



Interaction of Aqueous Cu^{2+} Ions with Granules of Crushed Concrete

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Abstract

The sorption of Cu^{2+} ions from synthetic wastewater using crushed concrete demolition waste (CCDW) which collected from a demolition site was investigated in a batch sorption system. Factors influencing on sorption process such as shaking time (0-300min), the initial concentration of contaminant (100-750mg/L), shaking speed (0-250 rpm), and adsorbent dosage (0.05-3 g/ml) have been studied. Batch experiments confirmed that the best values of these parameters were (180 min, 100 mg/l, 250 rpm, 0.7 g CCDW/100 ml) respectively where the achieved removal efficiency is equal to 100%. Sorption data were described using four isotherm models (Langmuir, Freundlich, Redlich-Peterson, and Radke-Prausnitz). Results proved that the pure adsorption and precipitation are the main mechanisms for removal of copper ions from aqueous solution onto CCDW and sorption data can be represented by Langmuir and Radke-Prausnitz model. The copper ion was successfully removed from aqueous solution during batch experiments using CCDW in the particle size range 2–1 mm. Scanning electron microscopy detected that the removal of Cu^{2+} was found to arise from surface precipitation.

Keywords: Sorption, Recycling, Concrete Demolition waste, Heavy metal ion, Isotherm

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1- Introduction

The contamination of water by toxic heavy metals through the discharge of industrial wastewater is a worldwide environmental problem. Many branches of the industry nowadays generate large quantities of waste containing toxic and carcinogenic organic and inorganic compounds [1]. The presence of heavy metals in streams, lakes, and groundwater reservoirs has been responsible for several health problems with plants, animals, and human beings [2].

Since copper is a widely used material, there are many potential sources of copper pollution such as plating, mining and smelting, brass manufacture, electroplating industries, petroleum refining, and it is used excessively in based agrichemicals mining [3].

These industries produce much wastewater and sludge containing Cu^{2+} ions with various concentrations having negative effects on the water environment. Although a limit of 2 mg/L was proposed by the World Health Organization as the provisional guideline value for copper content of drinking water, intake of excessively large doses of copper by man leads to severe mucosal irritation and corrosion, widespread capillary damage, hepatic and renal damage and central nervous system irritation followed by depression. Severe gastrointestinal irritation and possible necrotic changes in the liver and kidney could occur [4].

Removal of toxic heavy metal ions from the environment is an important challenge. Typically, a removal process must be simple, effective and inexpensive.

Thus, several processes have been employed heretofore including adsorption, chemical precipitation, reverse osmosis and ion exchange, coagulation and flocculation, biosorption and membrane process [5].

Lately, scientists have been studying the use of cheap and plentiful sorbents to remove heavy metals from wastewater. Natural and industrial geomaterials and biosorbents are well-known sorbents, and researchers have done many tests to identify the adsorption capacity of those materials [6].

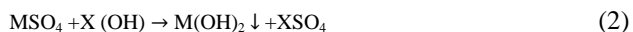
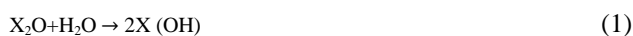
Rapid civilization, industrialization, and population growth in developing and developed countries produce millions of tons of construction and demolition waste (CDW) per year. Consequently, researchers predict that countries will produce massive amounts of CDW materials in the future, creating many environmental problems.

Therefore, investigation of the efficient recycling and usage of those abundantly available resources for different activities is timely.

The utilize of alkaline cement and another portland for the treatment and conditioning of sludge, liquid, and particulate wastes containing heavy metals is well decided.

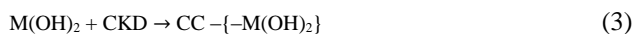
'Sorption' or ion exchange are the mechanisms implied immobilization of heavy metal ions by cement matrix and this process happens by the nanoporous hydration product, calcium silicate hydrate; lattice incorporation into crystalline components of the hydrated cement matrix and deposition of insoluble hydroxides and hydrated metal silicate salts [6], [8]. Cement compounds can be chemically categorized as a highly heterogeneous material, thus, dissolved metals in the aqueous solution can be removed by adsorption and precipitation [9], [10]. In order to describe the precipitation phase and the simultaneous adsorption-precipitation phase it is favored to adopt a gradual operation manner for the description of these mechanisms. The variance between these phases can be clarified by the adsorption phase, as follows [11]:

Precipitation by metal (M) hydrolysis



Where X : Ca, Mg, K; Na; M : Pb, Cu, Ni ...etc

Adsorption of metal onto Cement compounds (CC) particles



Accordingly, it is visualized that the basis of demolition waste is concrete maybe affect the removal of toxic pollutants from contaminant water current. The scientist announced herein has been conducted to examine crushed concrete demolition waste CCDW and to define the kind of the reaction of aqueous containing metal ion with it. It has been investigated in a batch sorption study the removal of heavy metals from contaminant aqueous solutions by the 2-1 mm fraction of CCDW, the granules of CCDW loaded metal ions were tested by scanning electron microscopy and energy dispersive X-ray analysis to define the kind of the CCDW-metal ion interactions.

2- Isotherm Models

In the current study, a range of isotherm models is used to simulate the performance of CCDW in removing heavy metals ions from wastewater. A summary of these models is presented below:

- **Langmuir model:** presume regular energies of sorption upon the surface and no emigration of sorbate in the plane of the surface. It can be written as follows:

$$q_e = \frac{q_m b C_e}{1 + b C_e} \quad (4)$$

Where: q_m is the maximum sorption capacity (mg/g) and b is the constant related to the free energy of sorption (l/mg) [12]. Where: q_e is the amount of adsorbate where q_m is the maximum sorption capacity (mg/g) and b is the constant related to the free energy of sorption (l/mg), C_e is the equilibrium concentration (mg/l).

- **Freundlich model:** is quantified by:

$$q_e = K_F C_e^{1/n} \quad (5)$$

Where K_F is the Freundlich sorption coefficient and n is an empirical coefficient indicative of the intensity of the sorption [12].

- **Redlich-Peterson Model:** In some combination of both Langmuir and Freundlich features, a Redlich-Peterson isotherm comes like a hybrid isotherm model often utilize to represent solute adsorption data on heterogeneous surfaces with an incorporation of three parameters into an empirical equation as follows:

$$q_e = \frac{K_R C_e}{1 + a_R C_e^{\beta R}} \quad (6)$$

Where K_R , a_R , and βR are constants, the βR takes the values ranged from 0 to 1. The Redlich-Peterson model is taken the form of the Langmuir model when βR equal to 1, while it is reduced to the form of Freundlich model for βR equal to 0 [13].

- **Radke-Prausnitz Model:** this model is given as follows:

$$q_e = \frac{q_{mRP} K_{RP} C_e}{q_{mRP} + K_{RP} C_e^{mRP}} \quad (7)$$

Where: q_{mRP} is the maximum adsorption capacity (mg/g), while K_{RP} and mRP are constants [14].

3- Experimental Work

3.1. Materials

Waste concrete was collected from a demolition site and then manually washed with tap water to remove any other undesirable material. It was crushed with Miller, sieved into (2-1) mm, and then it was washed before being used with distilled water to remove fine powder and then dried.

This was used for the batch experiments and characterization. The physical properties of the crushed concrete such as surface area=11.0956m²/g, bulk density=1.0481g/cm³, real density=2.6202g/cm³, and Porosity=0.6 were measured at Oil Research and Development Center-Ministry of Oil. Cu²⁺ was selected as a representative of heavy metal contaminants.

To simulate the water's copper contamination, the stock solution (1000 mg/L) of Cu(NO₃)₂.3H₂O (manufactured by HAMEDIA, India) was prepared by dissolving an appropriate quantity of metal salt in distilled water and then stored in a glass container at room temperature.

3.2. Characterization of CCDW

Samples of CCDW were tested by energy dispersive X-ray analysis (EDX) (TESCAN, Vega III, and the Czech Republic).

Energy Dispersive X-Ray analysis (EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM) used to recognize the elemental composition of materials. The data that resulted from (EDX) examination includes of spectrum showing crests identical to the elements that achieve true composition of the sample being analyzed [15].

3.3. Batch experiments

The batch experiments were conducted through a set of four experiments to identify the circumstances for the highest removal efficiency of the pollutant. These circumstances included shaking time, the initial concentration of contaminant, shaking speed and sorbent dosage.

The **first set** included shaking time variation (0-300 min) at an initial concentration of contaminant, shaking speed and sorbent dosage of 250 mg/l, 200 rpm and 0.2 g CCDW/100 ml of contaminant solution respectively.

The **second set** included a solution variation in the initial concentration of contaminant (100, 150, 250, 500 and 750 mg/l) at an optimum time of shaking obtained from the results of the first set. While maintaining Shaking speed and sorbent dosage at their former values.

The **third set** included variation in shaking speed (0, 50, 100, 150, 200 and 250 rpm) at an optimum initial concentration of contaminant obtained from the outcome of the second set; while the value of the rest of the factors remained the same as in the second set.

Finally, the **last set** included a variation of CCDW dosage (0.05-3g/ml) with a chosen shaking speed obtained from the outcome of the third set; while the value of the rest of the factors remained the same as in the third set. In the experiments mentioned above, 250 ml flasks were utilized, wherein every flask 100 ml of contaminant solution with a certain amount of sorbent dosage were added.

The flasks were shaken by an incubator shaker (ISO 9001, Model: LSI-3016, NO.B110416002, Korea). At the end of any specific experiment, 20 ml of solution sample pulled from the flask and subsequently filtered using filter paper. 10 ml sample was withdrawn from the filtered supernatant to determine the concentration of metal by using atomic absorption spectrophotometer (AAS) (Model: 210 VGP, USA). However, the efficiency of heavy metal removal (Removal %) was calculated as follows:

$$R = [(C_o - C_e)/C_o] \times 100 \quad (8)$$

Where: C_o and C_e are the initial and the equilibrium contaminant concentrations, respectively. R is the removal efficiency (%). From a mass balance it can be specified the amount of sorbed contaminant (q_e) retained in the CCDW phase, by taking into consideration its initial and equilibrium concentrations, therefore pure sorption can be calculated:

$$q_e = \frac{V(C_o - C_{eq})}{m} \quad (9)$$

Where C_o and C_e are the initial and equilibrium concentrations of the contaminant in the solution (mg/l), V is the amount of solution in the flask (l), and m is the weight of the adsorbent substance in the flask (g).

4- Results and Discussion

This section presents sets of batch mode experiments to examine the efficiency of copper removal from wastewater by the selected reactive material; the data obtained from these experiments have been subjected to four sorption isotherms namely Langmuir and Freundlich, Redlich-Peterson and Radke-Prausnitz models. The effect of many operating parameters such as shaking time, initial concentrations of Cu^{2+} , shaking speed and reactive material dosage was studied to choose the best conditions.

4.1. Characterization of CCDW Samples

It is essential that the cement matrix of CCDW chosen for this study was adequately ancient because immature cement pastes contain an appreciable ratio of residual anhydrous phases and contain unrefined pore networks which may affect the nature of their interactions with heavy metal ions [16]. For this reason, new concrete was not used and CCDW was selected for old buildings.

A corresponding energy dispersive X-ray (EDX) spectrum confirmed the existence of Ca, Si, Al, S, Fe, Mg, Na, and K as the major components were found to be approximately 27.13%, 9.26%, 3.48%, 1.24%, 1.15%, 0.81%, 0.78%, 0.41% respectively.

These elements are representative constituents of Portland cement, therefore, constituent concentrations is different according to the ratio of phases existent in a given position, though, it must be indicated that hydrated cement is heterogeneous on a microscopic scale [16].

4.2. Influence of Batch Operating Parameters

a. Effect of Shaking Time

The influence of shaking time on Cu^{2+} removal using 0.2 g CCDW add to 100 ml of contaminated solution with initial pH=4, as shown in Fig. 1 The plot reveals that the rate of the percentage of metal ion removal is higher at the beginning and gradually starts to slow down afterward [17].

That is probably due to diminishing in the sorption sites of the reactive material lead to slower sorption. As the active adsorption sites become exhausted, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to the interior sites of the adsorbent particles.

The kinetic data for CCDW presented that 43% Cu^{2+} was removed mainly at 180min. There was no evident change in the remaining concentration of contaminants after this time of 180 min.

Thus, for all equilibrium adsorption studies, the equilibration period was kept 180 min.

CCDW generally contains alkali and alkaline earth metals such as Ca^{2+} , Mg^{2+} , K^{2+} and Na^{2+} which are primarily presented from fresh water, So that, when CCDW reacts with heavy metals bearing solution, the light metals released causing an increase in the pH of the solution due to the form of light metal alkalis, as illustrated in Fig. 1.

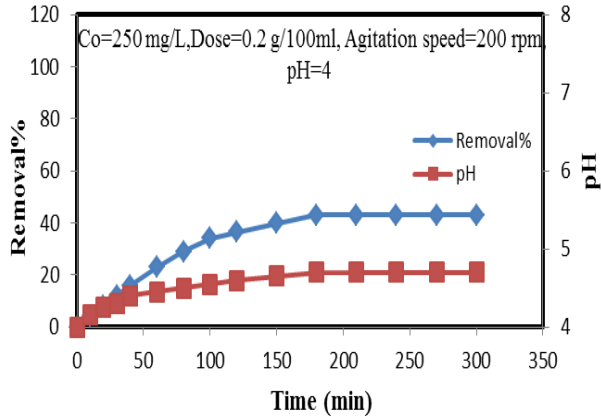


Fig. 1. Removal efficiency as a function of shaking time and final pH of copper ion onto CCDW

b. Effect Of Initial Concentration Of Contaminant

Fig. 2 presents that when the initial concentration increases from 100 to 750 mg/L the removal efficiency decreases from (56 to 13%) for Cu^{2+} by keeping all other parameters constant.

The results present that there was a higher removal percent of the metal at the lower value of initial concentration.

It may be because, at higher concentration of metal ion, the effective sites obtainable on the CCDW to interact with the contaminant approaches saturation, resulting in a decrease in overall percent removal [18].

The simple hydrolysis of generality divalent metal ions particularly Cu^{2+} can be written as follows:



Where M^{2+} is either Cu^{2+} or any other metal ion

The final pH increase when the concentration of the solution decreases this happens when M^{2+} (metal ions) is being taken up by the adsorbent, the reaction above shifts to the left, leading to the depletion of protons and hence a rise in pH [19].

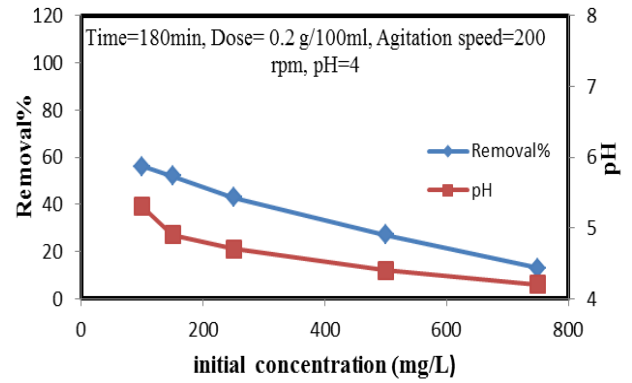


Fig. 2. Effect of initial metal concentration on removal efficiency of copper ion onto CCDW

c. Shaking Speed

The effect of agitation speed on removal efficiency of Cu^{2+} on CCDW was studied varying the speed of agitation from 50 to 250 rpm, Fig. 3 presents that approximately 13% of the Cu^{2+} was removed prior to shaking (shaking speed = 0) with conservation rest parameters equal to best ones acquired at the former steps.

There is a gradual increase in the efficiency of removal of copper ion when shaking speed was increased from 0 to 250 rpm at which about 58% of Cu^{2+} has been removed.

These results can be associated to the fact that the increase of the agitation speed, improves the spread of pollutant across the surface of the reactive material and this means more contact between the pollutant in the solution and the active sites [20].

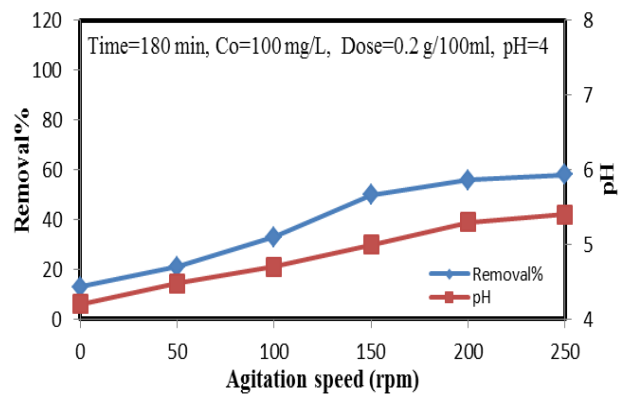


Fig. 3. Effect of agitation speed on percentage removal of copper ion onto CCDW

d. CCDW Dosage

The dependence of Cu^{2+} sorption on CCDW dosage was studied by varying the amount of sorbent within the ranges added to 100 ml of contaminated solution with conservation rest parameters constant as illustrated in Fig. 4.

This figure explains the greater availability of effective sites increases the removal efficiency as increasing the amount of dosage and this has been expected as a result of the high dosage amount of sorbent in the solution.

In addition, this supposed that after a certain dosage amount of sorbent, the greatest sorption sets in and hence the amount of Cu^{2+} attached to the substance and the remaining amount of this contaminant in solution remain consistent even with the further increase with dosage amount of sorbent [20].

The best values of dosage were found to be 0.7 g CCDW/100ml for Cu^{2+} . Increases in pH are because of the presence of carbonate content (HCO_3) in sorbent material, which gives buffer capacity (alkaline condition) to the sample and this was increased due to increase the dosage amount of sorbent.

Adding sorbent particles to an acidic aqueous solution, they will dissolve equalizing acids and will increase the concentration of dissolved calcium [22].

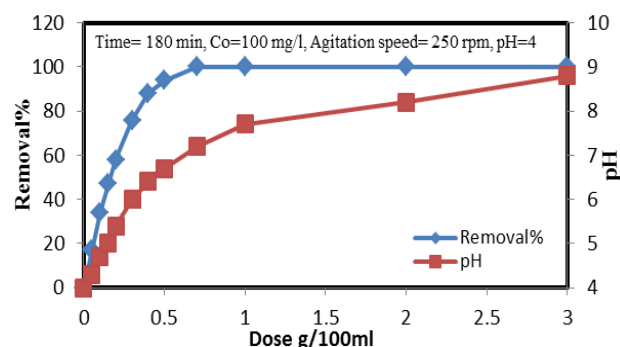


Fig. 4. Effect of CCDW dosage on removal efficiencies copper ion

4.3. Precipitated and Bounded to CCDW of Heavy Metals

Through the course of the study, a small amount of loose floc was observed to have deposited in the nearness of the CCDW particles.

For this reason, the word ‘removal’ has been more suitable than ‘sorption’ or ‘uptake’ in this instance.

The uptake of Cu^{2+} by attaching (bounded) to CCDW was specified by nitric acid digestion according to (Chaney/Mielke Method) [23] of the recovered CCDW and the corresponding proportions of Cu^{2+} present in the loose floc deposited was then calculated by a mass balance for the metal ion as follows:

$$\text{Precipitate} = \text{initial} - (\text{bound to CCDW} + \text{remain in solution}) \quad (11)$$

The final partitioning of the metal ion among that bounded to CCDW, precipitated, and that which remained in solution is depicted in Fig. 5.

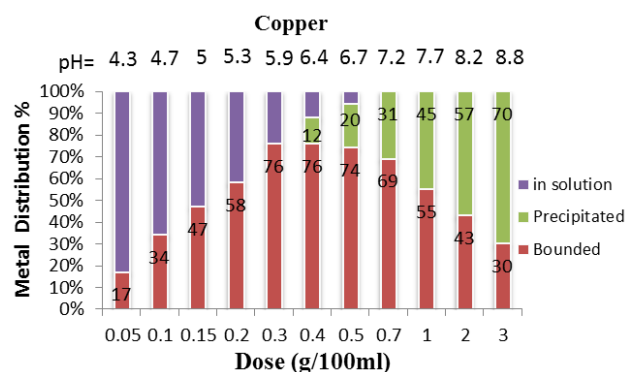


Fig. 5. The final partition of copper ion after 180min

4.4. Isotherm models

Four isotherm models are used to simulate the performance of CCDW in removing heavy metal ion from contaminated wastewater. These models have been applied separately as mathematical (fitting) equations for pure sorption data (bounded to CCDW) and pure precipitation data (precipitated at the bottom of the flask), and then find the most consistent model which describes the two simultaneous processes (present model). Accordingly, the constants with the coefficient of determination for each model were determined by using nonlinear regression method in the Microsoft Excel (2007) [24]. It's summarized in Table 1.

Table 1. Isotherm models constants with a coefficient of determination for removal of Cu^{2+} onto CCDW

Model	Parameter	Cu^{2+}	
		Bounded	Precipitated
Langmuir	q_m (mg/g)	39.286	1.895
	b (l/mg)	0.078	448760.4
	R^2	0.914	0.537
Freundlich	K_F	5.410	5.5820
	$1/n$	0.438	0.55433
	R^2	0.880	0.614
Redlich-Peterson	KR	3.050	1.995
	a_R	0.076	0.019
	BR	1.005	2.598
Radke-Prausnitzze	R^2	0.860	0.712
	q_{mRP}	39.687	107.443
	K_{RP}	3.057	1.987
	mRP	1.002	2.608
	R^2	0.874	0.850

It is clear from Table (1 and 2) that there is a low matching between experimental (bounded & precipitated) and theoretical (isotherm models) data compared with present model, and this may be due to a high heterogeneity of CCDW material [25].

However, Langmuir isotherm models have the highest value of R^2 in case of pure adsorption and Radke-Prausnitzze has the highest value of R^2 in case of pure precipitation.

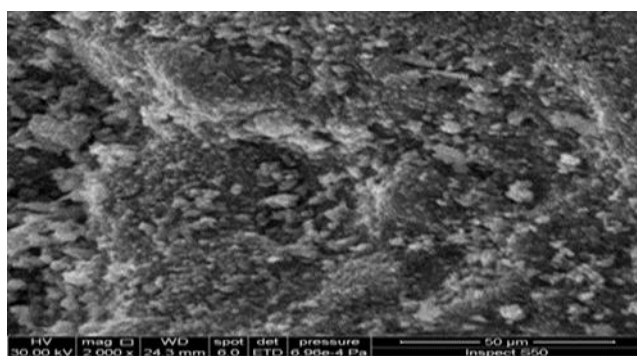
Accordingly, the Langmuir model can be used to represent the bounded and Radke-Prausnitz can be used to represent the precipitated portions for Cu^{2+} as illustrated in Table 2.

Table 2. Present model constants with the coefficient of determination for removal of Cu^{2+} onto CCDW

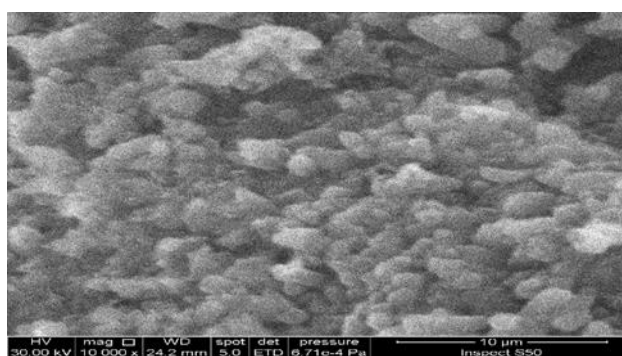
Contaminant	Present model	Parameters	
Cu^{2+}	$q_e)_{Total} = \frac{q_m b C_e}{1 + b C_e})_B + \frac{q_{mRP} K_{RP} C_e}{q_{mRP} + K_{RP} C_e})_P$	q_m (mg/g)	0.594
		b (l/mg)	107.720
		q_{mRP}	19.809
		K_{RP}	8.388
		mRP	0.859
		R^2	0.998

5- Scanning Electron Microscopy (SEM)

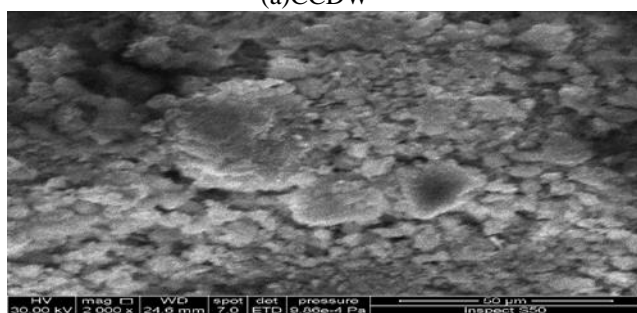
Secondary electron images of the surfaces of CCDW samples before and after loading of heavy metal Cu^{2+} using scanning electron microscope (TESCAN, Vega III, Czech Republic) are shown in Fig. 6. The littered, irregular, fractured surface of CCDW before loading is depicted in Figure 6(a,b). Two distinct copper-bearing phases were observed to have formed on the surface of Cu-CCDW: a copper-rich foliaceous mass and rare, copper-bearing, calcium-rich, distorted polygons (Figure 6(c,d)). The data indicate that the principal mechanism of uptake of copper by CCDW was by precipitation of the foliated crystals onto the surface and that no appreciable diffusion of copper ions into the cement matrix had occurred [16].



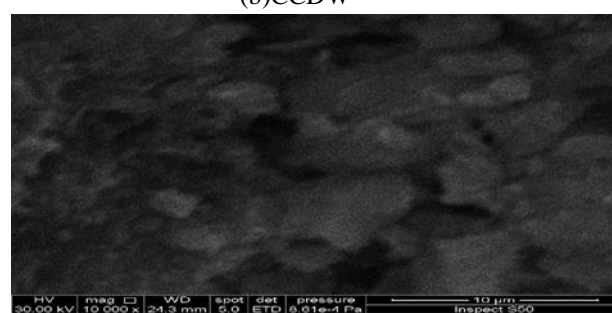
(a)CCDW



(b)CCDW



(c)Cu-CCDW



(d)Cu-CCDW

Fig. 6. Electron micrographs depicting the surfaces of (a, b) CCDW; (c, d) Cu-CCDW

6- Conclusion

- Shaking time, the initial concentration of contaminant, shaking speed, and CCDW dosage were the most important factors affecting the removal process of heavy metals. The best conditions were (180min, 100 mg/l, 250 rpm, 0.7 g CCDW/100 ml), for Cu^{2+} maximum removal
- The SEM images detect that the Cu^{2+} removal by CCDW happens through precipitation of the foliated crystals onto the surface. The results indicate that a rough surface of solid media, high sorptive capacity is beneficial in the removal of heavy metals from water. Thus, CCDW offers the possibility for cheap and effective media for remediation groundwater contamination with heavy metals using a permeable reactive barrier. Sorption and precipitation as metal

oxide were two of the mechanisms that contribute to the removal of metal from the solution.

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تفاعل محلول أيونات النحاس مع حبيبات الخرسانة المسحوقة

الخلاصة

تم دراسة إزالة أيونات النحاس من المياه العادمة الاصطناعية باستخدام نفايات هدم الخرسانة المسحوقة (CCDW) التي تم جمعها من موقع الهدم في نظام الدفعات. تم دراسة العوامل التي تؤثر على كفاءة الإزالة مثل زمن الرج، تركيز الابتدائي، سرعة الرج، الجرعات الممتزة. لوصف بيانات الإزالة تم استخدام أربعة نماذج isotherm (Langmuir و Freundlich و Redlich–Peterson و Radke–Prausnitz). أكدت تجارب الدفعات أن أفضل القيم لهذه العوامل كانت (180 دقيقة و 100 ملغم/لتر و 250 دورة بالدقيقة و 0.7 غم/100 مل) على التوالي حيث بلغت كفاءة الإزالة المتحققة 100%. أثبتت النتائج ان عملية الامتزاز و الترسيب النقي هم الآلية الرئيسية لإزالة أيونات الرصاص من المحاليل المائية ويمكن تمثيل بيانات الإزالة بواسطة موديل Langmuir و Radke–Prausnitz. تم إزالة أيونات النحاس بنجاح من المحاليل المائية خلال دراسة تجارب الدفعات باستخدام (CCDW) في نطاق حجم الجسيمات يتراوح من 2 إلى 1 ملم تم دراسة إزالة أيون النحاس من المياه العادمة الاصطناعية باستخدام نفايات هدم الخرسانة المسحوقة (CCDW) التي تم جمعها من موقع الهدم في نظام الدفعات. كشف المسح المجهر الإلكتروني أن الآلية الرئيسية لإزالة النحاس نشأت من تفاعلات ترسيب السطح.

الكلمات الداله: الإزالة، إعادة التدوير، نفايات هدم الخرسانة، أيون معدن ثقيل، خط التحاور