A study on effective adsorption of lead from an aqueous solution using Copper Oxide nanoparticles

Rumman Zaidi¹, Saif Ullah Khan², Ameer Azam¹, Izharul Haq Farooqi²

¹Department of Applied Physics, Zakir Hussain College of Engineering and Technology, Aligarh Muslim University

²Department of Civil Engineering, Zakir Hussain College of Engineering and Technology, Aligarh Muslim University.

* Corresponding author: ksaifullah.cv@amu.ac.in

Abstract. In this work, the co-precipitation method was used to prepare copper oxide nanoparticles, which were found 21.6 nm in size, as calculated from the Debye-Scherrer equation. The prepared material was characterized using XRD and UV-Visible spectroscopy and further evaluated for its efficiency in adsorptive removal of Pb(II). To optimize the adsorption process was tried in the range: nanoparticle dosage (0.1-1 g/L), reaction time (0.5-2 hour), initial pH of the solution (2-6), and initial concentration of Lead (5-15 ppm). A linear fitting of equilibrium data to Langmuir and Freundlich isotherms was also explored and carried out. The results indicated that data obtained from the experiment fit better with the Freundlich isotherm model, attaining $q_{\text{max}}$ for lead ions as 97 mg/g. Kinetic studies showed better fitting with a pseudo-second-order kinetic model signifying the mechanism of adsorption as chemisorption. The result advocate that synthesized adsorbent proved useful in adsorbing lead from water.

Keywords: lead removal; co-precipitation; metal oxide; chemisorption; adsorption kinetics

1. Introduction

Water pollution is caused by chemicals that lower the quality of water and make it unusable. The increase in water pollution may be attributed to the release of untreated toxic metal-rich industrial waste into water bodies. These heavy metals and a few other inorganic materials are very harmful to the human body and may seriously impact one’s health[1]. In the last few years, there has been an increase in water pollution by inorganic contaminants such as arsenic, copper, Lead, chromium, mercury, cadmium, fluoride, nitrate, etc., with the Lead being one of the most significant among them[2]. Lead is known to affect the nervous system resulting in a short-term loss of memory. In cases of high levels of accumulation in children, it can prove to be fatal as well. Kidney damage behavioral problems, attention deficit to Langmuir and Freundlich isotherms are also explored and carried out. The results indicated that data obtained from the experiment fit better with the Freundlich isotherm model, attaining $q_{\text{max}}$ for lead ions as 97 mg/g. Kinetic studies showed better fitting with a pseudo-second-order kinetic model signifying the mechanism of adsorption as chemisorption. The result advocate that synthesized adsorbent proved useful in adsorbing lead from water.

Many physicochemical methods including chemical precipitation, coagulation, chemical, and photocatalytic oxidation-reduction are used to remove heavy metals from polluted water[5]. Though effective to some extent, most of these methods have not been...
fully exploited due to their high cost of operation and their limitation to be selective to heavy metal[6]. Of these all, adsorption is a simple, inexpensive technique that provides both efficacy and flexibility in designing a process for removing different contaminants from contaminated water[7,8]. In recent years, nanotechnology has been advancing, which has led to the synthesis of nanomaterials to be used for wastewater treatment[9]. Nanoparticle synthesis has garnered much attention due to the high surface area, electrical and magnetic properties resulting in excellent adsorption potential[10]. Nanomaterials and their application in water treatment to remove toxic substances is a significant research area currently being explored. Recently, metal oxide nanoparticles have attracted much attention in removing heavy metals, organic and inorganic contaminants from polluted water. Various metal oxide nanoparticles, such as alumina [11], manganese oxide nanoparticles [12,13], iron oxide nanoparticles [14], zirconium oxide nanoparticles [15] have been recently employed as nanosized adsorbent materials for removing heavy metals. Here copper oxide nanoparticles were selected because of their low toxicity, ease of synthesis as raw materials are readily available and can be tailored to have a large surface area, good photocatalytic properties as well its anti-bacterial properties. Thus making it noteworthy for wastewater by removing organic and inorganic pollutants [16,17]. In this paper, we aim to successfully remove Lead from aqueous solutions using synthesized copper oxide nanoparticles. Synthesized nanoparticles were characterized by XRD, FTIR, and SEM. Various parameters like contact time, initial pH, initial Pb(II) concentration, and nanoparticle dosage were assessed for the effective elimination of Pb(II) from contaminated water. Kinetics of adsorption and isotherm models were also explored to establish the nature of sorption occurring.

2. Experimental Details

2.1. Materials and Apparatus required
The required chemicals were Copper (II) nitrate trihydrate, Ammonium standard solution, Hydrochloric acid, Sodium hydroxide, Lead standard solution (traceable to SRM from NIST consisting of 0.5 mol/l of Pb(NO₃)₂ in HNO₃) acquired from Merck, India. pH meter (HITEC-102 Digital) was used for checking pH. A mechanical rotary shaker (Brunswick-TM 2350) was used for shaking the samples over it. The magnetic stirrer (Scientific-SSI LAB) was used for preparing the adsorbent

2.2. Preparation n of Copper Oxide nanoparticles
Copper oxide nanoparticle was prepared at a temperature of 100 °C by an easy and straightforward co-precipitation process. Copper (II) nitrate trihydrate was thoroughly dissolved in a beaker of 500 ml capacity in deionized water. This solution was then stirred continuously for a few hours. Then, to increase the solution pH around 8-9, the ammonia solution was added slowly, resulting in the precipitation of copper oxides. The obtained extract was filtered and then calcinated for four hours at 400 °C. The dried product is crushed and collected for further use.

2.3. Adsorption experiments
A required amount of lead standard solution was used to prepare a Pb(II) stock solution of concentration1000 mg/L, and used for batch adsorption studies. By subsequent dilution, a required concentration of the solution was obtained. For pH change, sodium hydroxide and hydrochloric acid were used.
Using the synthesized nanomaterial, batch studies were conducted by exploring different parameters like initial Pb(II) concentration, nanoadsorbent dosage, contact time, and initial pH. For various experimental runs, 0.1,0.5, and 1 g/L of nanoadsorbent were added to a series of flasks, having Pb(II) conc. 5,10 &15 mg/L stirred at 350 rpm over a mechanical shaker for 0.5 to 2 hours as per experimental design. Further, centrifugation was done at high rpm (5000rpm) for 15 minutes. Pb(II) in samples was analyzed by AAS (PinAcle 900F). Value of qₑ (mg/g), were calculated as follows (Eq.1).
\[ q_e = \frac{V \ast (C_o - C_e)}{W} \]  

(1)

Where \( C_o \) & \( C_e \) are lead conc. initially and finally, \( V \) (L) is the volume and \( W \) (g) is the adsorbent mass.

2.4. Characterization

The structural properties of the prepared nanoparticle were assessed using XRD (Rigaku Miniflex II) with Cu-K\( \alpha \) radiations equivalent to 1.5418A\(^\circ\) and UV–visible spectroscopy in the range of 200–800 nm using Perkin Elmer Spectrophotometer. FTIR was done by Perkin Spectrum 100 FTIR spectrophotometer in the wavelength range of 400 to 4000 cm\(^{-1}\) by using KBr pellets as a reference point. The surface morphology and chemical composition of the synthesized nanomaterial were studied by the Scanning electron microscope.

3. Results and Discussions

3.1. Characterization

3.1.1. XRD Analysis

The prepared copper oxide nanoparticles' structural properties were studied using the XRD technique, as shown in Figure 1. The figure shows three prominent peaks at 2 theta of 35.5\(^{\circ}\),38.7\(^{\circ}\) and 49.9\(^{\circ}\) which occurred as a result of the diffraction from (002),(111), (202) planes copper oxide phase. The XRD pattern shows that the sample is crystalline in nature, and the peaks are attributed to the monoclinic crystal system having a space group of C2/c (JCPDS file no-041-0254). The average Crystallite size was found to be 21.6 nm using Scherrer equation.

3.1.2. Optical Analysis

The prepared material's optical properties were analyzed using UV–visible absorption spectroscopy, as shown in Figure 2. UV-visible spectra show an absorbance peak at 325 nm, as a result of dipole moment oscillations.

It is observed that the absorption coefficient in semiconductors near to the fundamental edge is dependent on photon energy. Tauc’s equation gives this observed dependence of absorption coefficient and incident photon energy. It’s a useful way to find the optical bandgap of the prepared material. As per Tauc ‘s relationship, the direct bandgap semiconductor material's absorption coefficient is determined as given below[18] (Eq. 2).

\[ \alpha h\nu = B(h\nu - E_g)^n \]  

(2)

Figure 1. X-ray Diffraction pattern of CuO nanoparticles
Here, $\alpha = 2.302 \, \text{A/t}$, in which $A$ is absorbance as obtained from UV–visible absorption spectroscopy and $t$ is the thickness of the cuvette, $h$ is the Planck’s constant, $B$ is a constant, $E_g$ is the optical band gap, $\nu$ is the incident photon frequency and $n$ is the index that is dependent upon the type of the electronic transition accountable for optical absorption.

The optical band gap calculated from Tauc’s plots absorption spectra for CuO nanoparticles is shown in Figure 2(b). The results showed a blue shift in which the optical band gap obtained for the nanomaterial ($E_g=3.4 \, \text{eV}$) was greater than the corresponding bulk copper oxides ($E_g=2.1 \, \text{eV}$). This higher value of the bandgap for nanomaterial was attributed to a decrease in particle size. When the nanomaterial size is significantly reduced and becomes lesser than Bohr’s exciton, a phenomenon called the quantum confinement is observed with which a decrease in particle size, the bandgap energy begins to increase.

![Figure 2. (a)UV-Visible spectra and (b) Tauc Plot.](image)

### 3.1.3. FTIR Analysis

The Fourier transform infrared spectroscopy (FTIR) for prepared CuO nanoparticles is shown in Figure 3. FTIR spectra are performed between the range of 400- 4000 cm$^{-1}$ to study the functional groups present in the material. The bands at 3400 cm$^{-1}$ and 1620 cm$^{-1}$ are due to the O-H stretching and O-H-O bending, respectively. This occurs mainly due to the presence of adsorbed water. The bands obtained in the range of 469-558 cm$^{-1}$ can be assigned to Cu-O stretching vibrations, as confirmed by previous studies [19].

![Figure 3. FTIR spectra of CuO Np’s.](image)
3.1.4. **SEM Analysis**

SEM for Copper oxide nanoparticles was performed to understand its surface morphology. The SEM micrograph of the material is shown in Figure 4. The micrograph shows that material appears to be agglomerated, and it is only possible to identify clusters of nanomaterials. These CuO nanoparticles have been found to have a more homogeneous morphology.

![SEM micrograph of CuO NPs](image)

**Figure 4.** SEM micrograph of CuO Np’s.

3.2. **Role of pH**

Solution pH directly affects the charge and state of the functional group of sorbents and the metal ion to be adsorbed, making it an essential parameter to study [20]. The adsorption experiments were explored in the pH range of 2.5-7.5 to examine the effect of pH on percentage removal of lead ions. Figure 5 clearly shows that the absorption potential increased with an increase in pH and was found to be maximum at pH 4.5. Subsequently, adsorption potential dropped significantly when pH was further increased, signifying the favorable adsorption of Pb(II) on copper oxide nanoparticles within 4 to 5pH. The results showed a maximum removal efficiency of 67.4% at a pH of 4.5 for 0.5 g/L dosages on nanoadsorbent in half-hour of reaction time.

Lead adsorption greatly depends on the chemistry of lead ions present in water. Lead exists in different forms like Pb(OH)$_3^-$, Pb$^{2+}$, Pb(OH)$^+$, Pb(OH)$_2$, etc., depending on the pH. At a much lower pH, the adsorbent surface is highly protonated, and positive charges are attained, resulting in the repelling the positively charged lead ions. Hence a lower percentage of lead removal is reported at low pH values. Lead occurs as an ion that is negatively charged [Pb(OH)$_3^-$] at higher pH values, which electrostatically repels the adsorbent's negatively charged surface. As a result, the removal potential of Lead got drastically reduced at a higher pH value. These results are reasonably consistent with reported literature [21].
3.3. Role of contact time and lead concentration

Adsorption of lead ions by copper oxide nonabsorbent at a different contact time and varying initial lead ion concentrations was explored to know the time duration required for adsorption to reach an equilibrium point. Therefore, short time periods were selected for analysis, and findings demonstrated that with longer durations of time, the removal efficiency of Lead was high for all initial Pb(II) concentrations (5, 10, 15 mg/L), as shown in Figure 6(a). It is also worth noting that removal efficiency increased in the initial stages of the adsorption process. This may be attributed to significant amounts of readily accessible active sites for adsorption and the presence of a large amount of lead ions present in the solution. With time, adsorption reached an equilibrium point, where both the free binding sites and the lead concentration gradient got reduced. Similar results were also reported by various studies carried out in the past [22].

Another thing worth noting was that at higher initial lead concentration, increased reaction time only slightly affected the removal efficacy as a large amount of Pb(II) is adsorbed on all available binding sites. Moreover, assessing the initial lead concentration assisted in analyzing the adsorption process for an optimum reaction time. Hence, all successive adsorption experiments were performed for 2 hours at an optimum pH of 4.5 to ensure maximum adsorption. It was also observed that for all three different dosages of nanoadsorbent, removal capacity decreased with an increase in initial lead concentration, as indicated in Figure 6(b). This may be because only small adsorption sites are available for a fixed amount of adsorbent that gets occupied with a rise in Pb(II) concentration. These findings, too, are fairly in line with several other researches in past [23].

Thus, maximum removal of about 84.2% was attained with an initial lead concentration of 5mg/L and a reaction time period of 2 hours.

![Figure 5](image_url). Effect of pH on the removal of Pb(II).

![Figure 6](image_url). (a) Effect of contact time and (b) Pb(II) concentration on the removal efficiency.
3.4. Role of nanoparticles dosing

To assess the influence of nanoparticles’ dosage on the efficacy of lead adsorption, three different dosages of 0.1, 0.5, and 1 g/L were tried. With the rise in the dosage of the nanoadsorbant, there is a significant increase in the adsorptive removal of Lead, as shown in Figure 7. This improvement in the adsorptive removal of Lead may be in relation to increase in the specific area available and the higher possibility of the collision between adsorbent material and metal ions. The findings of other researchers are also fairly aligned with our study[24]. However, when designing a suitable process for lead removal, other factors such as cost, availability of material, and initial concentration present in water should always be considered for achieving toxicant free water.

![Figure 7. Effect of adsorbent dosage on the removal efficacy of Lead.](image)

3.5. Adsorption Isotherm

Evaluation of adsorption isotherms is an essential step in understanding the relationships between adsorbent and adsorbate. To increase the adsorption efficiency, it is essential to create an adequate understanding of adsorption equilibria[25]. In the current work, the isotherm studies are applied to understand the process of adsorption. The linear variation of the Langmuir isotherm model can be expressed (Eq. 3).

\[
\frac{C_e}{q_e} = \frac{1}{q_{max}} b + \frac{C_e}{q_{max}}
\]  

(3)

Where, \( q_e \) = adsorption capacity(mg/g), \( C_e \) = equilibrium concentration of lead in water (mg/L), \( q_{max} \) = maximum adsorption capacity of adsorbent(mg/g) and \( b \) = constant of langmuir isotherm

For understanding the adsorption process, a graph was plotted between \( C_e/q_e \) vs. \( C_e \), as shown in Figure 8(a). Parametric values such as \( q_{max} \) was obtained from the slope of the graph, while the value of \( b \) was evaluated from the graph’s intercept (Table 1). The data obtained from batch adsorption experimental results fitted well with the isotherm, as evident from the high value of \( R^2 \) which is a correlation coefficient. This shows that monolayer, homogenous adsorption occurred on the active sites present on the surface of the nanoadsorbent, suggesting equivalent activation energy for all adsorption process.

To understand whether adsorption was favorable or not, another vital parameter called separation factor (\( R_L \)) was calculated from the isotherm model. The separation factor is calculated (Eq.4).
If the value of \( R_L < 1 \), then the adsorption process is favorable. In this study, the value of \( R_L \) obtained is 0.46, which indicates the adsorption process occurring here was quite favorable.

A linear form of the Freundlich adsorption isotherm model is given below (see Eq. 5).

\[
\ln(q_e) = \ln(K_f) + \left( \frac{1}{n} \right) \ln(C_e)
\]

Where, \( K_f \) is the Freundlich coefficient constant, and \( n \) = intensity parameter. This isotherm model presumes that the multilayer adsorption process occurs on the heterogeneous active sites of the adsorbent. A graph is plotted between \( \ln(C_e) \) against \( \ln(q_e) \), and values of the parameters \( n \) and \( K_f \) are determined from the slope and intercept of the graph, as shown in the Figure 8(b). The graph here shows a higher value of \( R^2 \) (Table 1) for all three adsorbent dosages (0.1, 0.5, and 1 g/L), thereby indicates multilayer adsorption. The value of \( n \) obtained from the Freundlich model is required to accurately validate the type of adsorption[26]. Therefore, if \( n \) is equal to 1, then the adsorption process is linear. The chemical adsorption process occurs when the value of \( n \) is less than 1. Consequently, physical adsorption occurs if the value of \( n \) is larger than 1. Here, the obtained value of \( n \) is 0.264, suggesting the ongoing adsorption process to be chemical in nature.

As noted from the graph, the value of the regression coefficient \( (R^2) \) for the Freundlich isotherm model for all three different dosages of nanoadsorbent was higher than in case with the Langmuir model. The above statement suggests that the Freundlich model fitted better to explain the adsorption of lead by prepared copper oxide nanoparticles.

\[ R_L = \frac{1}{1 + bC_0} \]  (4)

3.6. Adsorption Kinetics

Adsorption of lead ions by prepared copper oxide nanoadsorbent with respect to time is shown in Figure 9(a-b). The rate of adsorption process is understood by kinetics, which can help design and optimize the adsorption process[27]. The two kinetic models were applied at three different initial lead concentrations (5, 10, 15 mg/L) with two different nanoadsorbent dosages (1 and 0.5 g/L) at a fixed pH of 4.5.

The linear form of the Pseudo-first-order kinetics can be presented as (see Eq. 6):
A graph plotted between log (q_e-q_t) against time showed a significantly lower value of R^2 as well as the inconsistency between q_e experimental and calculated, indicating its unsuitability in explaining adsorption mechanism here (Table 1).

This kinetic model of pseudo-second-order kinetics is represented (See Eq. 7) below, which presumes that the rate of adsorption directly proportional to twice the active sites [28,29].

$$\frac{t}{q_t} = \frac{1}{K_{ads} q_e^2} + \frac{1}{q_e}$$  \hspace{1cm} (7)

Where k_{ads} (g/mg/h) is the adsorption rate constant, the K_1 represents the first-order rate constant, q_t and q_e represent the total amount of adsorbate getting adsorbed over the material surface at any time “t” & at equilibrium. A graph is plotted between t/q_t against time, and a slope and intercept of a straight line are used to calculate parameters such as q_e(calculated) and K_{ads}(Table 1). Also, R^2 values are greater than 0.99 for both adsorbent dosages; therefore, pseudo IIInd order equation can more precisely predict the rate of adsorption process.

**Figure 9.** Plots depicting pseudo IIInd order kinetics for (a) adsorbent dose 0.5 g/L & (b) adsorbent dose 1 g/L.
Table 1. Parametric values and constants obtained from isotherms & adsorption kinetics.

| Isotherm models          | Parameter                        | R²     |
|--------------------------|----------------------------------|--------|
| Langmuir Isotherm        | $q_{\text{max}} = 97 \text{mg/g}$ | 0.8596 |
|                          | $b = 0.103$                      |        |
| Freundlich isotherm      | $K_f = 4.8196$                   | 0.9454 |
| Adsorption Kinetic models|                                   |        |

4. Conclusions

In this study, CuO nanoparticles were synthesized successively using a co-precipitation technique with a crystallite size of 21.6 nm computed from XRD. The synthesized adsorbent successfully achieved 84.2 % of lead removal with the initial conc. of 5ppm with 1 g/L dose at pH around 4.5. The results indicate Freundlich isotherm fitting better achieving $q_{\text{max}}$ as 97 mg/g. Moreover, kinetic studies showed better fitting with a pseudo-second-order model signifying the process of adsorption to be chemisorption. The experimental study shows that the synthesized adsorbent removed lead efficiently from aqueous solution. The results provide a pathway in applying copper oxide nanoparticles and their different nanocomposites to remove toxic metals from water.

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