

Preparation and Characterization of Copper Nanocomposite Catalysts and its Activity for Adsorption of Lead from Aqueous Solution

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Abstract:

The efficiency of adsorption using copper nanocomposite catalysts for the removal of lead ions from aqueous solution was studied. Nano copper and its nanocomposition were prepared in laboratory in very accurate conditions. After preparing samples, the samples were analyzed using XRD and SEM and that shows very good morphology of catalytic performance and has nano sized of crystalline shape. In order to study the efficiency of these catalysts, all samples were tested using adsorption process. The preparing samples show very good efficiency for lead removing from aqueous solution, and reach maximum removal efficiency 96% for 0.05g of adsorbents at pH 10, otherwise increasing or decreasing the pH would give undesirable results for all samples. Langmuir isotherm fitted better than Freundlich isotherm for adsorption of lead.

Keywords: Nanocomposits, Adsorption, Lead

1. Introduction

Over the years, the percolation of heavy metals into the water bodies and scheme stay united of the foremost elusive and pervasive environmental threat to the world occupants. Heavy metal ions such as cadmium, lead, chromium, mercury, arsenic and copper are classified as priority pollutants supported their toxicity and quality in natural water streams. Even so, the toxic metal ions are stable and protracted to setting changes since they can't either be degraded or destroyed [1]. Among the various dangerous metals, lead is one in every of the common and most virulent pollutants into the natural waters from numerous industrial activities like plating, oil refinement and battery producing [2].

Various treatment approaches are applied by scientific community so as to clean the water free from any toxic metal ions. Most of these techniques are often expensive. However, the adsorption process is an attractive alternative due to its ease of operation, economic viability and effectiveness.[3-6].

A number of materials have been widely investigated as adsorbent in water pollution control. Some of the important ones include

zeolites [7, 8], activated carbon [9-12], rubber ash [13], clay [14-17], expanded perlite [18], vermiculite [19], algae [20-22], bacteria [23], coir pith [24], sugarcane bagasse [25], olive stone [26] and rice husks [27-35].

Many materials present among of them the supported and unsupported Copper and Copper oxide nano-particles are of nice interest attributable to their benefits like nontoxicity, abundance, high optical absorbance and low band gap energies [36,37].

In our study nanocomposite copper catalysts was prepared in three different methods and characterized using X-Ray and SEM to compare the crystal shape with literature and then using these samples to adsorb lead from aqueous solution in order to check it is activity and stability. The results of adsorption were applied to adsorption isotherm.

2. Experimental Work

2.1. Preparation of Stock Solutions

All the chemicals procured and applied during this study were analytical grade. Stock solution of lead was prepared by dissolving $Pb(NO_3)_2$ in deionized water. Totally different need concentrations of pb(II) were ready by diluting 1000ppm of the stock solution. Calibration curve were plotted by preparing different samples of standard stock solution of pb(II) (1000 mg/L) for atomic absorption photometer.

2.2. Preparation of Nanocomposite Adsorbent.

The starting material is copper sulfate $Cu(SO_4)_2 \cdot 4H_2O$ both supplied by BDH and the diammonium phosphate $(NH_4)_2HPO_4$ also from BDH. 100ml of distilled water was filled in 250ml beaker for each component, the two components were added continuously with stirring to dissolve will until get a saturated solution, and then the solutions were separated from the remaining undissolved chemicals in the bottom of beaker.

The saturated solution of $Cu(SO_4)_2 \cdot 4H_2O$ was putted in beaker and the saturated $(NH_4)_2HPO_4$ was filled into burette. The starting of experiments where slowly dropped of burette solution into the beaker solution at room temperature around 25°C, the pH of

Cu(SO₄)₂·4H₂O solution is 11 but when (NH₄)₂HPO₄ solution was added the pH of mixture will drop fast to 2, therefor NaOH solution was added to the mixture to increase the pH to 7, then the mixture was magnetically stirred until a homogeneous solution was obtained, viscous solution were separated via solution-precipitation method [38]. Different Morphologies of Nano-sized particles of copper were prepared as follows:-

- **Sample A:-** the precipitate was dried at 200°C for 2hr.
- **Sample B:-** the above homogeneous solution was heated using microwave radiation of power 800 Watt for 5 min, then the precipitation was filtered and finally dried at 110°C for 3 hr.
- **Sample C:-** The above homogeneous solution was mixed with 50% of activated carbon (1.5-9mm, rpiresearch products international corp.)

2.3. Adsorption of Lead Studies

Adsorption experiments have been carried out at room temperature using batch technique. From the above prepared stock solution different concentrations of lead were prepared by dilution of specified volume of the stock solution 100 ml of pb(II) ions solution of desired concentrations (10-50 mg/L) with varying doses of adsorbents (0.05-0.25g) was mixed by shaker for time intervals from (15-135min) then the samples separated by centrifuge and the residual concentration of lead ions was determined by atomics absorption spectrophotometer. The effect of pH on the metal sorption was adjusted in the range of (2-12). The initial pH of the metal solution was controlled to the desired pH value using 0.2M HCl or 0.2M NaOH. To get the best experimental results the experiments were carried in duplicated and the average value have been used in data analysis. The percentage removals of lead were calculated using the following equation:-

$$\%Removel = \frac{C_{initial} - C_{final}}{C_{initial}} \times 100 \quad .(1)$$

where, C_{initial} and C_{final} are the initial and final concentrations (mg/L) of pb(II).

3. Results and Discussion

3.1. Morphology Analysis

3.1.1 X-Ray Diffraction Analysis (XRD)

An XRD measurement of various prepared adsorbents was employing a Siemens D5000 advance diffractometer. The patterns were run with Cu Ka radiation at 44 kV and 35 mA over 2θ range of 5°-55° with step 1° at residence time 5s at each point.

The results of three samples were shown in figures 1, 2 and 3, the structure of sample A and B

is best from structure of sample C and have crystalline molecules this due to drying process and have same molecules, but for sample C the different shape come from mixing with another adsorbent cased no interaction between Cu and activated carbon.

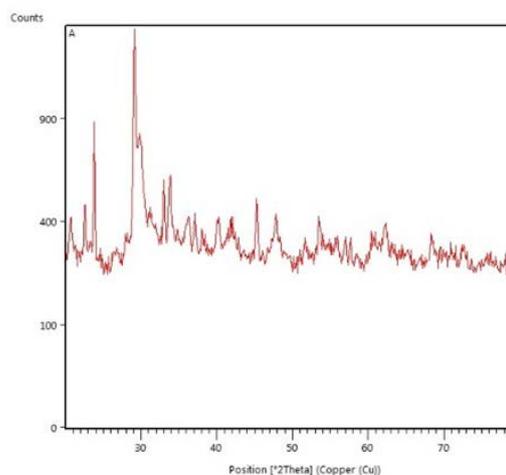


Figure 1: XRD of Sample A

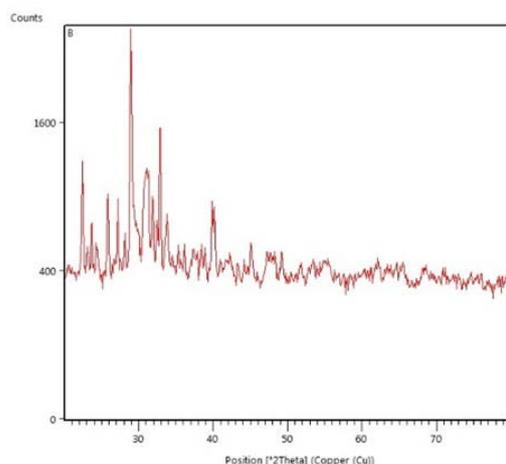


Figure 2: XRD of Sample B

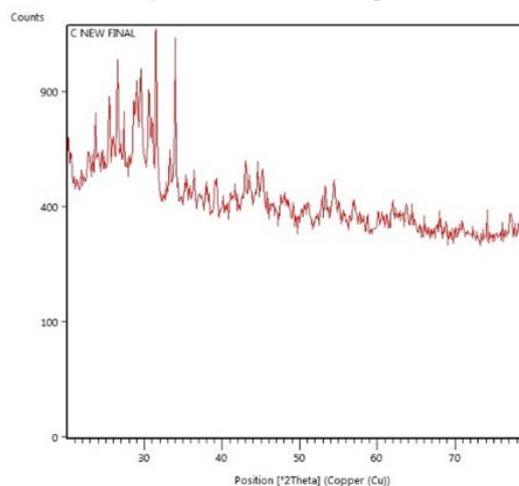


Figure 3: XRD of Sample C

3.1.2 Scanning Electron Microscope (SEM)

Scanning electron micrographs (SEM) has been recorded at chemistry department, Nahrain University. The samples were dispersed in alcohol and then treated ultrasonically so as disperse individual particles over a gold grids.

Figure 4,5 and 6 show the SEM of sample A, B and C respectively, these figures show the molecules agglomeration and crystal shape occurring and pour shape. From these figures the nanoparticles are grown with well-defined morphology. The nanoparticles are almost spherical in shape and have different diameters. It is clear from these figures that copper nano composite has regular structure after modifications.

3.2. Test of Lead Adsorption

3.2.1 Effect of Contact Time

Adsorption process was carried out using 50 ml solution containing 10ppm of lead solution and 0.1 g of adsorbent at room temperature with pH adjusted to 8. Fig. 7 shows Lead adsorption capacity at different time periods, 15 min until 135 min. From this plot it can be discovered that the rapid period was fast and accounted for a majority of lead uptake (between 50 to 70 %) and the time needed for this region is about 25 minutes. After the first rapid phase the rate of lead removal slowed down and this was accompanied by the slow phase which contributed relatively a tiny part to the adsorption process. The results reveals that sample A do the uptake good and still have got surface area for adsorption but sample B and C would decrease the adsorption efficiency after 100min and 80min respectively.

3.2.2 Effect of pH

The result of hydrogen ion concentration performs a vital role on the active sites of adsorbent as well as the heavy metal speciation during the adsorption reaction [3]. In order to exhibit the effect of pH on the adsorption uptake of the copper nanocomposite samples, experiments have been carried out at initial concentration of 10ppm and in the pH range 2.0–12.0.

Figure 8 shows the varying lead ion adsorption capacity on the catalysts, at selected pH ranging values. The discovered lower uptake in an acidic medium may be due to particle attrition, partial protonation of the functional groups and the competition between H^+ and metal ions for binding to the adsorption sites of the adsorbent [39]. Thus, removal of heavy metals increases with increasing solution pH, reaching a maximum value at an optimal equilibrium pH of around 10.

The lead sorption capacity increased steadily with increase in pH up to 10, these for

sample A and B while sample C is increased up to pH 5 after which it decreased slowly up to pH 12 for each samples. The decrease in uptake rate may be explained to metal hydroxylation yielding metal hydroxides or hydrated oxides which cause metal passivation [40].

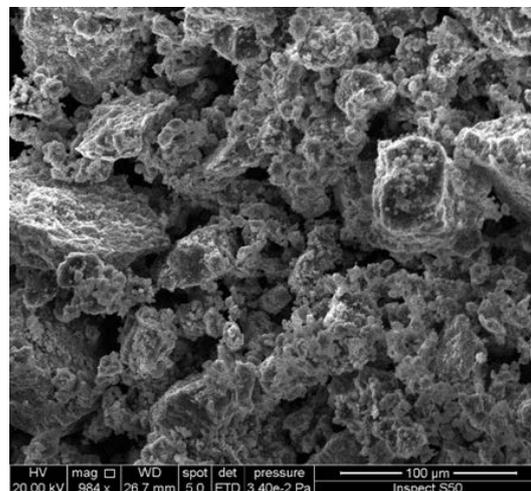


Figure 4: SEM of Sample A

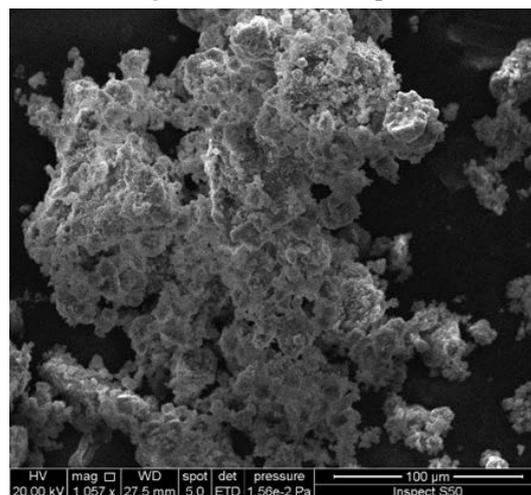


Figure 5: SEM of Sample B

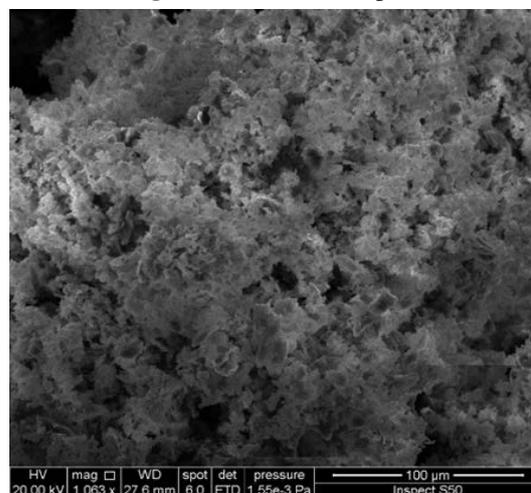


Figure 6: SEM of Sample C

Consequently, the prepared nanocomposite appears to be effective for the removal of heavy metal ions at pH ranging from 5 to 10, which might be promising in wastewater treatment. Figure 8 shows that the maximum adsorption efficiency for the three prepared samples are 84% with pH 10 for sample A, 75% with pH 10 for sample B and 90% with pH 5 for sample C.

From the above information, sample C is different from sample A and B due to the mixing with activated carbon, the adsorption is decreased at pH > 5.0, the Pb(II) gets precipitated due to hydroxide anions forming a lead hydroxide precipitate [12].

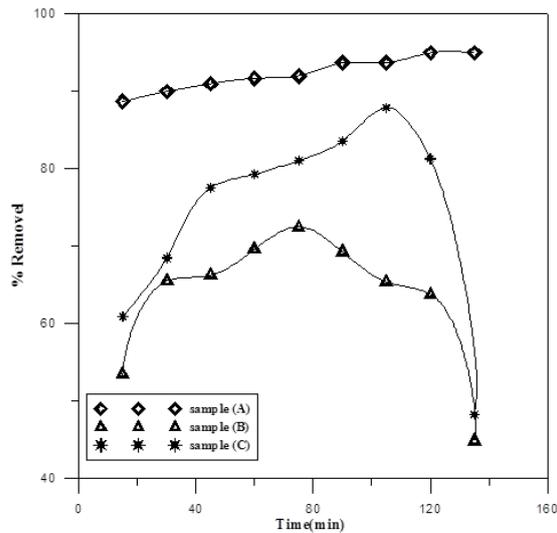


Figure 7: % removal of lead vs time for three samples

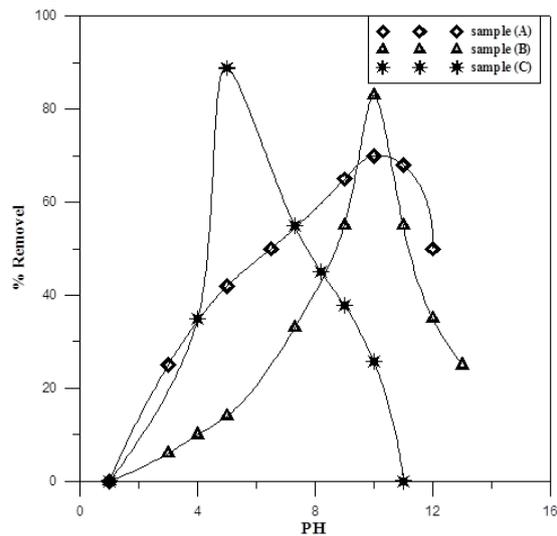


Figure 8: % removal of lead vs pH for three samples

3.2.3 Effect of Lead Concentration

The lead adsorption efficiencies have been examined as a function of the initial metal concentration. The selected concentrations of lead ion were taken between 10 to 50ppm, the pH of

the solutions was fixed to 8.0, the experiments were performed at surrounded temperature, and the lead concentration was measured after 30min of adsorption time. It had been found that an increase in lead concentration causes a reduction in adsorption efficiency by adsorbents as shown in figure 9. This decrease in adsorption happen with relation to the lead ion concentration could be explained with the low driving force for mass transfer or for a little active sites due to increase in metal concentration [9,10,12].

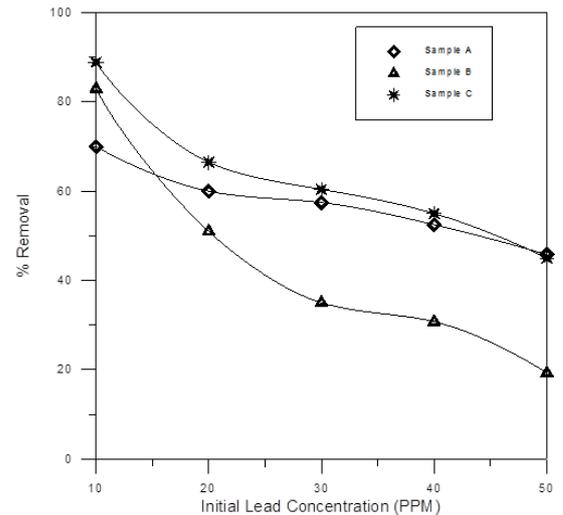


Figure 9: % removal of lead vs intial concentration for three samples

3.2.4 Effect of Adsorbent Dose

The results of adsorbent dose on the quantity of lead adsorbed efficiency for 10ppm concentration and 30min of stirring are shown in figure 10. The adsorption of lead ions is observed to increase as the amount of adsorbent is increased gradually from 0.05g to 0.25g. All other optimum conditions like pH was chosen, where select pH =10 for sample A and B and pH is 5 for sample C. The greatest adsorption efficiency of lead onto the catalysts adsorbents have been discovered, the figure show that possibility to remove 90 % of lead from aqueous solution for sample A, 70% for sample B and 74% for sample C. The maximum adsorption is obtained at the adsorbent dose of 0.2 g where a further increase thereafter, in the quantity of the adsorbent up to 0.25 g has no more effect to the adsorption rate. The data exhibited that increasing the adsorbent weight will enhance the adsorption of lead since the number of metal vacant sites increase as well. Longer contact time provides adequate duration for the adsorption process to take place and thus enhance the metal ions uptake from the solution onto the metal binding sites of the adsorbent [6].

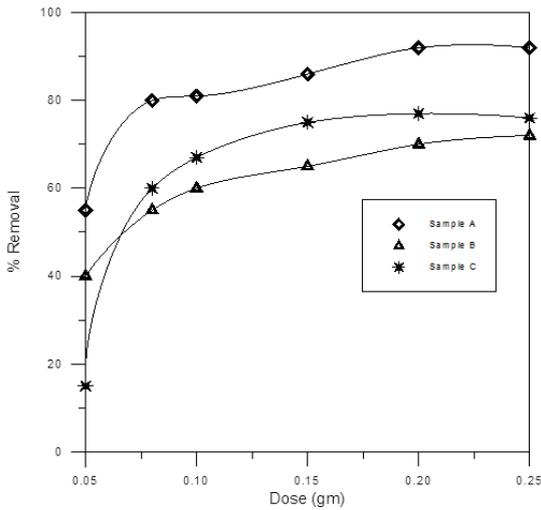


Figure 10: % removal of lead vs Adsorbent dose for three samples

4. Adsorption Isotherm

The equilibrium data for metal ions over the concentration range from 10 to 40 mg/L at room temperature have been correlated with the Langmuir and Freundlich isotherms as shown in equations below [6].

Langmuir isotherm

$$q_e = \frac{k_L C_e}{1 + \alpha_L C_e} \quad \dots(2)$$

Where, K_L is the constant related to overall solute adsorptivity (l/g); α_L is the constant related to the energy of adsorption (l/mg). Therefore, a plot of C_e/q_e versus C_e for the adsorption of Pb(II) ions onto sample A gives a straight line of the slope (α_L / k_L) and intercept ($1/ k_L$) as shown in Fig. (11).

Freundlich model

$$q_e = K_f C_e^{1/n} \quad \dots(3)$$

Where; K_f is the constant related to overall adsorption capacity (mg/g); $1/ n$ is the constant related to surface heterogeneity (dimensionless) [10]. where the Freundlich constants K_f and n , which respectively indicating the adsorption capacity and the adsorption intensity, have been calculated from the intercept and slope of the plot of $\ln q_e$ versus $\ln C_e$ for the adsorption of lead ions sample A as shown in Figure 12.

The Langmuir and Freundlich parameters for the adsorption of lead on sample A are shown in table 1. In alternative words, the Langmuir isotherm model fits well than that of Freundlich isotherm model when the R^2 values are compared in table1.

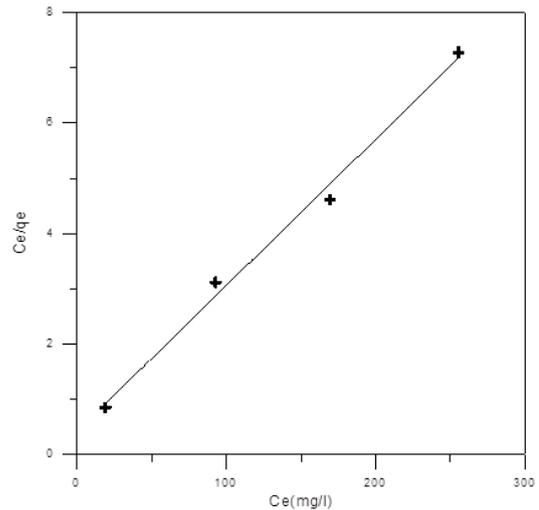


Figure 11: Langmuir isotherm of Pb ions adsorption using different amounts of sample A at constant temperature 25°C, contact time 0.5hr and pH 8

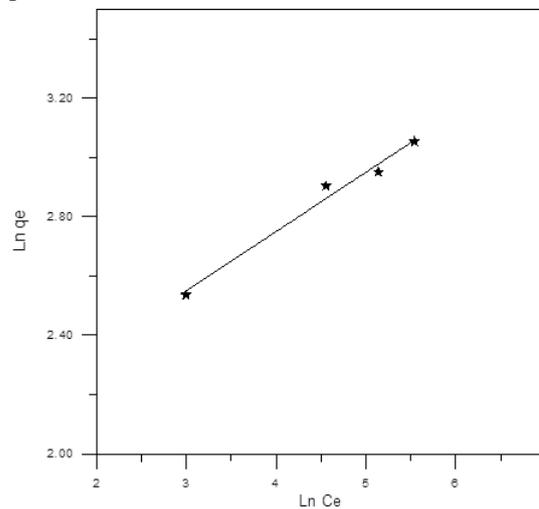


Figure 12: Freundlich isotherm of Pb ions adsorption using different amounts of sample A at constant temperature 25°C, contact time 0.5hr and pH 8

Table 1: of parameters and correlation coefficient (R^2) for each isotherm model

Langmuir	K_L	α_L	R^2
	0.041	50.1	0.988
Freundlich	K_f	n	
	10.21	4.23	0.974

5. Conclusions

Copper nano particle and it is nanocomposite a catalyst was used as vital source for adsorption of lead from solution. The efficiency of copper nanocomposite for the removal of metal ions depends on batch sorption of contact time, initial lead concentration (ppm), catalysts weight (g) and pH. The adsorption increase up to 40 minutes and

virtually constant after 160 to 200 minutes. At the certain time, the adsorption become constant because the low availableness of adsorbent surface area to disturb undesired heavy metals. Removal efficiency of lead obtained at pH from 4 to 8 with percent removal of lead of 96 % for sample A. Furthermore, the sample A can turns to be a good, inexpensive for adsorbent. Langmuir isotherm is better fitting for adsorption of lead than Freundlich and gave correction factor off 0.988.

6. References

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تحضير وتحليل مركبات النحاس النانوية كعامل مساعد لامتماز الرصاص من محلوله المائي

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الخلاصة

تم دراسة كفاءة الامتماز باستخدام مركبات النحاس النانوية لإزالة أيونات الرصاص من محلوله المائي. حضرت مركبات النحاس النانوية في المختبر بطرق دقيقة جدا. بعد إعداد العينات، تم تحليل العينات باستخدام حيود الأشعة السينية (XRD) مسح المجهر الإلكتروني (SEM) والتي أظهرت التشكل جيدة جدا وتوزيع متناسق مع شكل نانوي بلوري. من أجل دراسة كفاءة هذه المحفزات، تم اختبار جميع العينات باستخدام عملية الامتماز. أظهرت العينات المحضرة كفاءة جيدة جدا في إزالة الرصاص من محلوله المائي، وتصل إلى أقصى قدر من الكفاءة إزالة 96% للـ 0.05غم من العوامل المساعدة في درجة الحموضة 10، زيادة درجة الحموضة أو تقليلها من شأنه إعطاء نتائج غير مرغوب فيها لجميع العينات. أعطت معادلة لانكموير (Langmuir) تقارب للنتائج العملة أكثر من معادلة فريندليش (Freundlich) لامتماز الرصاص.