Mansoura University
Faculty of Engineering
Mansoura Engineering Journal

Investigation of Heavy Metals Removal from Aqueous Solutions by Ceramic Filters

Gehan Mostafa Kotb Tolba*, Ali Gad, Abdallah Mohammed and Ahmed Farghaly

KEYWORDS:
Wastewater, Heavy metals, Ceramic membranes, Filtration.

Abstract— The study utilized the membrane filtration technique to remove two common heavy metals from aqueous solutions; the first was Pb+2, and the second was Cd+2. Manufacturing of ceramic filters was carried out through the dimensions of 20 by 20 cm and thickness of 4 cm from local clay and sawdust at different percentages of sawdust: 1.0%, 2.5%, and 4.0%. The study examined the key factors affecting the removal efficiency of the ceramic filters, such as the initial pH value (5.5 and 7.0) and initial concentration (5.0 and 10.0 mg/L) of heavy metals at a constant pressure head of 30 cm. Moreover, the study applied a microstructural analysis using the scanning electronic microscope (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD) on the utilized ceramic membranes before and after the filtration process. The results showed that the percentages of heavy metals removal are more than 80% at pH 5.5 and 82% at pH 7.0. The efficiency of removing heavy metals from aqueous solutions declines as the initial metal concentrations increases while it increases with the applied water head. Further, although the sawdust percentages had a slight effect on the removal efficiency, ceramic filters with a sawdust percentage of 1.0% were the best.

I. INTRODUCTION

HEAVY metals contain harmful chemical elements such as Pb and Cd. Numerous publications such as [1-3] have extensively investigated heavy metals in wastewater. Among the heavy metals, Cd and Pb are of great interest due to their harmful effects on human health since their concentration in drinking water is relatively high.

Thus, Engineers have widely utilized various techniques for metals removals from wastewater, such as Ion-exchange processes. Further, Low-cost adsorbents are considered an effective technique to remove heavy metals at low concentrations from wastewater. Moreover, Fu and Wang 2011 found that membrane filtration technology is highly efficient in removing heavy metals.

Among the promising filters are ceramic membranes. They are simple, inexpensive, have different shapes and sizes, and utilize materials from the local environment[4-6]. Clay and burnout materials are the most common raw materials in manufacturing ceramic filters). Furthermore, Clay is also utilized for copper removal from wastewater. Compared to alternative adsorbents, it has several advantages, including high surface area and remarkable physical properties, such as shrinkage, bonding strength, and plasticity, chemical properties (monobasicity, large zeta potential, and cation exchange property), and structural/surface properties (chemical attack resistance, water resistance, and strong load-bearing) [7-9].

Received: (10 June, 2022) - Revised: (30 September, 2022) - Accepted: (19 October, 2022)
***Corresponding Author:** Gehan Mostafa Kotb Tolba, Assistant Professor, at Chemical Engineering department, faculty of engineering, Minia university, Minia, Egypt (e-mail: jehan.kotb@mu.edu.eg)*
Ali Gad Professor at Civil Engineering. Department, Faculty of Engineering, Assuit University (e-mail: aligad64@eng.au.edu.eg)
Abdallah Mohammed, Instructor at The Higher Institute of Engineering and Technology, Minia, Egypt (e-mail: civileng.abdallah@yahoo.com).
Ahmed Farghaly, Assistant Professor at Civil Engineering. Department, Faculty of Engineering, Assuit University (e-mail: ahmed_m_farghaly@eng.au.edu.eg).

(Ser. NO. BFEMU-2205-1274)

Another burnout material utilized as an adsorbent for heavy metals removal that showed good properties is sawdust [10], such as hydrochloric acid-modified oak sawdust for the removal of chromium. Results showed that the reported maximum efficiency of the Cr (VI) removal was 84% at a pH of 3.0, and the maximum capacity was 1.70 mg/g at a pH of 3.0 [11].

Therefore, the article aims to investigate heavy metals (Pb^{+2} and Cd^{+2}) removals from artificial aqueous solutions at different pH values and initial concentrations using ceramic filters manufactured with various sawdust ratios[12].

II. Materials and Methods

A. Preparation of Ceramic Membranes Filter

In order to prepare the filter, the study followed the procedure specified in the works of [13, 14]. Moreover, the study utilized sawdust as an agent for pore-forming according to the process of the sacrificial fugitive [15]. First, the clay materials were collected from northern Aswan in Upper Egypt, dried, and ground. Then, the study carried out a sieve analysis to determine the clay particle size. Second, the clay mass was crushed, dried at 100 ºC in an oven for 24 hours, and then grounded into a fine powder. Regarding the sawdust, it was collected from a wood workshop in the local area and then left in the fresh air to dry. After that, it was sieved through an openings sieve of 425-μm Table (1).

Table (1)
Particle size distribution for the utilized sawdust sample.

Sieve analysis for used sawdust sample = 200 gm

Sieve No.	Sieve opening (mm)	Mass retained (gm)	% Retain	% Cumulative retained	% Passing
25	0.707	87.25	43.65	43.65	56.35
40	0.425	56.25	28.25	91.9	8.1
50	0.297	35.2	17.6	89.5	10.5
100	0.149	9.65	4.85	94.35	5.65
200	0.075	8.55	4.3	98.65	1.35
pan	0	1.75	0.875	100	0

Later, the study prepared three different mixes of clay and sawdust with 1.0%, 2.5%, and 4.0 sawdust by weight. The study applied Scanning Electron Microscope (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS) techniques on membrane filters before and after the filtration process to identify the morphology of materials. Additionally, a sample of raw clay powder was scanned by X-ray Diffraction (XRD) analysis.

The clay and sawdust dried powders were manually mixed for about minimum of 20 minutes to mix the powders accurately. After that, water was added to the dry mixture (about 25% by weight), and then they were manually mixed again for 30 minutes. The mixture takes a few minutes to get a homogenous and standardized body. The kneaded mixture was placed in a mold of 20 cm by 20 cm and compressed to get the green body of ceramic sheets, as shown in the photo (1). The higher face was shaped to provide an associate close edge with a 2 cm thickness. The green sheets were left to dry for two weeks in the fresh air away from the sun to avoid the presence of superficial cracks.

Photo (1): Forming the membranes filter shape

After that, the samples were sintered in a muffle furnace with a heating rate of 10º C /minute. The furnace reaches1000º C in 1 hour and 40 minutes. The ceramic filters were sintered at a fixed temperature of concerning 1000º C for five and a half hours. Slowly, the furnace was allowed to cool down for 12 hours. The manufactured ceramic membranes had a red color.

During the preparation of the filtration unit, the manufactured membranes were submerged in distilled water for 24 hours before the filtration tests start. The study utilized a glass basin with an internal dimension of 20 x 20 cm and a height of 50 cm connected to a water pump to pump the chemical solution into another hollow glass basin with an internal dimension of 16 x 16 cm and a height of 50 cm. The two glass basins were connected by water pipes, as shown in Photo (2). The hollow glass, with a cross-sectional area of 16x16 cm and a height of 50 cm, was fastened and sealed at the center of the ceramic sheets (20x20 cm).

Photo (2): Experimental set-up for filtration tests.

B. Preparation of Aqueous Solutions

The study prepared laboratory solutions with high and low initial concentrations to examine ceramic filters' efficiency in heavy metals removal. Lead standard solution was prepared using Lead in lead nitrate $Pb(NO_3)_2$, Pb^{+2} solution with concentrations (5.0 and 10.0 mg/L) by dilution of a stock solution (100 mg/L) of $Pb(NO_3)_2$ with distilled water. Cadmium standard solution was prepared using Cadmium acetate $C_4H_6Cd.2H_2O$, Cd^{+2} solution with concentrations (5.0 and 10.0 mg/L) by dilution of a stock solution (100mg/L) of $C_4H_6Cd.2H_2O$ with distilled water.

Furthermore, the study investigated the effect of pH (5.5 and 7.0) and 5.0 and 10.0 mg/L concentrations of heavy metals at an initial driving pressure head of 30 cm on the filter efficiency of heavy metal removal. Moreover, the pressure head effect on both the removal of heavy metals and filtration rate was investigated at an initial head of 30 cm with heavy metals concentrations.

C. Filtration Experiments

The study used stock solutions of the investigated heavy metals nitrate with concentrations of 1000 mg/L as toxic waste resources. Furthermore, the study utilized a spectrophotometer to determine the initial metal ion concentrations. Moreover, all experiments were carried out in 10000 ml bottles at the ambient laboratory temperature of 32 ± 2ºC. The effect of pH was examined at pH values 5.5 and 7.0, which were adjusted by adding HNO_3 and NaOH solutions. The effect of metal concentrations was investigated through ion concentrations 5.0 and 10.0 mg/L. The Tests were carried out under flux pressure head-on the removal of heavy metals using a constant water head of exact 30.0 cm with heavy metal concentrations of 5.0 and 10.0 mg/L at pH values 5.5 and 7.0 for both lead and cadmium with different sawdust percentages (1.0, 2.5, and 4.0). The polluted water solutions were discharged and influx inside the glass prisms on the top of the ceramic membranes, as shown in Photo (3). Then, plastic pots were held underneath the ceramic membranes to receive the yielded water from filtration. After that, the initial driving water head over each ceramic membrane and the weight of the collected-filtered water through the filtration time were observed. The study calculated the mass filtration rate by dividing the weight of the collected water in the lower plastic container over the corresponding filtration time. Consequently, the volumetric filtration rate could be estimated.

Photo (3): Experimental filtration set-up for metals Pb, and Cd removal by ceramic filters.

D. Microstructural Analysis

The study utilized Scanning Electronic Microscope (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS) images (JSM IT 200) before and after the filtration tests to investigate the surface morphology of the ceramic membranes. A small sample was cut from the membrane center and coated with gold under vacuum pressure to increase materials conductivity and to comply with the constraints imposed on the specimen size for the SEM analysis. Each specimen was allocated in an electron beam to be imaged at the desired magnification scale. Also, the EDS technique detects x-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analyzed volume.

III. Results and Discussion

A. Effect of pH on the Removal Efficiency of Heavy Metals

1. pH Effect on Pb^{+2} Removal by Ceramic Membrane Filters

The solution's pH value controls the charge on the surface. Therefore, it significantly influences metal removals from the solution [16]. Figures 1 and 2 present the ceramic membrane filters' removal efficiency of lead (Pb^{+2}) from aqueous solutions with 5.0 and 10.0 mg/L metal concentrations at different pH values and different sawdust percentages. Figure (1) shows that the 1.0% sawdust-ceramic membrane removal percentage of Pb^{+2} was about 96.04% for the 5.0 mg/L-metal concentration, and it rises as the value of pH increases. Likewise, the efficiency of the 2.5% sawdust-ceramic membrane increases as the value of pH increases since the removal percentage at pH of 5.5 was 94.571%, compared to 99.04% at pH of 7.0. Additionally, the removal percentage of the 4.0% sawdust-ceramic membrane reached 87.34% at pH of 5.5, compared to 94.7% at pH of 7.0. At lower values of pH, the density of the positive H^+ ions charges on the membrane surface sites increases, lowering metal removal and generating an electrostatic repulsion between the positively charged edge groups and metal cations **[17].**

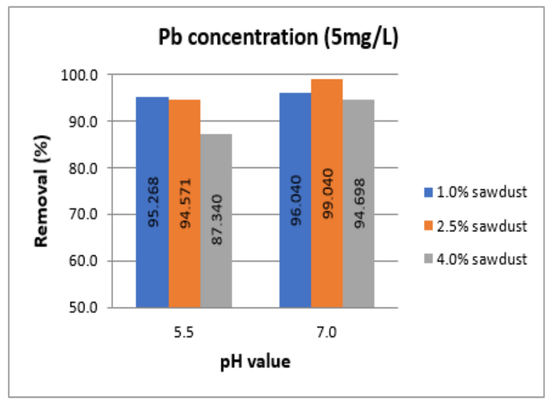

Fig. 1. Efficiency of Pb^{+2} removal for the 5.0 mg/L-concentration at different ratios of sawdust.

Figure (2) illustrates the removal percentage of Pb^{+2} for the aqueous solutions with a 10.0 mg/L-metal concentration. As shown in the figure, the removal percentage of the 1.0% sawdust-ceramic membrane was 86.18%, and it increases as pH increases. Further, the efficiency of the 2.5% sawdust-ceramic membrane rises as the pH increases since the removal percentage at pH of 5.5 was 82.96%, compared to 88.97% at pH of 7.0. However, the removal percentage of the 4.0% sawdust-ceramic membrane reached 84.6% at a pH of 5.5, compared to 83.01% at a pH of 7.0.

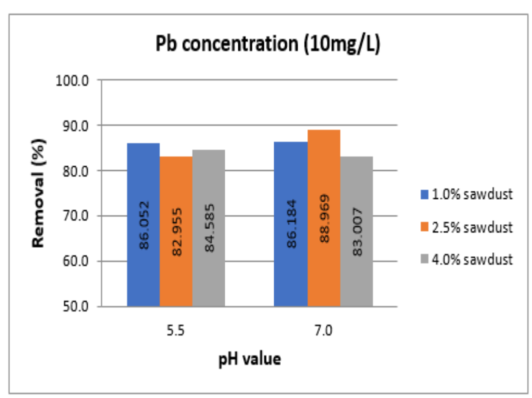

Fig. 2. Efficiency of Pb^{+2} removal for the 10 mg/L-concentration of at different ratios of sawdust.

2. pH Effect on Cd^{+2} Removal by Ceramic Membrane Filters

Figures 3 and 4 present the efficiency of cadmium removal from the aqueous solutions with of 5.0 and 10.0 mg/L metal concentrations at different pH values by ceramic filters with different sawdust percentages. Figure (3) shows that the 1.0% sawdust-ceramic membrane removal percentage of Cd^{+2} was about 87.8%% for the 5.0 mg/L-metal concentration, and it rises as the value of pH increases. Likewise, the efficiency of the 2.5% sawdust-ceramic membrane increases as the pH value increases since the removal percentage at pH of 5.5 was 82.5%, compared to 85.2% at pH of 7.0. However, the removal percentage of the 4.0% sawdust-ceramic membrane reached 84.7% at a pH of 5.5, compared to 81.70% at a pH of 7.0.

Figure (4) illustrates the removal percentage of Cd^{+2} for the aqueous solutions with a 10.0 mg/L-metal concentration. As shown in the figure, the removal percentage of the 1.0% sawdust-ceramic membrane was 92.91%, and it increases as pH increases. Further, the efficiency of the 2.5% sawdust-ceramic membrane increases as the pH increases since the removal percentage at pH of 5.5 was 90.73%, compared to 94.18% at pH of 7.0. Likewise, the removal percentage of the 4.0% sawdust-ceramic membrane reached 89.48% at a pH of 5.5, compared to 93.41% at a pH of 7.0. Higher rates of metal removal in the experiment at a pH of 7.0 are due to the coupling effect of two treatment mechanisms, the precipitation of insoluble metal hydroxides and membrane filtration [18].

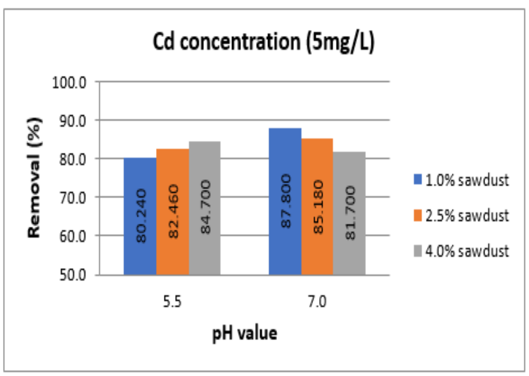

Fig. 3. Efficiency of Cd^{+2} removal for the 5.0 mg/L-concentration at different ratios of sawdust.

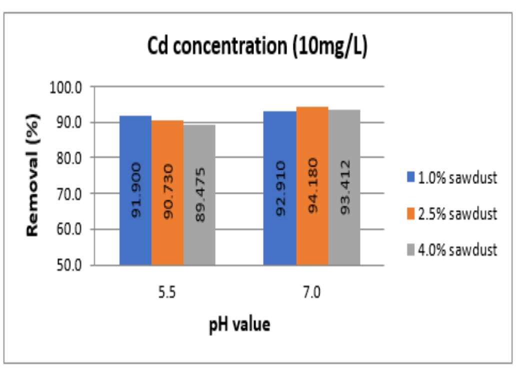

Fig. 4. Efficiency of Cd^{+2} removal for the 10.0 mg/L-concentration at different ratios of sawdust.

B. Effect of Aqueous Solution Concentration on Efficiency of Heavy Metals Removal

1. Pb^{+2} Concentration Effect on Removal by Ceramic Membrane Filters

Figures 5, 6, and 7 present the removal efficiency of lead (Pb^{+2}) from the aqueous solutions with 5.0 and 10.0 mg/L-metal concentrations at different pH values by ceramic filters with various sawdust percentages. They show that as the initial metal ion concentrations increase, the removal percentage of Pb^{+2} decreases. This deficiency at higher concentrations results from the membrane pores' capacity that becomes relatively saturated with ions, reducing the available active sites.

Figure (5) presents the filtration results of the removal of Pb^{+2} by the 1.0% sawdust-ceramic membrane filters at 5.0 and 10.0 mg/L-metal concentrations. At a pH of 5.5, the Pb^{+2} removal decreases from 95.27% at the 5.0 mg/L-initial concentration to 86.05% at the 10 mg/L-initial concentration. At a pH value of 7.0, the Pb^{+2} removal decreases from 96.04% at the 5.0 mg/L-initial concentration to 86.18% at the 10 mg/L-initial concentration.

Figure (6) illustrates the filtration results of the removal of Pb+2 by the 2.5% sawdust-ceramic membrane filters at 5.0 and 10.0 mg/L initial concentrations. At a pH of 5.5, the Pb^{+2} removal decreases from 94.57% at the 5.0 mg/L-initial concentration to 82.96% at the 10 mg/L-initial concentration. At a pH value of 7.0, the Pb^{+2} removal decreases from 99.04% at the 5.0 mg/L-initial concentration to 88.97% at the 10.0 mg/L-initial concentration.

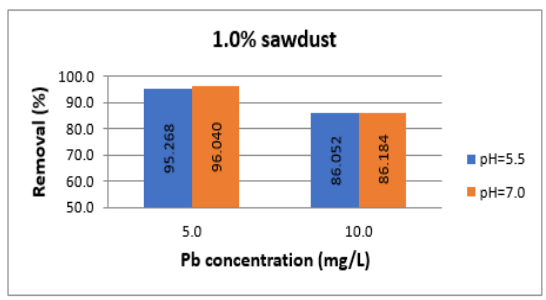

Fig. 5 Efficiency of Pb^{+2} removal for the 1.0 % sawdust at different pH values.

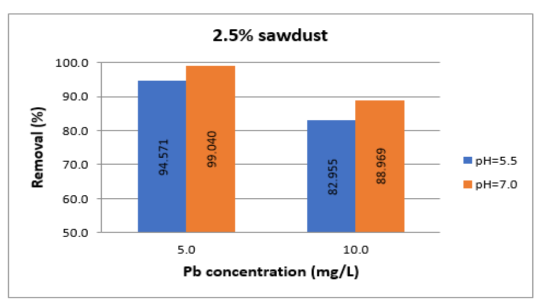

Fig. 6. Efficiency of Pb^{+2} removal for 2.5 % sawdust at different pH values.

Figure (7) demonstrates the filtration results of the removal of Pb^{+2} by the 4.0% sawdust-ceramic membrane filters at 5.0 and 10.0 mg/L initial concentrations. At a pH of 5.5, the Pb^{+2}

removal declines from 87.34% at the 5.0 mg/L-initial concentration to 84.59% at the 10 mg/L-initial concentration. At a pH value of 7.0, the Pb^{+2} removal declines from 94.7% at the 5.0 mg/L-initial concentration to 83.01% at 10.0 mg/L-initial concentration. This is due to a reduction in the active pore size and the available adsorption sites at the higher concentration.

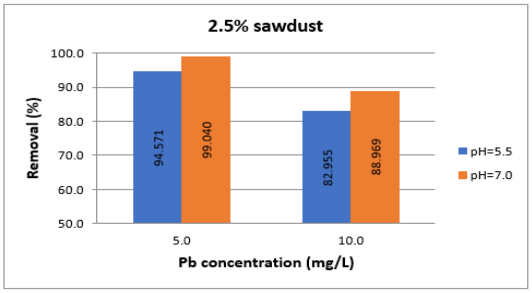

Fig. 7. Efficiency of Pb^{+2} removal for 4.0 % sawdust at different pH values.

2. Cd^{+2} Concentration Effect on Removal by Ceramic Membrane Filters

Figures 8, 9, and 10 present the efficiency of Cd^{+2} removal from the solutions with metal concentrations of 5.0 and 10.0 mg/L at different pH values by ceramic filters with various sawdust percentages.

Figure (8) presents the filtration results of the removal of Cd^{+2} by the 1.0% sawdust-ceramic membrane filters at 5.0 and 10.0 mg/L initial concentrations. At a pH of 5.5, the percentage of Cd^{+2} removal increases from 80.24% at the 5.0 mg/L-concentration to 91.9% at the 10 mg/L-concentration. At the value of pH of 7.0, the percentage of Cd^{+2} removal increases from 87.8% at the 5.0 mg/L-concentration to 92.91% at the 10 mg/L-concentration.

Figure (9) illustrates the filtration results of the removal of Cd^{+2} by the 2.5% sawdust-ceramic membrane filters at 5.0 and 10.0 mg/L initial concentrations. At a pH of 5.5, the removal percentage of Cd^{+2} increases from 82.50% at the 5.0 mg/L-concentration to 90.73% at the 10 mg/L-concentration. At the value of pH of 7.0, the percentage of Cd^{+2} removal increases from 85.18% at the 5.0 mg/L-concentration to 94.18% at the 10 mg/L-concentration.

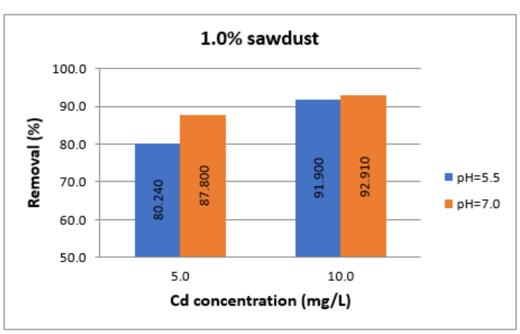

Fig. 8. Efficiency of Cd^{+2} removal with 1.0 % sawdust at different pH values.

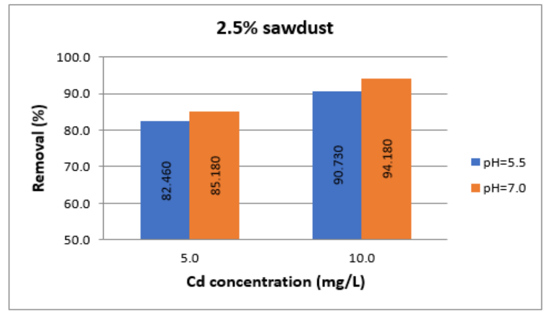

Fig. 9. Efficiency of Cd^{+2} removal with 2.5 % sawdust at different pH values.

Figure (10) demonstrates the filtration results of the removal of Cd^{+2} by the 4.0% sawdust-ceramic membrane filters at 5.0 and 10.0 mg/L initial concentrations. At the value of pH of 5.5, the percentage of Cd^{+2} removal increases from 84.7% at the 5.0 mg/L-concentration to 89.5% at the 10 mg/L-concentration. At the value of pH of 7.0, the percentage of Cd^{+2} removal increases from 81.7 at the 5.0 mg/L-concentration to 93.41% at 10 mg/L-concentration.

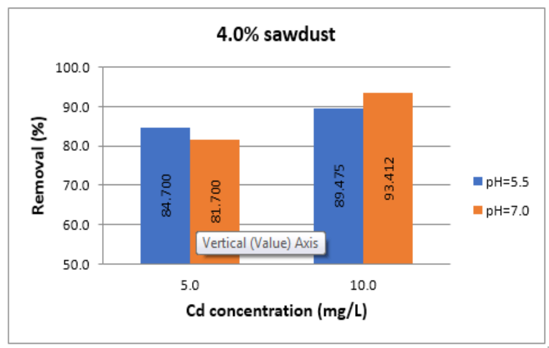

Fig. 10. Efficiency of Cd^{+2} removal with 4.0 % sawdust at different pH values.

IV. Results and Discussion of Microstructural Tests

A. EDS of Ceramic Membrane Filters Before and After Filtration

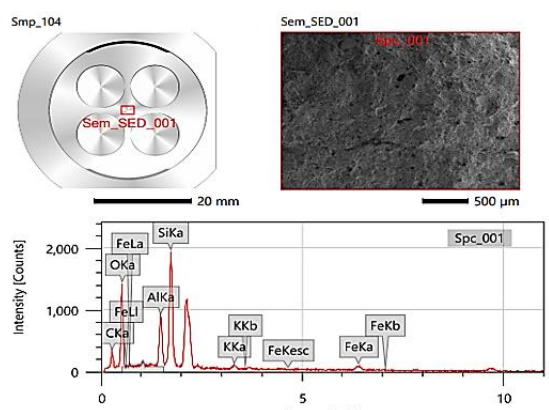

Fig. 11. EDS before filtration.

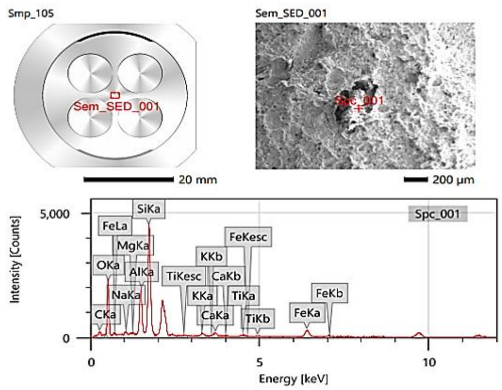

Fig. 12. EDS after filtration.

Figure (11) illustrates the EDS analysis utilized at an acceleration voltage of 15kV and enlargement of 50X before the filtration process, confirming a 19.52% carbon, 36.31% oxygen, 0.9% sodium, 10.28% aluminum, 24.15% silicone, 1.68% 7.17% potassium, and 1.19% iron in the 1.0% sawdust-ceramic membrane filter.

While Figure (12) shows the EDS analysis utilized at an acceleration voltage of 20kV and enlargement of 70X after the filtration process, confirming the presence of 12.13% carbon, 38.45% oxygen, 1.15% sodium, 0.18% magnesium, 10.36% aluminum, 27.42% silicone, 1.18% potassium, 1.39% calcium, 0.76% titanium and 6.99% iron in in the 1.0% sawdust-ceramic membrane filter.

B. SEM Analysis of Ceramic Membrane Filters Before and After Filtration

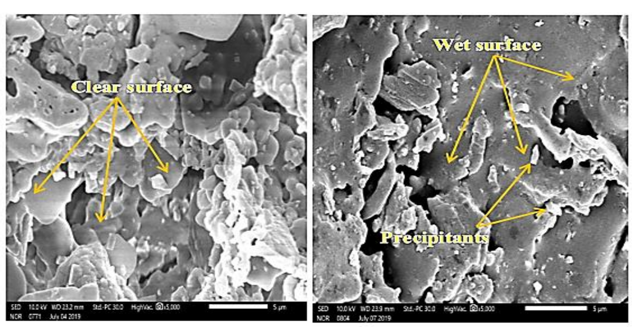

Photo (4): SEM images enlarged at 5000X from ceramic membranes with 2.5% sawdust (a) before and (b) after filtration test.

The above Photos show SEM images of the 2.5% sawdust-ceramic membrane enlarged at 5000X before and after the filtration process. Photo (4. a) illustrates that, before filtration, the membrane's surface contained only clay crystals, membrane pores were clear, and cracks were not present. Moreover, there is a good connection between interior and top layers. Photo (4. b) displays the metal ions adsorbed on the membrane's pores surface and the surface wetness. The pores surface of the ceramic membrane with negative charges attracts the metal ions carrying positive ones, resulting in a process of surface adsorption. The interior surface of the pores seems darker after the process of filtration compared to before.

C. XRD Pattern of the Raw Clay Powder

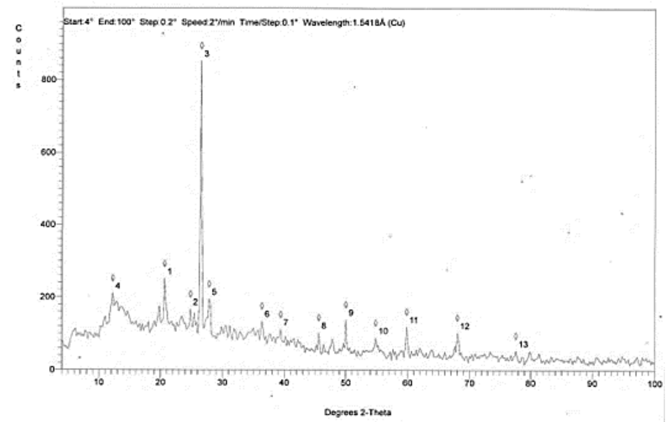

Fig. 13. X-ray diffraction (XRD) pattern of the raw clay powder (natural clay).

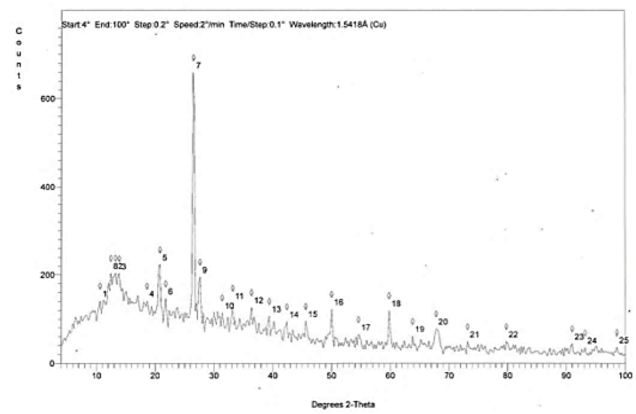

Fig. 14. X-ray diffraction (XRD) pattern of the sintered clay powder.

The study utilized X-ray diffraction (XRD) to characterize the mineralogy of the supported metal oxide pattern-ray of the natural clay and sintered membrane (clay and sawdust). Figures (13) and (14) present the diffractograms of the two cases, which are similar, indicating no significant changes in the clay structure due to the sintering process. Slight changes appeared among the diffractograms of the sample of natural clay and the sample calcined at 1000 ºC. Figure (13) shows the powder x-ray diffraction pattern of the natural clay indicating diffraction crests attributed to quartz. These crests disappear from the sintered membrane indicating its transformation to silicon oxide, as shown in Figure (14). The x-ray diffraction results revealed that the effect on the clay sample was a component of two compounds, Silicon Oxide (quartz) and Sodium Aluminum Silicate Hydroxide Hydrate (kaolinite). Each compound has a unique diffraction pattern.

V. CONCLUSION

The study utilized ceramic membrane filters manufactured from natural clay with different sawdust percentages to remove heavy metals from aqueous solutions. Further, the experiments were carried out at different initial heavy metals concentrations, pH values, and a constant driving head of 30 cm. The removal efficiency of Pb^{+2} declines as the initial metal concentration increases. Contradictory, the removal efficiency of Cd^{+2} increases as the initial metal concentration increases. The XRD analysis indicated a percentage of quartz (SiO_2) in the raw clay and a slight effect of the sintering process on its composition components. Increasing the contribution of sawdust to the substrates of the ceramic membrane makes the ceramics brittle and has significant interior cracks that do not impact the efficiency of metal removal.

AUTHORS CONTRIBUTION

Gehan M. K. Tolba

She substantially contributed to methodology, experimental work, writing the paper and provided the final approval for the version to be published.

Ali A. M. Gad

He is the supervisor of this work, and contributed to methodology and experimental work.

Abdallah S. E. Mohammed

He substantially contributed to the experimental work and writing the paper.

Ahmed M. Farghaly

He contributed to the methodology and critical revision of the article.

FUNDING STATEMENT:

The author did not receive any financial support of the research authorship and publication of this article.

DECLARATION OF CONFLICTING INTERESTS STATEMENT:

The author declared that there are no potential conflicts of interest with respect to the research authorship or publication of this article.

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Arabic Title

دراسة فى ازالة المعادن الثقيلة من المحاليل المائية باستخدام المرشحات السيراميكية.

Arabic Abstract

يهدف هذا البحث إلى دراسة قدرة مرشح السيراميك على إزالة المعادن الثقيلة الشائعة مثل الرصاص (Pb) والكادميوم (Cd) من المحاليل المائية. ولهذا، تم تصنيع مرشح سيراميكي بأبعاد ٢٠ × ٢٠ سم وسمك ٢ سم من الطين المحلي (الطين الأسواني الأحمر) وبنسب مختلفة من نشارة الخشب وهي (١,٠ ٪ & ٢,٥ ٪ & ٤,٠ ٪). تم تحضير المحاليل المائية الملوثة بالمعادن الثقيلة وترشيحها من خلال المرشحات السيراميكية. و قد تمت دراسة العوامل المؤثرة على كفاءة إزالة هذه المعادن بواسطة المرشحات السيراميكية وذلك من خلال المتغيرات التالية: قيم الاس الهيدروجيني للمحاليل المائية (pH) وكانت (٥,٥ & ٧,٠)، وكذلك التركيزات الأولية للمحاليل المائية وكانت (٠.٥ & ١٠,٠ مليجرام لكل لتر) ، وأيضا ثبات ارتفاع ضغط المياه المؤثر على المرشح السيراميكي بقيمة ٣٠ سم. كما أجريت تحاليل مجهرية قبل وبعد عملية الترشيح باستخدام المجهر الإلكتروني الماسح (SEM) وكذلك التحليل الطيفي للأشعة السينية المشتتة (EDSأو EDX أو XEDS) وتم عمل تحاليل حيود الأشعة السينية (XRD) لعينة الطين المستخدمة في المرشح السيراميكي في حالتها الطبيعية قبل الحرق وللمرشحات السيراميكية بعد الحرق. أوضحت النتائج أن النسب المئوية لإزالة المعادن الثقيلة تزيد عن ٨٠,٠٪ في درجة الحموضة ٥,٥ بينما كانت ٨٢,٠٪ في درجة الحموضة ٧,٠. وقد تزايدت كفاءة الإزالة مع تناقص التركيزات الأولية للمحاليل المائية. كما اتضح أن هناك تأثير صغير لتغيير نسب نشارة الخشب في كفاءة الإزالة ؛ وبالرغم من ذلك فإن المرشح السيراميكي مع نسبة نشارة الخشب ١,٠٪ هو الأفضل ويوصى به بسبب سهولة عملية التصنيع وقلة انتشارالشقوق الدقيقة التي تتولد من الانكماش أثناء تجفيف العينة.