

# Adsorption of Copper and Lead ions from aqueous solutions using Nickel oxide nanostructure

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**Abstract-** Nickel oxide (NiO) has been synthesized by novel simple method using solution combustion technique employing glycine as a fuel. The synthesized NiO nanostructure is characterized for Surface area by BET analyzer. The ability of NiO nanostructures as adsorbent was investigated for adsorptive removal of Cu (II) and Pb (II) ions from aqueous solutions. Various physical & chemical parameters such as  $p^H$ , initial metal ion concentration, adsorbent dosage and equilibrium contact time were studied. The optimum solution  $P^H$  for adsorption of Pb (II) and Cu (II) ions in aqueous solutions were at 9.0 and 7.0 respectively and the optimum contact time was found to be 30 min. The adsorption isotherms were obtained using concentrations of the metal ions ranging from 0.1 to 1.0 mg/l. This study revealed that NiO nano structure is an effective adsorbent for removal of Cu (II) and Pb (II) ions from aqueous solutions.

**Key words:** Solution combustion, Nickel oxide nano structure, Heavy metals, Adsorption studies, Optimum solution, BET Analyzer, Adsorption Isotherm

## 1. Introduction:

Because access to safe drinking water is the key to protect public health, clean water has become a basic need of all properly functioning societies[1]. Despite their presence at low concentration ranges, environmental pollutants possess serious threats to freshwater supply, living organisms, and public health[2]. Contamination of water with toxic metal ions (Hg(II), Pb(II), Cr(III), Cr(VI), Ni(II), Co(II), Cu(II), Cd(II),) etc becoming a severe environmental and public health problem. In order to achieve environmental detoxification, various techniques like adsorption, precipitation, ion exchange, reverse osmosis, electrochemical treatments, membrane filtration, evaporation, flotation, oxidation and biosorption processes are extensively used.[3,4,5]. Among these, adsorption is a conventional but efficient technique to remove toxic metal ions and bacterial pathogens from water. Activated carbon, possessing high BET surface areas, was adopted as an adsorbent to remove the heavy metals and better adsorption efficiency was achieved[6]. Removal of heavy metals from industrial waste water streams has been studied by adsorption phenomenon due to the high threat of such pollutants to public health and environment[7,8]. The adsorption process is arguably one of the more popular methods for the removal of heavy-metal ions because of its simplicity, convenience, and high removal efficiency[9]. Development of novel and cost-effective nanomaterials for environmental remediation, pollution detection and other applications has attracted considerable attention. Recent advances suggest that many of the issues involving water quality could be resolved or greatly ameliorated using nanoparticles, nanofiltration or other products resulting from the development of nanotechnology [10, 11].

Nanoparticles exhibit good adsorption efficiency especially due to higher surface area and greater active sites for interaction with metallic species[12,13]. Furthermore, adsorbents with specific functional groups have been developed to improve the adsorption capacity[14]. Recently, application of nanoparticles for the removal of pollutants has come up as an interesting area of research. The unique properties of nanosorbents are providing unprecedented opportunities for the removal of metals in highly efficient and cost-effective approaches, and various nanoparticles and dendrimers have been exploited for this purpose[15,16]. Nanoscale metal oxides potentially offer a more cost efficient water treatment and remediation technology due to their size and adsorption efficiency[17,18].

We here in the present study able to synthesize nickel oxide using solution combustion technique by employing glycine as a fuel. Subsequently the synthesized metal oxide used for adsorption studies for the removal of metals like Lead and Copper.

## **2.Experimental**

### **2.1Material and Methods**

All chemicals used in this study were of analytical grade. Nickel nitrate and glycine were obtained from Merck, India. The NiO nanostructure was prepared using Nickel nitrate by solution combustion process with glycine employed as a fuel. Specific amount of Nickel nitrate and glycine were dissolved in Millipore water and kept for stirring for 30 min. The solution was then transferred in to a porcelain crucible and kept on hot plate until syrupy state attained. The contents were heated in a muffle furnace at 500°C for 3 hr. The crystal water was gradually vaporized during heating and when a crucible temperature was reached, a great deal of foams produced and spark appeared at one corner which spread through the mass, yielding a black voluminous and fluffy product in the container. The product was carefully grinded by using mortar. A fine black colored Nickel Oxide was obtained.

The surface area of the particles was measured by a Brunauer, Emmett, and Teller (BET) surface area analyzer (Nova 1200e) for nitrogen adsorption. The BET method was carried out under relatively high vacuum and measured primarily the external area of the particles and aggregates.

### **2.2 Adsorption Studies:**

Nanoparticles formed by metal or metal oxides are inorganic nanomaterials, which are used broadly to remove heavy metal ions in wastewater treatment. Nanosized metal oxides provide high surface area and specific affinity. Besides, metal oxides possess minimal environmental impact and low solubility and no secondary pollution have been adopted as sorbents to remove heavy metals.

Adsorption studies were performed by batch process by taking 0.35 gm of synthesized NiO in a 100 ml clean and dried stoppered bottle. Known concentration of Lead (II) and Copper (II) solution was added in the same stoppered flask. The flask was placed on a mechanical shaker (180 rpm rate) and agitated for a time period of 35 min.. The solution was then filtered using Whatman filter paper no. 41 and the aliquot is used for metal ion concentration determination using Atomic Absorption Spectroscopy.

In order to obtain the optimal conditions for Pb(II) and Cu(II) removal by NiO nanoparticles, the effect of various parameters such as adsorbent mass, contact time, pH, and initial concentration of metal ions were investigated. The surface area of the freeze-dried NiO particles measured using the Brunauer, Emmett, Teller (BET) method and was found to be 71.985 m<sup>2</sup>/g.

## **3.Results and Discussion**

### **3.1 Effect of pH:**

The adsorption experiments were carried out in a series of 250 ml Erlenmeyer flasks containing 0.35 g synthesized NiO with different concentration of Lead (II) and Copper (II) ions at serial pH conditions (3 to 10). The prepared samples were stirred in mechanical shaker for different time intervals. Then solid/liquid phases were separated by filtration. The concentration of the Lead (II) and Copper (II) ions before and after adsorption was determined using Atomic Absorption mass spectrometry (Agilent 240 AA). The recovery efficiency at different pH conditions was shown in the Fig.1 & Fig.2.

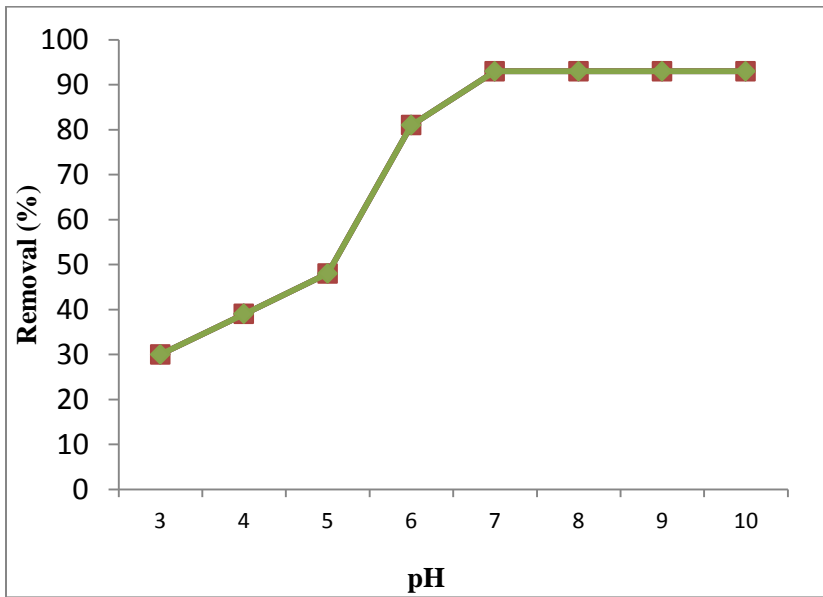


Fig.1-Adsorption percentage of copper at  $p^H=7.0$

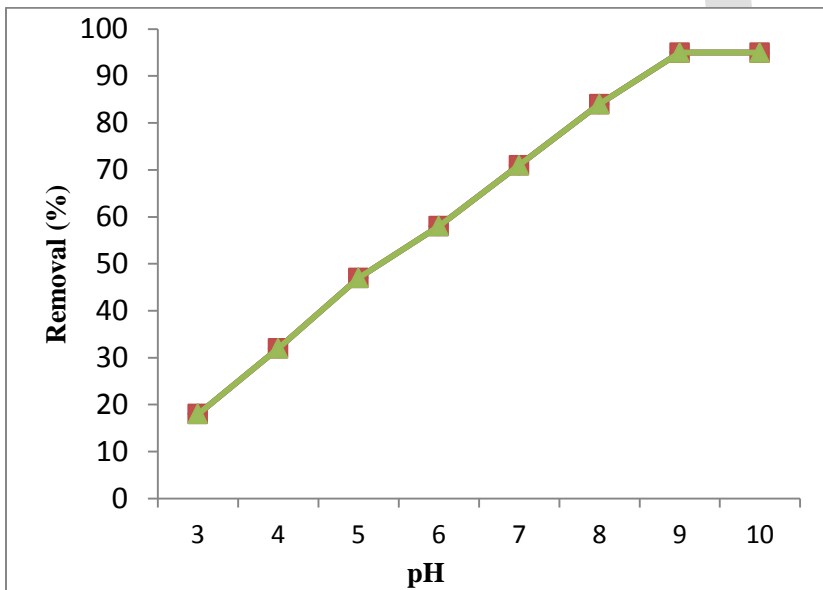


Fig.2-Adsorption percentage of Lead at  $p^H=9.0$

### 3.2 Effect of Addition of Oxide:

Adsorbent dosage is an important parameter because it determines the capacity of an adsorbent for a given initial concentration of the adsorbate. The effect of adsorbent dosage was studied on Lead (II) and Copper (II) from aqueous solutions by varying the amount of NiO ranging from 0.1 g to 0.5 g, while keeping other parameters constant. (pH, agitation speed, temperature, initial Lead and Copper ion concentrations and contact time). Adsorbent dosage of 0.35g is found to be optimum for effective removal of Copper (II) and Lead (II) represented graphically as shown in Fig.3 & Fig.4.

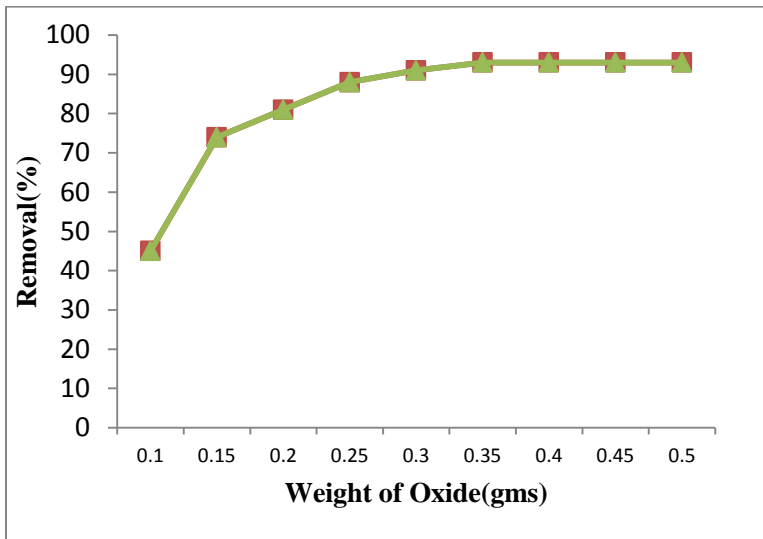


Fig.3-Percentage of copper ions removal at different adsorbate concentration.

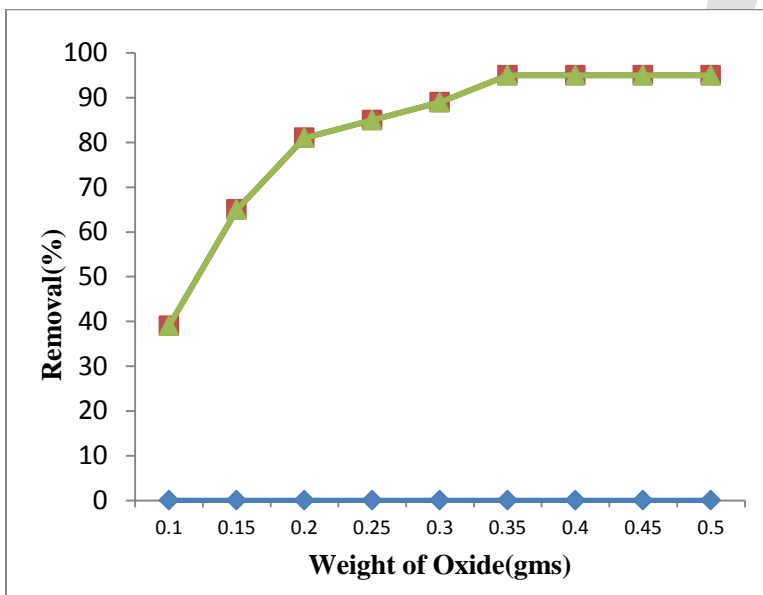


Fig.4-Percentage of lead ions removal at different adsorbate concentration

### 3.3 Effect of Stirring time:

The effect of time on the removal efficiency of Copper (II) and Lead (II) were also studied at fixed adsorbent dosage of 0.35g at a pH of 7 for Copper(II) and at a pH of 9 for Lead(II) ions at different time intervals starting from 20, 40, 60, 80, & 100 min and the results are shown graphically in Fig.5 & Fig.6. It has been established that optimum time required to attain the equilibrium between the Copper (II) and lead (II) ions adsorbed on NiO particles was in between 35-40 min.

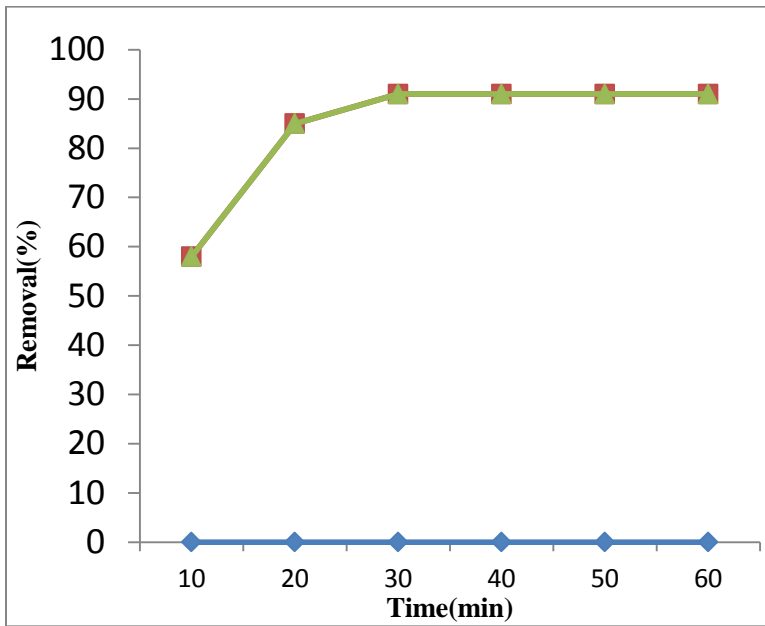


Fig.5-Adsorption capacity of Copper at different time intervals

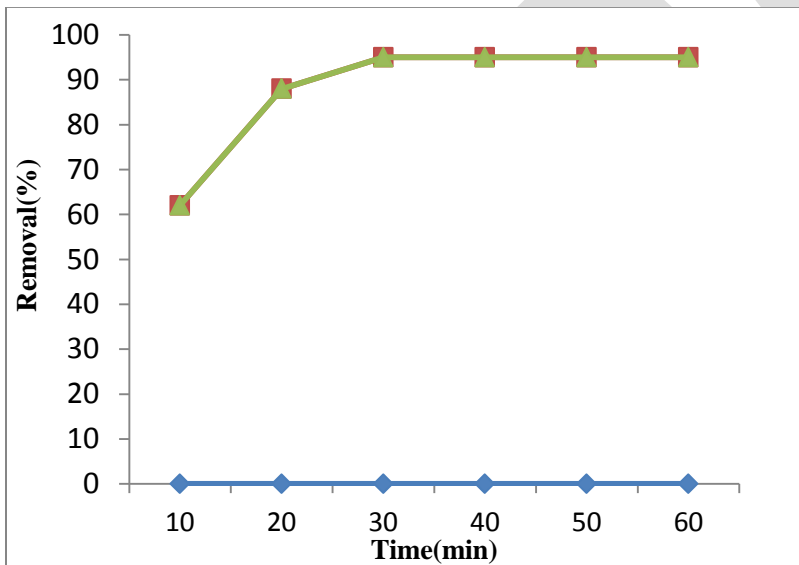


Fig.6-Adsorption capacity of Lead at different time intervals

#### 4. Adsorption isotherm:

Adsorption isotherm is important to describe how solutes interact with the sorbent. Developing an appropriate isotherm model for adsorption is essential to the design and optimization of adsorption processes. The more common models used to investigate the adsorption isotherm are Langmuir and Freundlich equations. The equilibrium adsorption isotherms are important in determining the adsorption capacity of metal ions and diagnose the nature of adsorption onto the NiO nanostructures.

The equilibrium adsorption capacity of adsorbent was calculated by the following equation

$$q_e = V (C_0 - C_e) / W$$

Where  $q_e$  is the equilibrium adsorption capacity of adsorbent in mg metal/g adsorbent,  $C_0$  is the initial concentration of the metal ions in mg/l,  $C_e$  is the equilibrium concentration of metal ions in mg/l,  $V$  is the volume of metal ions solution in L, and  $W$  is the weight of the adsorbent in g. The experimental results of this study are fitted with Langmuir & Freundlich models shown in Fig.7 to Fig.10

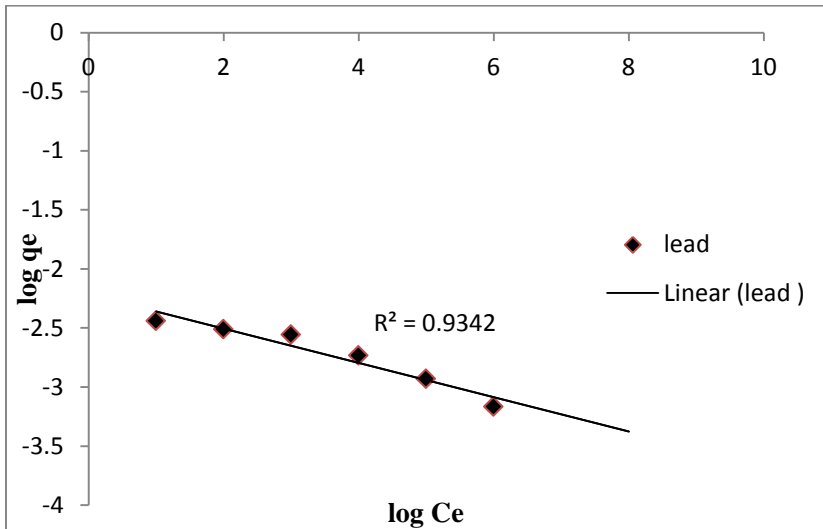


Fig.7-Freundlich isotherm for adsorption of Pb onto NiO nano powder

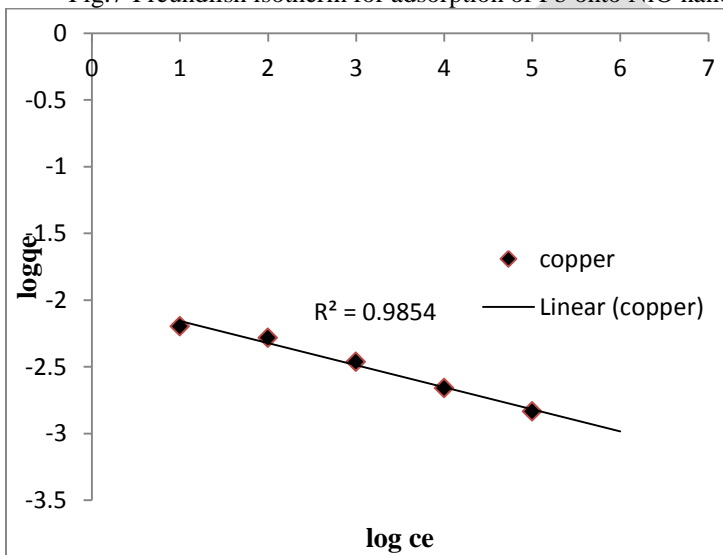


Fig.8-Freundlich isotherm for adsorption of Cu onto NiO nano powder

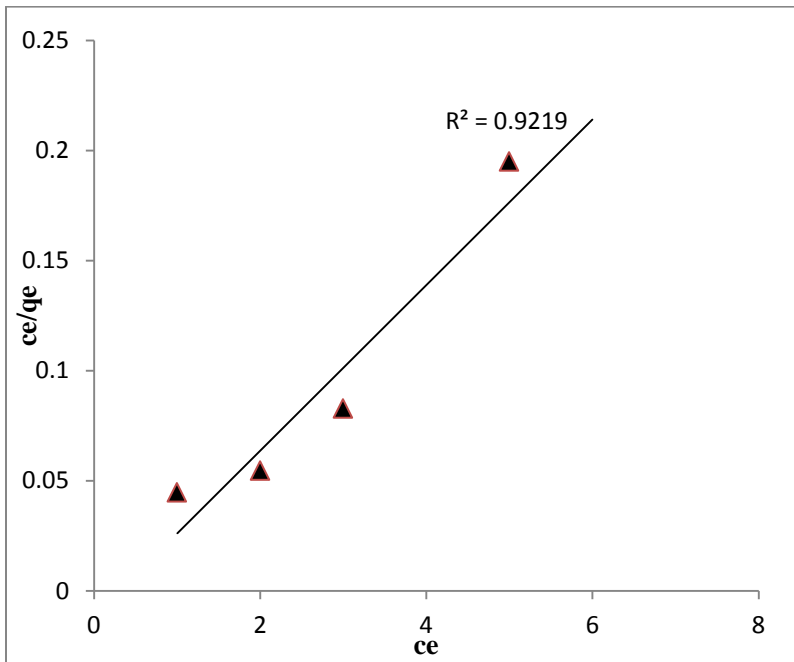


Fig.9-Langmuir isotherm for adsorption of Cu onto NiO nano powder

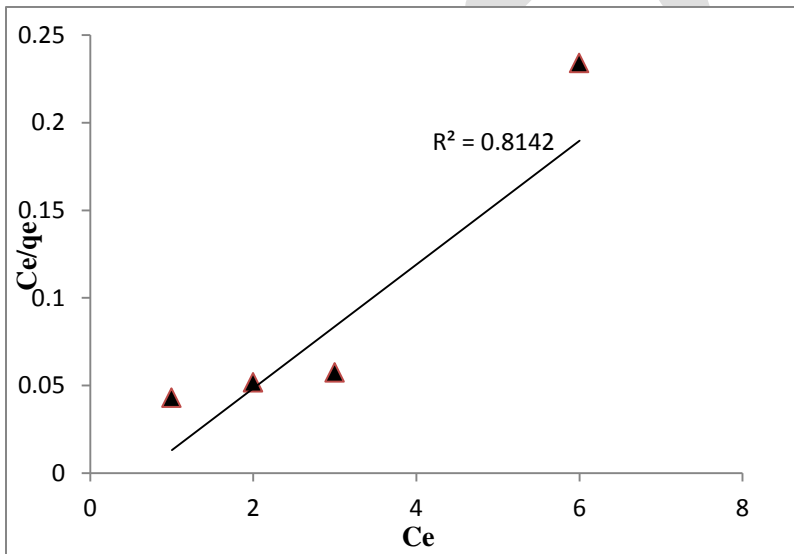


Fig.10- Langmuir isotherm for adsorption of Pb onto NiO nano powder

**Acknowledgement:**

Generous funding from the University Grants Commission (UGC),New Delhi ,Government of India in the form of Major Research Project( Project file no: F.No.42-327/2013(SR) is gratefully acknowledged.

## Conclusions

NiO nanostructure has been synthesized using solution combustion method employing glycine as a fuel. The produced Nickel oxide was characterized by BET and the surface area was found to be 71.985 m<sup>2</sup> /g. In this study, batch adsorption experiments for the removal of Pb (II) and Cu (II) ions from aqueous solutions have been carried out using the synthesized nano nanostructure as adsorbent. The adsorption characteristics have been examined at different pH values, contact time, adsorbent dosage and initial metal ion concentrations. The obtained results can be summarized as follows:

- ❖ The surface area of the freeze-dried NiO particles measured using the BET method and was found to be 71.985 m<sup>2</sup> /g.
- ❖ The pH experiment result showed that the removal of Pb increased significantly as the pH increased from 3.0 to 9.0. While the removal of Cu increased significantly as the pH increased from 2.0 to 7.0
- ❖ The optimum contact time for adsorption of the heavy metals was considered to be 35 min.
- ❖ The adsorption experimental results of these heavy metals are in a good correspondence with the Langmuir and Freundlich isotherms.
- ❖ The produced metal oxide was found to be potential adsorbent for the removal of Pb and Cu from aqueous solution.

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