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# Evaluation of copper adsorption from industrial wastewater using sewage sludge ash

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*ORIGINAL RESEARCH ARTICLE*

## ABSTRACT

The objective of this research work is to highlight the importance of using waste material such as sewage sludge, as a starting material for adsorption of heavy metals. Sludge incineration can help to stabilize and reduce the volume of sewage sludge; and the resultant ash (SSA) has been considered as an adsorbent for the removal of heavy metals from industrial waste water. In batch process, the effect of environmental condition such as adsorbate concentration (12.5, 25, 50 and 100 mg/L) was studied to evaluate the removal efficiency of SSA. Also, the effects of weight ratio of SSA (0.1, 0.5, 1 and 3 mg/100 mL), agitation speed (60, 150, 200 and 250 rpm), mesh size of adsorbent (60, 65 and 90 µm), temperature (40, 50 and 60 °C) and contact time (10, 20, 30, 60, 110 and 180 min), on the removal of copper ions were investigated. The optimized experimental conditions were used for the removal of copper ions from wastewater obtained from El-Sewedy cables industry. Experimental investigations indicated that at low solute concentration of 25 mg/L and high agitation speed of 250 rpm, the removal efficiency of copper ions by SSA reached 98% with the SSA dosage equal or higher than 3 g/100 mL. Adsorption isotherm studies indicated that Langmuir model described the experimental data with $R^2$ values greater than 0.994. The study highlighted the ability of SSA for the removal of copper ions from industrial wastewater.

**KEYWORDS**

adsorption; copper ions; industrial wastewater; Langmuir model; SSA

## 1. INTRODUCTION

The production of sewage sludge (SS) from wastewater treatment is estimated to reach 2 million tons dry solids/year by 2020 (Hassan, 2015). This may cause pollution problem if it is not well managed by incineration, land filling, road surfacing or used as fertilizer (Yu and Zhong, 2006; Mattenberger et al., 2008). On the other hand, waste reuse may minimize the environmental problems associated with their build-up and reduce the use of noble starting materials (Karnitz et al., 2007).

Sludge handling represents a bottleneck in wastewater treatment plants due to the unstable nature of biomass. In order to achieve acceptable volume-reduction and stabilization of sewage sludge, many sludge incineration facilities were built and operated. The residue of sewage sludge produced from incineration facilities is the sewage sludge ash (SSA), which is primarily an inorganic and stable material comprises mainly of silicon oxide and aluminum oxide (Xu et al., 2015). Among the feasible reuse technologies, SSA was mostly used in building materials (Pan et al., 2002) or fine aggregates (Chang et al., 2007) in cement concrete, used as a raw material in brick manufacturing, in cement manufacturing, and in ceramic industries (Garcés et al., 2008; Jamshidi et al., 2012).

In addition to the silicon and aluminum-rich characteristics, the SSA also exhibits significant specific surface area and cation-exchange capacity (Pan et al., 2003). Also, SSA showed capability to adsorb and remove heavy metals from wastewater (Zare et

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al., 2015). Copper is one of the most important and frequently used metals in industries such as plating, mining and petroleum refining. These industries produce large amount of wastewater containing a high concentration of copper ions, which have toxicity effects on humans and other forms of life. The excessive intake of copper accumulate in the liver and produces gastrointestinal problems, and continued inhalation of copper-containing sprays is linked with an increase in lung cancer among exposed workers (Grassi et al. 2012; Andreazza et al., 2013). Removal of heavy metals from contaminated aquatic systems is deemed important for the protection of environmental health. Many technologies have been developed to remove heavy metals from contaminated waters before discharging in order to meet the wastewater standards according to EPA regulations for industrial effluents pollution of surface waters (Eckenfelder et al., 2008). These treatment technologies include chemical precipitation, ion exchange, membrane separation and biological treatment. However, these methods are relatively expensive due to costly equipments or elaborate procedures. In contrary, adsorption techniques are most effective and well established methods for removal of metal ions.

The present study aims to investigate the feasibility of using sewage sludge ash (SSA) as an adsorbent for the removal of copper ions from wastewater and to assess the influence of different parameter such as contact time, ash concentration and agitation speed on copper adsorption by SSA as well as the adsorptive characteristics of SSA including specific surface area and chemical composition. Finally, adsorption isotherms and mechanisms of SSA for copper ions removal were evaluated.

## 2. MATERIALS AND METHODS

### 2.1. Materials and equipment

The sewage sludge waste sample used in this study was collected from EL-Berka municipal wastewater treatment plant in the South of Egypt. The sampled dewatered sludge cake was first burned in a modular incinerator at 600 °C for 2 h to produce incineration ash and then grounded. The grounded SSA was sieved using 75 μm sieve and subsequently used in the experiments in Housing and Building Research Center (HBRC).

Scanning electron microscope (SEM) (Philips XL30) and energy dispersive X-ray analysis (EDAX) (Philips EDAX9100) were used to characterize the ash samples for chemical constituents and morphology. The surface area of ash sample was determined by a BET-$N_2$ surface area analyzer (Model QS-7, Quantachrome Co., Greenvale, NY).

### 2.2. Preparation of copper solutions

#### 2.2.1. Synthetic copper ions

Synthetic copper solutions were prepared from copper salt ($CuSO_4.5H_2O$) at different concentrations. All chemicals used for the treatment of the different sorbents and for adsorption tests were of analytical grades. A pH meter (model pH 540 GLP) equipped with a combined glass electrode SENTIX 41 was used to measure the pH of the solutions. A preliminary calibration is systematically carried out using suitable buffer solutions. The concentrations of copper ions were measured by an atomic absorption spectrometer (ASS).

#### 2.2.2. Industrial wastewater

Industrial wastewater sample were collected from effluent of El-Sewady Cables Company for electrical products, 10th Ramadan city in Upper Egypt. Table 3 shows physico-chemical analysis of industrial wastewater sample.

### 2.3. Batch experiments

Batch experiments were performed to study adsorption capacity of SSA towards copper ions. A series of bottles containing ash slurries at different weight ratio from 1 to 30 g/L and different copper concentrations ranging from 12.5 to 100 mg/L were agitated at speed ranging from 60 to 250 rpm at different temperatures ranging from 27 to 60 °C for a time ranging from 10 to 180 min. The pH of solution was adjusted to 5.5 using 0.5 N HCl and 0.5 N NaOH solutions before the contact of ash with copper ion solution. The sample from each bottle was collected and analyzed for residual copper using atomic absorption spectrometer (Model ICE 3000 Series - Thermo Scientific), with air acetylene flame at wave length of 324.8 nm.

Adsorption isotherm studies were conducted by adding 3 g of ash sample to a 100 mL solution containing various copper ion concentrations (12.5, 25, 50 and 100 mg/L). The initial pH was maintained at 5.5 and the suspensions were stirred for 1 h. After filtration through a 0.42 μm membrane filter (MFS), the filtrates were analyzed by AAS. In order to determine the effect of temperature, the adsorption isotherms were established at 27, 40, 60 °C. The percentage removal of copper ions was calculated using the equation,

$$\% \text{ Removal} = (C_o - C_t)/C_o \qquad (1)$$

where $C_o$ and $C_t$ are initial and final concentrations of copper ions (mg/L), respectively.

### *2.4. Adsorption Isotherm Models*

Adsorption isotherms are important to describe the adsorption mechanism of a solute on adsorbent surface thus aid in optimizing the design of a specific adsorption process. In the present study, the equilibrium data obtained for copper removal using SSA was tested with two isotherm models available in the literature to reveal the best fitting isotherm. Adopted isotherm models were the Langmuir and Freundlich. Isotherm coefficients and correlation coefficients ($R^2$) were computed from linearized equations of these isotherms in Microsoft Excel.

#### *2.4.1. Langmuir isotherm*

Langmuir isotherm has been extensively used to describe adsorption of heavy metals, dyes, organic pollutants, etc. (Yang and Al-Duri 2005; Garg et al. 2007). It is applicable for monomolecular layer adsorption. This isotherm is described as a homogeneous one assuming that all the adsorption sites have equal adsorbate affinity and that the adsorption at one site does not affect the adsorption at an adjacent site (Zare et al., 2015). The Langmuir isotherm is used to obtain a maximum adsorption capacity produced from the complete monolayer coverage of adsorbent surface. The linear isotherm equation is represented as:

$$C_e/q_e = 1/bq_{max} + (1/q_{max})\, C_e \qquad (2)$$

Thus, a plot of $C_e/q_e$ versus $C_e$ should yield a straight line if Langmuir isotherm is obeyed by the adsorption equilibrium. The qmax and b values can be calculated from the slope and intercept of the plot, respectively. The constant b is the adsorption equilibrium constant in L/mg related to the apparent energy of adsorption and $q_{max}$ is the maximum quantity of adsorbate required to form a single monolayer on unit mass of adsorbent in mg/g. A further analysis of the Langmuir equation can be made using a dimensionless equilibrium parameter, the separation factor $R_L$ as given by Eqn. (3):

$$R_L = 1/\,(1 + b\, C_o) \qquad (3)$$

For a favorable adsorption, the value of $R_L$ should lie between 0 and 1; $R_L > 1$ represents an unfavorable adsorption, $R_L = 1$ represents linear adsorption, whereas $R_L = 0$ translates into irreversible adsorption (Gupta and Babu, 2009).

#### *2.4.2. Freundlich isotherm*

The Freundlich expression is an exponential equation and therefore assumes that, the concentration of adsorbate on the adsorbent surface increases with the adsorbate concentration. Theoretically, using this expression, an infinite amount of adsorption can occur (Freundlich, 1907). The equation is widely applied in heterogeneous surfaces with sites that have different energies of adsorption and are not equally available. The Freundlich isotherm is more widely used but provides no information on the monolayer adsorption capacity in contrast to the Langmuir model and can be written in linearized form as:

$$\log (q_e) = \log K_f + 1/n \log (C_e) \qquad (4)$$

where $K_f$ is the experimental constant (Freundlich constant indicative of the adsorption capacity of the adsorbent (L/mg) and n is the exponential constant indicative of the adsorption intensity of the adsorbent. By plotting log qe versus log ($C_e$), values of $K_f$ and n can be determined from the slope and intercept of the plot. The magnitude of the exponent 1/n gives an indication of the favorability of adsorption. Values of n, where $n > 1$ represent favorable adsorption condition. In general, as the $K_f$ value increases, the adsorption capacity of the adsorbent, for the given adsorbate, increases. The values of n between 1 and 10 indicate beneficial adsorption. If n is close to 1, the surface heterogeneity could be assumed to be less significant and as n approaches 10 the impact of surface heterogeneity becomes more significant (Dávila et al., 2005; Gupta, 2009).

## 3. RESULTS AND DISCUSSION

### *3.1. Physicochemical characterization of ash*

As determined by Energy Dispersive Analysis of X-rays (EDAX), the predominant chemical composition of SSA is given in Table 1. The results indicated that SSA contains silicon oxide, aluminum oxide, calcite as well as other oxides in trace amounts. It is thus expected that copper ions may be mostly adsorbed either by silica, alumina or by a combined influence of these oxides.

**Table 1.** Chemical composition of sewage sludge ash.

| Element primary composition, % | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | Cao | $P_2O_5$ | MgO | $SO_3$ | $K_2O$ | $Na_2O$ | ZnO | $TiO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 33.83 | 5.4 | 9.56 | 18.4 | 5.65 | 2.08 | 8.01 | 1.63 | 0.62 | 0.5 | 0.95 |

X-ray diffraction analyses of sewage sludge before and after heating for 2 h at 600 °C are presented in Figure 1. It can be seen that an increase in temperature increases the level of mineralization since more peaks appear. In the case of materials obtained at 600 °C, the degree of mineralization was much lower. The samples of sewage sludge dried at 105 °C are predominantly amorphous. Combustion at 600 °C results in formation of aluminosilicates, calcite and the oxides of aluminum, iron magnesium, sodium and potassium (Seredych and Bandosz, 2006).

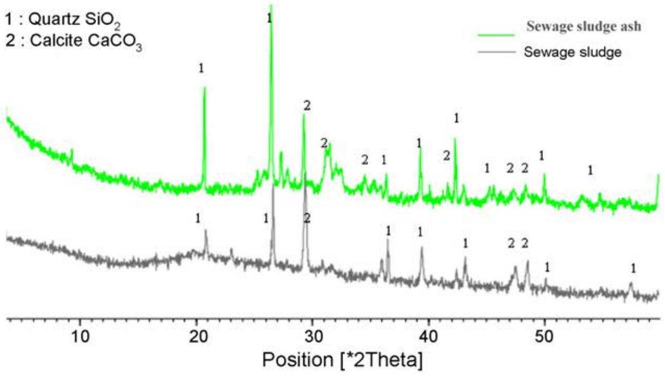

**Figure 1.** X –Ray Diffraction of sewage sludge and sewage sludge ash.

The Brunauer, Emmett and Teller (BET) surface area of SSA was found to be 31.6 $m^2/g$. These results revealed that the SSA was porous material with significant pore surface area.

Figure 2 shows Scanning Electron Microscope (SEM) images of the sewage sludge before and after combustion at 600 °C. It can be seen that the surface morphologies of the raw and calcined samples were different. SEM observation of samples heated at 600 °C indicates the common feature of porous and irregular morphology of the ash resulting in a large surface area.

### *3.2. Effect of adsorbate concentration on percentage removal of copper ions*

The effect of initial adsorbate concentration on copper adsorption was investigated by varying the initial concentration of copper between 12.5 and 100 mg/L by adjusting pH to 5.5, weight ratio of SSA to 3 g/100 mL, and agitation speed at 200 rpm with different time intervals 10, 20, 30, 60, 110 and 180 min. The experimental data are presented in Figure 3. The results showed that SSA adsorption has increased with the decrease in initial copper concentration. The results also showed that the removal efficiency increased with increase in contact time. For instance, the removal efficiency of SSA increased from 95.4 to 99% on increasing contact time from 10 to 180 min. At copper concentration of 100 mg/L, removal efficiency reached 30% and 72% at 10 and 180 min, respectively. This indicates that the adsorption rate increases with the increase in contact time. This increase may result from an increase in electrostatic interactions between surface oxides such as $SiO_2$, $CaCO_3$ which carry negative charge as illustrated by X-ray diffraction in Figure 1 and positive charge of copper ions (Aydin et al., 2008).

### *3.3. Effect of ash concentration on percentage removal of copper ions*

The effect of ash concentration was studied by varying the dose between 0.1 and 3 g/100 mL. It was observed that the adsorption percentage of copper ions onto the SSA increased rapidly with the increase in adsorbent concentration (Figure 4). This result is expected because the increase of adsorbent dose leads to greater surface area. The removal efficiency was recorded as 3.4% for 0.1 g/100 mL, 9.8% for 0.5 mg/100 mL, 17.2% for 1 g/100 mL and 57% at 3 g/100 mL after 10 min at 200 rpm. This indicates that the removal percentage of copper increase with increase in weight ratio of SSA and the best result was obtained at 3 g/100 mL. Figure 4 also showed that increase in contact time increased the percent removal of copper to reach 57% and 92% at the end of 10 and 180 min, respectively, at SSA dose of 3 g/100 mL.

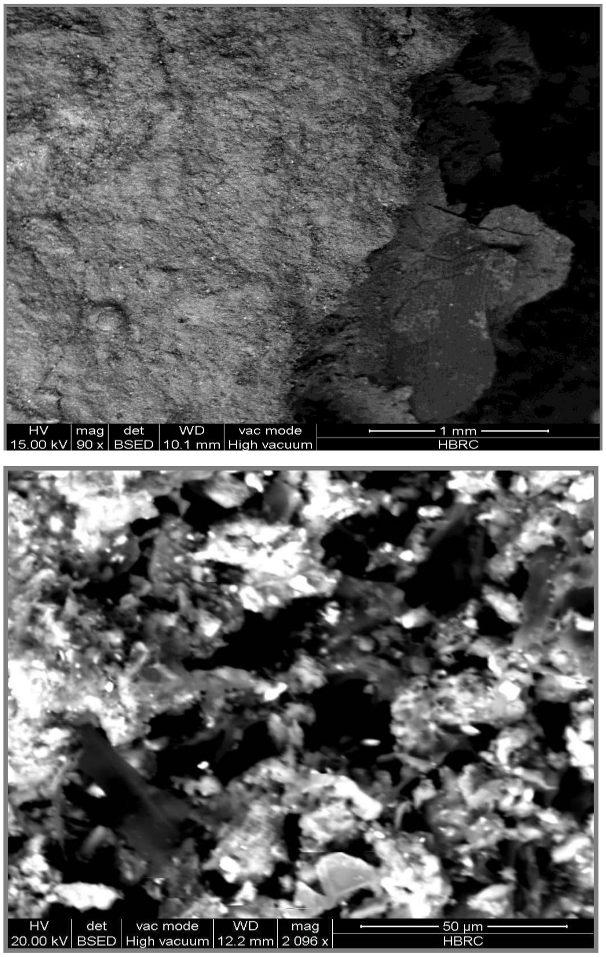

**Figure 2.** Scanning electron microscope of sewage sludge (top image) and sewage sludge ash (bottom image).

### *3.4. Effect of agitation speed on the percentage removal of copper ions*

The effect of agitation speed on the rate of uptake of copper ions was studied using the optimum SSA dosage of 3 g/100 mL, SSA size of 75 µm, adsorbate concentration of 50 mg/L, temperature of 27±2 °C and equilibrium pH of 5.5 by varying the agitation speed at 60, 150, 200 and 250 rpm at different time intervals 10, 20, 30, 60, 110 and 180 min. As shown in Figure 5, the adsorption percentage of copper ions onto the SSA increased with the increase of agitation speed reaching from 39.6 to 75.8% at 60-250 rpm, respectively after 10 min.The results illustrated that the rate of removal of copper ions has increased by increasing the agitation speed. This is due to disperse of adsorbent particles in aqueous solution which lead to reduce the boundary of mass transfer resistance and even it may increase the velocity of particles occurred easily and quickly at agitation speed of 250 rpm, the copper removal increase with time to reach maximum adsorption rate of copper ions of 75.8 and 98% at 10 min and 180 min, respectively.

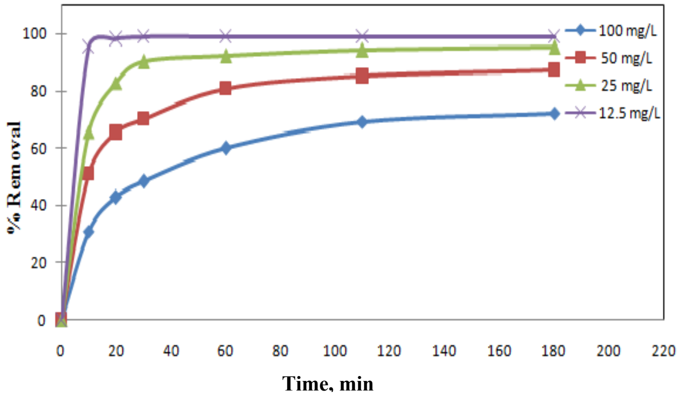

**Figure 3.** Effect of initial copper concentration on the % removal of copper using SSA, pH at 5.5, weight ratio of SSA 3 g/100 mL with 75 µm, temperature ± 27 °C, agitation speed 200 rpm.

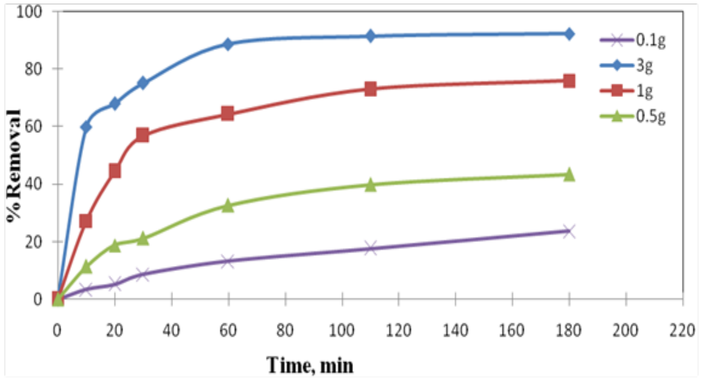

**Figure 4.** Effect of weight ratio of SSA on %removal of copper ions at mesh size of 75 µm, concentration of copper at 50 mg/L, temperature of 27 °C, stirring speed of 200 rpm and pH of 5.5.

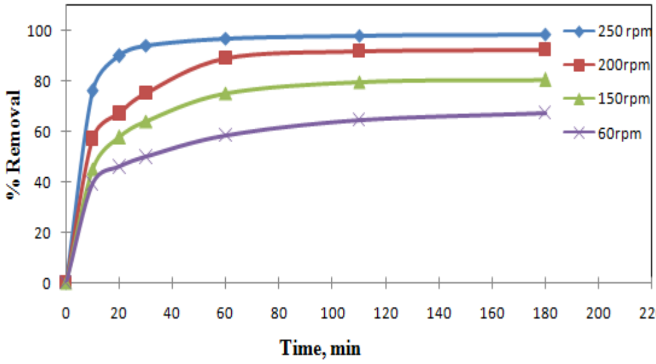

**Figure 5.** Effect of agitation speed on % removal of copper ions using SSA at weight ratio 3 g/100 mL with 75 µm sieve size, concentration of copper at 50 mg/L, pH at 5.5 and temperature of 27±2 °C.

### *3.5. Effect of temperature on the percentage removal of copper ions*

The effect of different temperatures of 40 °C, 50 °C and 60 °C on the removal of copper ions was evaluated at optimum pH of 5.5, weight ratio of SSA 3 g/100 mL, adsorbate concentration of 50 mg/L and agitation speed of 200 rpm at different time intervals 10, 20, 30, 60, 110 and 180 min. From Figure 6, it can be observed that the percentage removal of copper increase from 60 to 90% when temperature increase from 40 to 60 °C. This suggests that the adsorption is spontaneous and endothermic, as previously mentioned in other report (Varma et al., 2013), indicating that increasing temperature increases the interactions with surface silica sites on SSA. From Figure 6, it may be observed that removal of copper ions increase with time from 60 to 90 % at 40 °C, 71 to 94% at 50°C and 80 to 98% at 60 °C on increasing the contact time from 10 min to 180 min. The highest removal efficiency of copper ions was observed at temperature of 60 °C at 180 min.

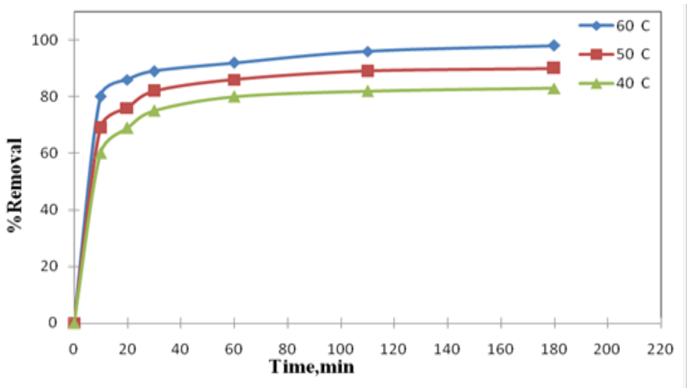

**Figure 6.** Effect of temperature on % removal of copper ions using weight ratio of SSA 3 g/100 mL with 75 µm sieve size, pH at 5.5, concentration of copper at 50 mg/L and agitation speed 200 rpm.

### *3.6. Effect of mesh size of SSA on the percentage removal of copper ions*

The effect of different mesh size of SSA such as 60, 65 and 90 µm on the removal of copper ions was studied by adjusting pH at 5.5, weight ratio of ash at 3 g/100 mL, adsorbate concentration at 50 mg/L, temperature at 27 °C and agitation speed at 200 rpm with different time intervals of 10, 20, 30, 60, 110 and 180 min. Results are illustrated in Figure 7, from which it was clear that the removal efficiency decreased from 75 to 40.1% after 10 min, on increasing the mesh size from 60 to 90 µm. This is because decreasing mesh size of SSA increases the surface negative charges, which in turn increase the electrostatic potential near to solid surface and increase the adsorption capacity. The percentage removal of copper ions increases with time and the rate of adsorption of copper ions reached a maximum value 94.78% for 60 µm SSA after 180 min.

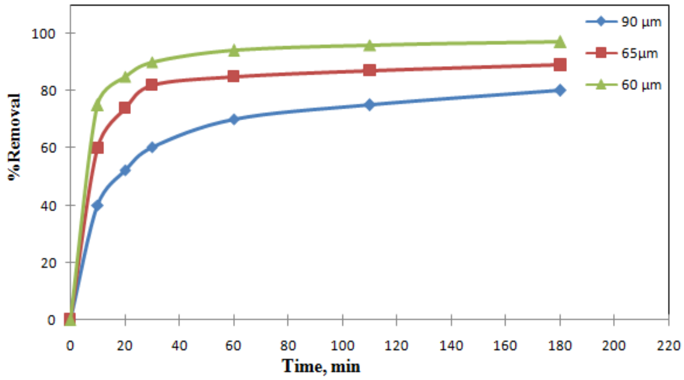

**Figure 7.** Effect of mesh size of SSA on the % removal of copper ions using SSA, pH at 5.5, weight ratio of SSA 3 g/100 mL, concentration of copper at 50 mg/L, temperature at 27± 2 °C and agitation speed of 200 rpm.

### *3.7. Adsorption isotherm*

Adsorption isotherm for SSA was evaluated by contacting 100 mL test solution containing different concentration of copper ions ranged from 12.5 to 100 mg/L at pH 5.5 with 3 g of SSA. The adsorption constants and correlation coefficients of the Langmuir and Freundlich isotherms are presented in Table 2. The Langmuir plot ($C_e/q_e$ and $C_e$) are shown in Figure 8. The Langmuir constant qm, which is a measure of the monolayer adsorption capacity of SSA, was calculated as 2.56 mg/g. The Langmuir constant, b which denotes adsorption energy, was found to be 0.4992 L/mg. The high value of coefficient of determination $R^2$ = 0.994 obtained for the present system indicates a good agreement between the experimental values and isotherm parameters. The Freundlich constants, $K_f$ and n were obtained by plotting the graph between log qe versus log $C_e$ as shown in Figure 9. The intensity of adsorption, as indicated by n value was found to be 2.97 for SSA and adsorption capacity as indicated by the value of $K_f$ was determined as 0.824 L/mg. The application of present isotherm data to the Freundlich model resulted in relatively lower correlation coefficient of 0.977.

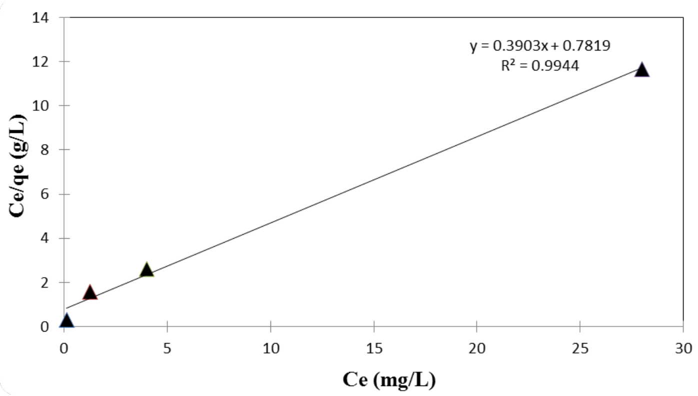

**Figure 8.** Langmuir plot for copper adsorption onto sewage sludge ash.

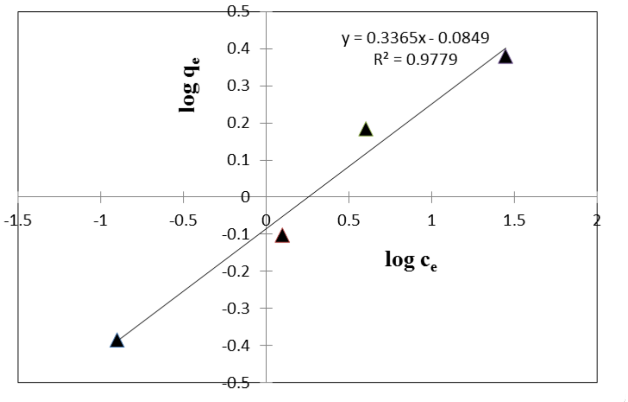

**Figure 9.** Freundlich plot copper adsorption onto sewage sludge ash.

**Table 2.** Isotherm model parameters obtained for removal of copper ions using sewage sludge ash.

| Waste | Langmuir parameters | | | Freundlich parameters | | |
|---|---|---|---|---|---|---|
| SSA | $q_{max}$ (mg/g) | b (L/mg) | $R^2$ | $K_f$ | n | $R^2$ |
| | 2.56 | 0.4992 | 0.994 | 0.824 | 2.97 | 0.977 |

### *3.8. Effect of SSA on physicochemical characterization of wastewater*

The effect of SSA on the removal of copper from industrial wastewater collected from El-Sewedy cables industry was evaluated. The characteristics of the wastewater (COD, $BOD_5$, ammonia, sulphate, nitrate and turbidity) was measured according to Standard Methods for the Examination of Water and Wastewater (2005) and it is tabulated in Table 3. The results show that the removal efficiency of copper ions by SSA reached 98% along with 77.06% reduction of COD and 65% of $BOD_5$. In addition, removal efficiencies of 71% for ammonia, 89.7% for sulphate, 100% for nitrate and 97% for turbidity were also achieved using SSA at 3 g/100 mL. The color of wastewater obtained from El-Sewedy cables industries was turbid white but after adsorption by SSA the effluent become colorless and clear. As illustrated in Figure 10, SSA was an effectively active agent to remove color from industrial wastewater (as shown in Figure 2b) due to porosity and high surface area of SSA after incineration (Weng and Pan, 2006; Hu and Hu, 2014).

**Table 2.** Physicochemical analysis of El-Sewedy company before and after treatment with SSA.

| Parameter | Influent before adsorption | Influent after adsorption | % Removal |
|---|---|---|---|
| pH | 8.6 | 6.02 | ---- |
| Copper, mg/L | 5.6 | 0.112 | 98 |
| COD ,mg/L | 1851 | 424.6 | 77.06 |
| $BOD_5$, mg/L | 534 | 183 | 65 |
| Ammonia, mg/L | 13.5 | 3.8 | 71 |
| Sulphate, mg/L | 15.6 | 1.6 | 89.7 |
| Nitrate, mg/L | 1 | 0 | 100 |
| Turbidity, NTU | 127.8 | 3.5 | 97 |

**Figure 10.** Images of industrial wastewater from El-Sewedy cables industries (a) before adsorption and (b) after adsorption of copper ions.

## 4. CONCLUSIONS

It may be concluded from the present research that SSA is an effective adsorbent for the removal of copper ions from aqueous solution as well as from industrial wastewater. The adsorption process was strongly affected by parameters such as weight ratio (ash concentration) of SSA, adsorbate concentration, agitation speed, mesh size of SSA and temperature. Based on the percentage removal of copper ions for a time ranging from 10 to 180 min, adsorption was achieved in about 60 min. The Langmuir model provided best fit to SSA adsorption isotherm, with correlation coefficient of 0.994. The maximum monolayer coverage capacity from Langmuir isotherm model was determined to be 2.56 mg/g. This study also demonstrated that the SSA could be used as an effective adsorbent for the treatment of industrial wastewater containing copper ions. In addition, SSA is a low cost natural abundant biomass and it may be alternative to more costly materials such as activated carbon.

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