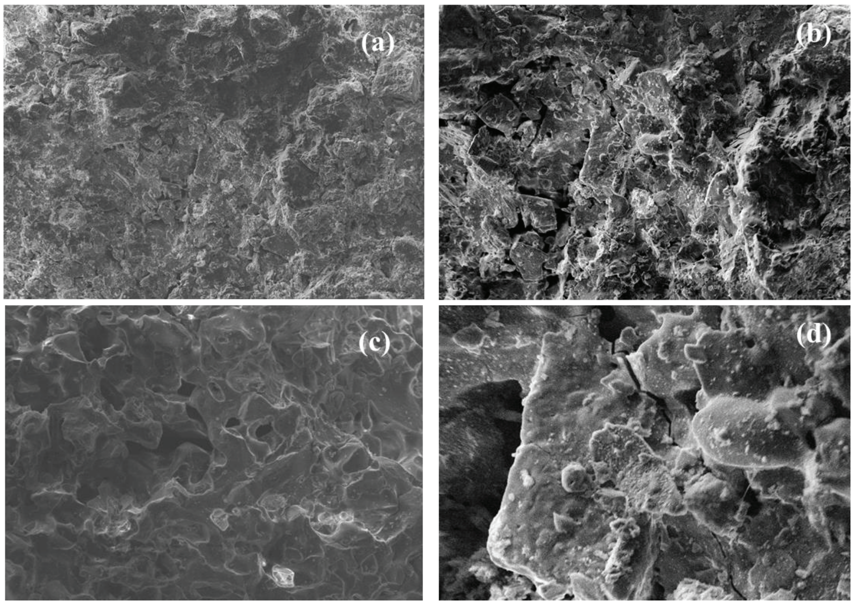

Fig. 4. Surface morphology of a) raw brick particles; b) after adsorption of Cr; c) after adsorption of Pb ppm solution; (d) after adsorption of Fe solution

tion of heavy metals. The brick particles' porous structure ensures a high possibility of adsorption of the metal ions in the pores after adsorption. The adsorbent surface showed blurry pores indicating that adsorption had occurred (Al-Ghouti *et al.* 2020). Fig. 4 shows surface structural proof of modified with toxic metals loaded onto the adsorbents. The raw brick particles SEM image was mostly rough and micro pores visible surface. In the case of adsorption of Cr metal, the surface was highly rough and had many micro pores than that of raw brick particles. It might be due to the reaction between hexavalent ions of Cr and the electrical negative charge of brick particles. The surface morphology of the adsorption of Pb by brick particles is represented in Fig. 4(c). The Pb metal-saturated brick particles surface morphology is also rough and some holes or cavities appeared. Fig. 4(d) is representing the adsorbent for the Fe solution and the surface morphology of the brick particles appeared with some cavities. From Fig. 4(b-d) is markedly viewed that the adsorbent surface is covered by heavy metals. With the SEM images, it is not easy to observe or explain the adsorption process of heavy metals onto the brick particles. This is because brick particles themselves contain some other element in it.

The pH of the solution can control the adsorption process and affect the adsorbent surface charge (Mayakaduwa *et al.* 2016). The point of zero charges can be explained by the pH function in the adsorption process. The point of zero charge value of brick particles was 7.5 as shown in Fig. 5. When the pH of the solution is lower than pHpzc, the adsorbent surface is predominating by a negative charge, and when the pH is above pHpzc, the adsorbent surface is

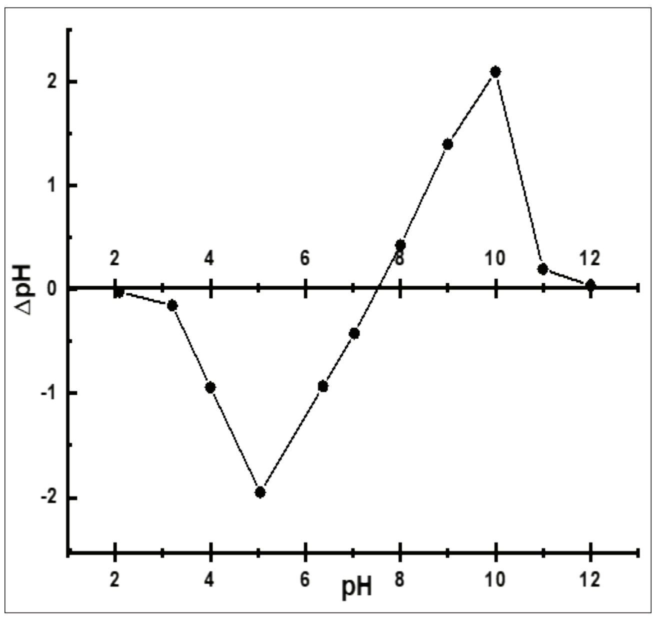

Fig. 5. Brick particles Point zero charge (pHPZC) for the adsorption process

positive in charge. The SEM images are agreed with the concept of pH_{Pzc}.

EDX analysis

To confirm the adsorption of heavy metals onto brick particles, we analyzed the EDX experiment to get a clear idea about adsorption elements. The saturated brick particles EDX image were taken only for 200 ppm solution adsorbed by the adsorbent for every heavy metal as shown in Fig. 6.

Therefore, from the EDX analysis, we may conclude that toxic metals Cr, Pb, and Fe were accumulated onto the surface of bricks particles.

Conclusions

In this study, the heavy metals (Cr, Pb & Fe) were successfully removed by using a column chromatography process with low-cost, easily available adsorbent brick particles. We removed 94% Cr, 24% Pb, and 69% Fe from the aqueous

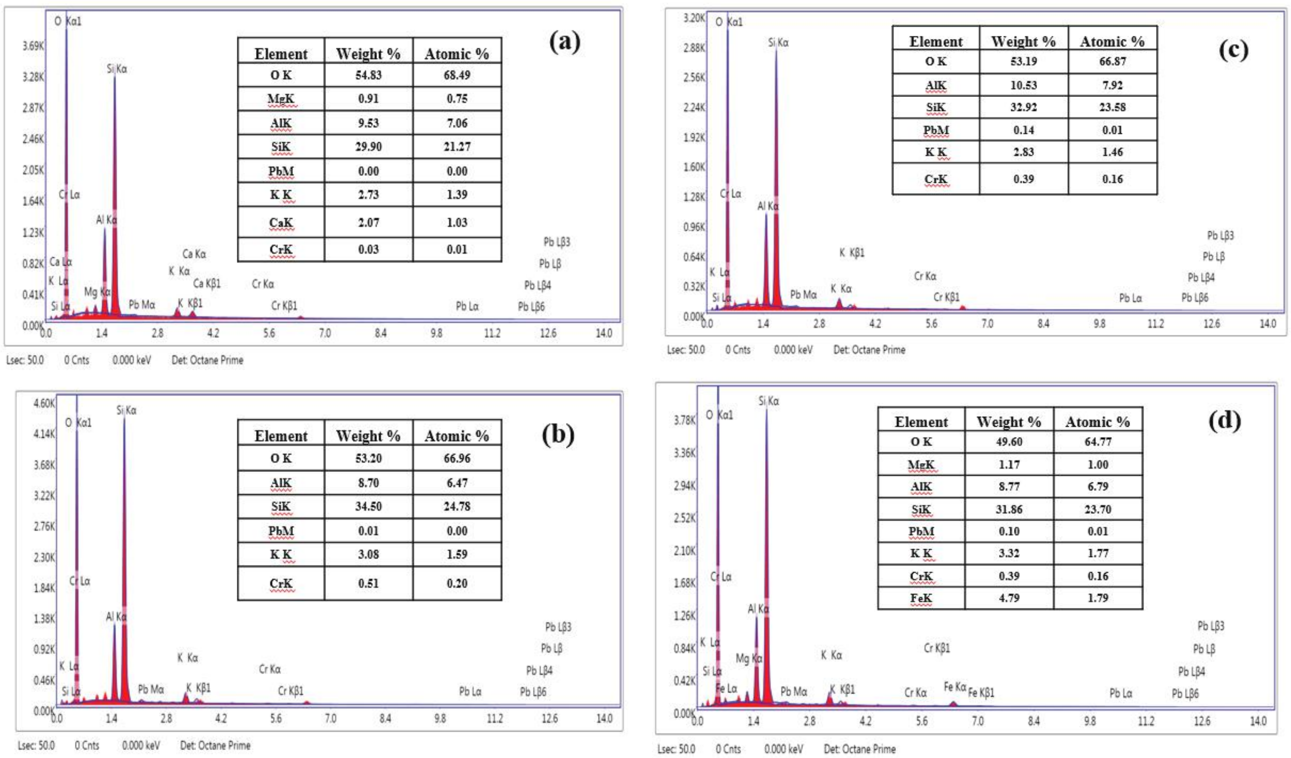

Fig. 6. EDX spectroscopy analysis of (a) raw brick particles; (b) brick particles with adsorption of Cr; (c) after adsorption of Pb; (d) after adsorption of Fe solution

Fig. 6 and the inset table represent the wt% and atomic % of the elements that are present in raw brick particles (Fig. 6(a)) as well as the after-absorption process in 200 ppm of Cr (Fig. 6(b)), Pb (Fig. 6(c)) and Fe (Fig. 6(d)). The atomic number of oxygens is increased for chromium in Fig. 6(b) and Pb in 6(c) was increased compared to raw brick particles in Fig. 6(a). It is because of the thin layer formation of metal Cr and Pb on the surface of brick particles. The atomic % of oxygen elements in the inset table of Fig.s 6(b) and 6(c) confirmed the adsorption of Cr and Pb metals on brick particles. Fig. 6(d) shows a somewhat strong signal of Fe Kα which indicated the adsorption of Fe onto the brick particles surface.

solution through this process. Therefore, we may conclude from the percentage of removal of the metals that the brick particles might be a better adsorbent to remove Cr metal from the aqueous solution by the low-cost and effective chromatography method. The average particle size of the brick particles used in the adsorption process was analyzed by XRD which was 204.7 nm. The surface of the adsorbent (analyzed by SEM) indicates the adsorption of the metals onto the brick particle surface is successfully done, and forms a thin layer onto the adsorbent surface. The EDX analysis confirms the adsorption of toxic metals onto the adsorbent surface by elemental analysis.

Moreover, the saturated adsorbent can be used as an element in construction. The application of adsorbent as a construction element can control further environmental pollution. Considering the socio-economical condition in our country, the low-cost and easily available brick particles can be a better choice of adsorbent for the removal of toxic heavy metals from solution or wastewater.

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Conflict of interest: No conflict of interest

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