The Adsorption Study of Copper Removal by Chitosan-Coated Sludge Derived from Water Treatment Plant

Meng-Wei Wan, Chan-Ching Wang, and Chien-Min Chen

Abstract—The rapid development of science and technology in recent years created a standard of living that caused serious deterioration in the environment. Heavy metal contamination in the soil, surface water and groundwater is one of the major environmental problems due to their non-biodegradability and toxicity. Thus, how to solve efficiently the heavy metal pollution in groundwater has become the most essential issue around the world. Commercialized adsorbents such as activated carbon are effective in removing heavy metals but remain costly. In this study, the removal of copper (II) from aqueous solutions using a biodegradable material, known as chitosan coated sludge (CCS), was investigated under static conditions. The sludge was obtained from a water treatment plant. Moreover, batch experiments were conducted to investigate the adsorption effect under different initial concentration ($C_0 = 100$, $500$, $1000$ and $2000$ mg/L), solution at pH $= 3$ to pH $= 4$ and contact time (0.5, 1, 2, 4, 6, 12 and 24 h). Results indicated that the data fitted well with Langmuir model at $Q_{max}$ value of 18.83 mg/g CCS for Cu(II) ions at two hours contact time. The kinetic data best fit the pseudo-second order equation, indicating that chemisorption is the rate-limiting step of the Cu(II) adsorption.

Index Terms—Adsorption, chitosan, copper, sludge, water treatment plant.

I. INTRODUCTION

Due to dramatic development of industry, heavy metal pollution has become a global environmental consideration. Heavy metals, such as cadmium, chromium, cobalt, copper, nickel and mercury are common water pollutants [1]. These metals exist in aqueous waste from industries, such as mining, metal plating, and electronics [2]. On the other hand, the presence of heavy metal ions in industrial effluents and drinking water resources is also relevant due to their toxicological properties and potential impacts on human health and environment [3]. Thus, efficient solution to heavy metal pollution in groundwater has become the most essential issue around the world.

Copper (Cu), in particular, is a natural element predominantly used in production industry applications. Copper is also a very common substance that occurs intrinsically in the environment and spreads through natural phenomena. It is extensively utilized by electrical industries, in fungicides and in antifouling paints. It is toxic to humans, producing oxidative stress when ingested at high concentrations [4]. Among the ionic species of copper, Cu(II) ions can have alarming effects in aqueous solution, attaching easily to organic and inorganic matter based on solution pH [5].

Application of biopolymers such as chitin and chitosan is one of the emerging adsorption methods for the removal of dyes and heavy metal ions, even at low concentrations [6]. Chitosan, a biopolymer prepared through the deacetylation of chitin, which is the major component of crustaceans shells and the second most abundant biopolymer in nature, has been widely used for adsorption of heavy metal ions [7]–[10]. Its chelating properties are attributed to the amino and hydroxyl groups in chitosan chain, that therefore, can act as chelation sites for different metals [11].

The sludge which is found in the bottom of a sedimentation tank in water treatment plants is primarily composed of water. The solids in the sludge are mainly flocs, excess coagulant, such as alum. Alum sludge has a solids concentration of only about 1% when automatically removed from the basin, or about 2% if manually removed. Many options exist for disposal of sedimentation sludge such as disposal in streams, lagoons and landfills.

Applying permeable reactive barrier for the recovery of metals is a practically environmental remediation technology by building filters along a contaminated stream of groundwater. The remediation of the contaminated waste stream would be costly when chitosan is used alone because construction of filters along the stream requires large quantities of adsorbents. Immobilizing chitosan with a low-cost material results to a lesser amount of chitosan used without affecting the overall metal adsorption capacity [12]. Unfortunately, there are limited references to adsorption studies that provide readily available support for chitosan in spite of its inherent practical advantages.

In this research, the application of an appropriate immobilization material is essential for the utilization of chitosan in industrial applications. Providing a support material (sludge) for the immobilization of chitosan is considered to be both an economical and practical method, which can further enhance its metal binding capacity. The aim of this study is to investigate the removal of heavy metals using chitosan coated sludge (CCS) derived from a water treatment plant. The batch adsorption experimental data were
used to determine the effectiveness of CCS to uptake copper ions individually from solutions.

II. MATERIALS AND METHODOLOGY

A. Chemicals and Reagents

Chitosan (low molecular weight, CAS No. 9012-76-4) was purchased from Sigma–Aldrich; anhydrous CuSO4; HCl, fuming 37%; NaOH; ICP Standard Solution, 1000 mg/L Cu(II) were supplied from Merck Company and Sand (EM Science, Gibbstown, NJ). Deionized (DI) water of milli-Q purity was used for all reagent solutions.

B. Preparation of CCS

Immobilization of the chitosan was similar to the methods found in the literature [12] with only slight modifications. About 5 g of chitosan and 100 g of dry sludge were stirred in 300 mL of 5% (v/v) HCl for 5 h. 1 N NaOH was added drop by drop until neutralization occurred. The adsorbent was allowed to settle, washed with DI water and was oven-dried for 24 h at 65 °C. After drying and grinding, the materials were sieved, passed through Mesh No. 25 (0.700 mm) and collected on Mesh No. 35 (0.500 mm). The homogenous particle size of 0.500 mm which illustrated the best adsorption efficiency based on previous optimization study of adsorbent’s particle size for Cu adsorption was used in the entire experiments. The new adsorbent is called chitosan-coated sludge (CCS).

C. Characteristic Analysis

SEM photographs were taken with a HITACHI S-3000N Scanning Microscope to examine the morphology and surface structure of the adsorbents at the required magnification at room temperature. Thermal gravimetric analyses (TGA) were performed on freeze-dried sludge, chitosan and chitosan-coated sludge (CCS) samples using Rigaku Thermo Plus TG 8120 in the temperature range of 30 – 800°C at a heating rate of 10°C per minute. Surface area of the adsorbents were measured by the single point BET (Brunauer, Emmett and Teller) method using the Accelerated Surface Area and Porosimetry System (Micromeritics, ASAP 2010).

D. Elemental Analysis by ICP-OES

A Perkin Elmer 2000DV Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) was used for quantitative determination of metal ion concentration. Standard solutions for the instrument’s calibration curve were prepared as indicated: 10 mL of 1000 mg/L of ICP standard diluted to 100 mL with DI water which served as stock solution. Subsequently, different volumes of the stock solution (0.05, 0.1, 0.5, 1.0, 2.0, 4.0, 6.0 and 10.0 mL) were pipetted into 100 mL volumetric flasks and diluted to mark.

E. Batch Adsorption Studies

The adsorption of Cu(II) ions were analyzed in a batch system at room temperature with varied concentrations ranging from 100–2000 mg/L.

I) Single metal ions experiment

The experiments were performed using single solutions of the adsorbents maintained at pH 4.5. The solutions were prepared in DI water using the anhydrous CuSO4. Thirty mL of single metal ion solution was placed in 100-mL Erlenmeyer flasks and was then combined with 2.5 g of CCS. The equilibration (shaking) time was 24 h at a static speed of 50 rpm. After equilibrium, the solution was filtered using Whatman #40 filter papers. A Whatman #40 filter was selected as a clean-up filter in preparation for ICPOES analysis. The filtrate was analyzed with ICP-OES. The amount of Cu(II) ions adsorbed per unit mass of CCS was calculated using the equation:

\[ Q_0 = \frac{(C_0 - C_e)V}{m} \]  

where \( C_0 \) and \( C_e \) are the initial and equilibrium concentrations of metal ion solution (mg/L), respectively; \( V \) is the volume of the solution in liter (L); and \( m \) is the amount of CCS in grams (g). The percentage adsorption was determined using the equation:

\[ \text{Percentage adsorption (\%)} = \frac{(C_0 - C_e)100}{C_0} \]  

where \( C_0 \) and \( C_e \) are the initial and equilibrium concentration of metal ion solution (mg/L).

III. EFFECT OF \( C_0 \) AND CONTACT TIME

In this study, the initial concentration and contact time are two main parameters in the adsorption of Cu(II) ions in aqueous solution. This study determined the capacity of CCS for removing Cu(II) ions in the solution at the equilibrium time. The Co concentration were 100, 500, 1000 and 2000 mg/L prepared by diluting the stock solution of Cu(II) ions. The contact times were varied from 0.5, 1, 2, 4, 6, 12 and 24 h.

A. Effect of Solution pH

The Co of 100 mg/L was adjusted to the required pH value using 1 N HCl and 1 N NaOH. CCS (2.5 g) were equilibrated at the desired pH of the specified volume of the adsorbates (30 mL) for 24 h at a static speed of 50 rpm. The final pH of the filtrate was determined and its metal ion content was analyzed using ICP-OES.

B. Kinetics of Adsorption

In order to investigate the controlling mechanism of the adsorption processes, pseudo first-order and pseudo second-order kinetics were applied in the data. This includes the mass transfer and chemical reactions due to the large number and variation of different chemical groups on chitosan chains [13].

1) Pseudo first-order kinetics

The pseudo first-order kinetics can be expressed as:

\[ \log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \]  

where \( k_1 \) is the rate constant of pseudo first-order adsorption.
(min⁻¹); qₑ and qₜ are the amount of metal ion adsorbed per gram of chitosan-coated sludge (mg/g CCS) at equilibrium and at any time t, respectively. A straight line for the plot of log (qₑ - qₜ) versus t would give the first-order rate constant k₁ and equilibrium adsorption capacity qₑ, from the slope and the intercept of the line.

2) Pseudo second-order kinetics

The pseudo second-order kinetics can be expressed as:

\[ t = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \]  

where \( k_2 \) (g/mg min) is the rate constant of pseudo second-order adsorption. The plot of \( t/q_t \) versus t would give the pseudo second-order rate constant \( k_2 \) and \( q_e \). The pseudo second-order kinetic model is based on the assumption that the rate-limiting factor maybe chemisorption involving valence forces through sharing of electrons between the amino group and metal ions [14].

C. Equilibrium Isotherms Studies

Adsorption isotherm models describe the relationship between adsorbates and adsorbents at equilibrium. The two most wellknown isotherm models are the Langmuir and the Freundlich. In this study, equilibrium isotherms were used to determine the adsorption mechanism of CCS for Cu(II) ions. The adsorbents were combined with fixed volumes (30 mL) of metal ion solutions varying the initial concentrations (100, 500, 1000, and 2000 mg/L) at 4 h contact time. The relation between the amount of adsorbed metal and the remaining concentration of metal ions in solution is described by the isotherm studies.

1) Langmuir isotherm model

The Langmuir equation, which is valid for monolayer adsorption onto a surface with a finite number of identical sites, can be expressed as:

\[ Q_o = \frac{Q_{max} k_L C_e}{1 + b C_e} \]  

where \( Q_{max} \) (mg/g) is the maximum amount of the metal ion per unit weight of CCS capable of forming complete monolayer coverage on the surface, bound at a high equilibrium concentrations \( C_e \); \( Q_o \) is the amount of metal ion adsorbed per unit weight of CCS at equilibrium; \( k_L \) and \( b \) are the Langmuir constants, where \( b \) is related to the affinity of binding sites [15]. According to the Langmuir equation, the maximum adsorption capacity was derived from the following equation:

\[ Q_{max} = \frac{k_L}{b} \]  

where \( k_L \) and \( b \) can be determined from the linear plot of \( 1/Q_o \) against \( 1/C_e \).

2) Freundlich isotherm model

Another widely used empirical equation, the Freundlich equation, was based on adsorption on a heterogeneous surface. The equation is represented by:

\[ \ln Q_o = \ln k_f + \frac{1}{n} \ln C_e \]  

where \( k_f \) is roughly an indicator of the adsorption capacity and 1/n of the adsorption intensity. A linear form of the Freundlich equation will yield the constant \( k_f \) and 1/n.

Therefore, \( k_f \) and 1/n can be determined from the linear plot of \( \ln Q_o \) versus \( \ln C_e \). The magnitude of the exponent 1/n gives an indication of the favorability of adsorption. The obtained values of \( n > 1 \) signify favorable adsorption conditions [15].

IV. RESULT AND DISCUSSION

A. Characteristic Analysis

Sludge, chitosan and CCS bioadsorbents were characterized and among these three, the CCS illustrates higher surface area and pore volume when compared to the other two adsorbents. Obviously the metal uptake capacity of this adsorbent is higher due to its free adsorption sites. SEM/EDS analysis was exploited to illustrate the variation of morphology, mineralogical and chemical composition for each adsorbent.

![Fig. 1. SEM pictures of different adsorbents](image_url)

The SEM images of the surface on sludge, chitosan and CCS are shown in Fig. 1. Fig. 1(a) and 1(b) indicates that sludge displays a rough structure on surface with a large surface area while chitosan displays a relative smooth structure on surface with a lesser surface area. Fig. 1(c) confirmed the presence of many pores and some cracks on
the surface of the CCS adsorbent.

B. Batch Adsorption Experiment

1) Effect of Co and contact time

As shown in Fig. 2, it is remarkable that an increase in the initial concentration (Co) for both Cu(II) ions led to an increase in the adsorption capacity of chitosan-coated sludge (CCS) at various contact times. This can be attributed to the mass transfer effects and the driving force of the concentration gradient being directly proportional to the initial concentrations. Fig. 3 demonstrated that the higher adsorption rates were examined at the beginning. The plateau values indicated that the adsorption equilibrium was gradually attained. Retention of Cu(II) ion was noted to be inversely related to Co. At the lowest (100 mg/L) and the lower (100~500 mg/L) concentrations, the equilibrium adsorption capacities were related to the ionic size. At the higher Co (1000~2000 mg/L), the equilibrium adsorption capacities were attributed to the hardness of the metal ions.

Masel (1996) expounded that hardness is inversely related to the ability of the molecule to share electrons [16]. It follows that when the hardness is small, the number of shared electrons will be large and vice versa. Therefore, more numbers of electrons were shared, which resulted in stronger covalent interactions with the chitosan at a higher Co. In this case, the maximum adsorption capacity was achieved after the binding ability of the adsorbent approached saturation at equilibrium time.

As shown in Fig. 3, an exposure time of 24 h was used during batch studies to achieve the equilibrium. It demonstrated that CCS can potentially remove more than 50% of Cu (II) ions at the highest concentration and reached the equilibrium at 1 h regardless of the initial concentrations (Co). Moreover, when adsorbate concentrations were raised, binding capacity of the adsorbent reached saturation instantaneously, which resulted in diminishing the overall % adsorption of Cu ions.

2) Effect of pH

To prevent precipitation, experiments were carried out at pH < 6 to ensure the solubility of metal ions [14], [17], [18]. At high pH, precipitation usually occurred with the metallic ions attached to hydroxide ions forming Cu(OH)2 at pH > 8.

To clarify the influence of solution pH on the adsorption capacity, chitosan-coated sludge (CCS) was subjected to different pH values (pH= 2,3,4) at an oscillating time of 1 hour. The choice of the oscillating time was in accordance to CCS adsorption equilibrium experiments. The results of experiments are shown in Figure 4. The CCS illustrates good adsorption phenomena at pH 4 and pH 3. However, it demonstrated the downward trend as the pH value decline.

The CCS adsorption capacity is influenced significantly by the pH value. The chitosan attached to the water treatment plant sludge provides larger contact surface resulting to an increase in adsorption capacity. However, in acidic condition, there is competition between H+ ions and the heavy metals in terms of binding with the basic NH2 groups of chitosan. Metal adsorption percentage decreased since most of the NH2 groups of CCS are in the protonated form (NH3+) [19].

Taboada et al., (2003) and Wan et al. (2010) described the similar phenomena as the following competing reactions, where M2+ represents the divalent metal ion and R is the polymer structure of chitosan [19], [20].

\[
\text{M}^{2+} + \text{RNH}_2 \rightarrow \text{M} (\text{RNH}_2)^2+
\]

The amino group of chitosan may react with hydrogen ions (H+) according to:

\[
\text{H}^+ + \text{RNH}_2 \rightarrow \text{H} (\text{RNH}_2)^+
\]

Conversely, there was an improvement in the metal adsorption percentage while the pH value was increased. CCS exhibited enough binding capacity. The adsorption percentage of different Cu (II) concentrations increased with
pH range from 2 to 4. However, the influence on retention time was minimal for the succeeding values (pH 3-4) regardless of contact time. This effect suggested that the amino group was deprotonated. The chelation mechanism outranked the adsorption one where more metal ions chelating with chitosan.

C. Kinetics of Adsorption

To investigate the controlling mechanism of the adsorption processes, pseudo first-order and pseudo second-order kinetic equations were both examined. Table I shows the adsorption kinetic studies of Cu(II) ions onto CCS at different C0. The pseudo first-order kinetic model did not adequately describe the adsorption results with a low correlation coefficient for the entire data (0.1855–0.6038). However, the pseudo second-order kinetic model provided an impressive and comparable correlation for the adsorption of both ions in contrast to the pseudo first-order model (0.9977–0.9998).

The pseudo first-order rate model has been widely used for sorption of metals [14], which was widely used for reversible reactions with an equilibrium being established between liquid and solid phases. In many cases, the pseudo first order do not fit well to the whole range of contact time and is generally applicable over the initial stage of adsorption process [21].

<table>
<thead>
<tr>
<th>Cu(II) (C0, ppm)</th>
<th>Pseudo-first order kinetic model</th>
<th>Pseudo-second order kinetic model</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>k1</td>
<td>qe</td>
</tr>
<tr>
<td>100</td>
<td>0.00005</td>
<td>2.08</td>
</tr>
<tr>
<td>500</td>
<td>0.00005</td>
<td>6.95</td>
</tr>
<tr>
<td>1000</td>
<td>0.00016</td>
<td>11.81</td>
</tr>
<tr>
<td>2000</td>
<td>0.00023</td>
<td>16.81</td>
</tr>
</tbody>
</table>

Ho (2006) illustrated that the pseudo second-order reaction was used to distinguish the kinetic equation based on the concentration of a solution from the adsorption capacity of a solid [22]. In this study, the rate constant k2 was determined experimentally by plotting the slopes and intercepts of t/qt against t. The high correlation coefficient shown in Table I illustrated an agreement of experimental data with the pseudo second-order kinetic model (Eq. (4)) for different C0. The pseudo first-order kinetic model did not adequately describe the adsorption results with a low correlation coefficient for the entire data (0.1855–0.6038). However, the pseudo second-order kinetic model provided an impressive and comparable correlation for the adsorption of both ions in contrast to the pseudo first-order model (0.9977–0.9998).

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demonstrated high efficiency in removing Cu(II) ions from inexpensive large-scale filter as a permeable reactive barrier sludge derived from water treatment plant to establish an these preliminary results indicate the possibility of using CCS to remove copper ions from water. In conclusion, chemisorption.

indicated that the adsorption was controlled by pseudo second-order kinetics, which agreed well with pseudo second-order kinetics, which indicated that the adsorption was controlled by chemisorption. This study has illustrated the possible adsorption behavior of CCS to remove copper ions from water. In conclusion, these preliminary results indicate the possibility of using sludge derived from water treatment plant to establish an inexpensive large-scale filter as a permeable reactive barrier for metal removal, the goal of waste recycle and reuse.

V. CONCLUSION

Biosorbent, chitosan-coated sludge (CCS), has demonstrated high efficiency in removing Cu(II) ions from aqueous solution. The amount of Cu(II) ions adsorbed increased with increasing pH. The equilibrium studies were described using both Langmuir and Freundlich isotherm models. The Langmuir isotherm illustrated the best description of the metal adsorption mechanism. The maximum adsorption capacity of Cu (II) ions was 18.83 mg/g after 2 hours contact time. Moreover, the equilibrium kinetics agreed well with pseudo second-order kinetics, which indicated that the adsorption was controlled by chemisorption.

This study has illustrated the possible adsorption behavior of CCS to remove copper ions from water. In conclusion, these preliminary results indicate the possibility of using sludge derived from water treatment plant to establish an inexpensive large-scale filter as a permeable reactive barrier for metal removal, the goal of waste recycle and reuse.

APPENDIX

<table>
<thead>
<tr>
<th>Contact time (hrs)</th>
<th>Langmuir isotherm</th>
<th>Freundlich isotherm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>K_L B Q_max (mg/g)</td>
<td>R^2 n K_f R^2</td>
</tr>
<tr>
<td>0.5</td>
<td>0.29 0.025 11.88 0.9957 2.08 0.6636 0.9846</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.49 0.039 12.56 0.9964 2.14 0.8972 0.9776</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.58 0.031 18.83 0.9994 2.06 1.1293 0.9379</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.75 0.045 16.50 0.9992 2.23 1.3110 0.9325</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1.10 0.066 16.61 0.9987 2.14 1.5025 0.9512</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>1.58 0.091 17.45 0.9983 2.15 1.9099 0.9265</td>
<td></td>
</tr>
<tr>
<td>24</td>
<td>2.32 0.132 17.54 0.9928 1.92 2.1498 0.9470</td>
<td></td>
</tr>
</tbody>
</table>


REFERENCES


Meng-Wei Wan was born at Tainan City, Taiwan in 1974. He received his B.S. degree in Environmental Science, Tunghai University, Taichung, Taiwan from 1992 – 1996. He received his M.S. degree from department of Civil and Environmental Engineering, University of Southern California, USA from 1999 – 2001. Finally, He received his Ph.D. from department of Civil and Environmental Engineering, University of Southern California, USA from 1999 – 2001.

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Dr. Wan awarded total 2 gold medals and 2 silver medals at the 2012 64th and 2011 63th IENA Nuremberg - International Trade Fair Ideas-Inventions - New Products, Nürnberg, Germany. He also awarded total 2 gold medals, 1 silver medal and 1 bronze medal at the 2011 and 2012 Taipei International Invention Show & Technomart, Taipei, Taiwan.

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