



## A Facile Route for Removal of Reactive Dye Yellow 145 and Cu (II) by Using Bentonite/slag based Geopolymer

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### Abstract

The treatment of pollution (heavy metals and dyes) has drawn a lot of interest due to the serious effects of the accumulation of heavy metal ions in organisms and toxic effect of dyes. For the treatment of pollution, geopolymer materials have been suggested as suitable adsorbents because, compared to conventional treatment procedures, they are more widely produced, convenient, affordable, and environmentally friendly. In this paper, Bentonite/slag based geopolymer adsorbents were made by mixing different amounts of slag with bentonite to remove Reactive yellow 145 dye and copper ions ( $\text{Cu}^{2+}$ ). On the adsorption characteristics of the bentonite/slag based geopolymers, the effect of slag addition, adsorbent dose, pH value, initial mass concentration and contact duration were evaluated.

**Keywords:** bentonite/slag based geopolymer, Adsorption, Reactive dye, Heavy metal.

### 1. Introduction

The output of industrial wastes has increased since the industrial revolution of the 18th century. Metal ions, anionic metal complexes, herbicides, antibiotics, and dyes are a few examples of drinking water contaminants that are harmful to both human health and the environment [1]. There are numerous threats of contamination of the drinking water, however industrial waste is one of the most worrisome and growing dangers in our days. Synthetic dyes and heavy metal are just examples of these dangerous contaminants [2]. Synthetic dyes have great importance in textile industry, paper, cosmetic, food, printing, pharmaceutical industries and laboratories [3]. When textile factory effluent enters water sources, it can lead to several illnesses, including high blood pressure, respiratory problems, allergic reactions, etc. Moreover, dyes are long-lasting in the environment and absorb and reflect sunlight, they kill plants and aquatic organisms, which has a negative impact on the food chain [4]. Dye stability against oxidation or hydrolysis will

determine whether it can produce dangerous byproducts in wastewater [5]. Hazardous heavy metal ion pollution of the world's water supplies has increased as a result of the rapid development of technology and industry [6,7]. Due to the ability of heavy metal ions to bind to proteins, nucleic acids, and small metabolites in living organisms leading to serious health issues in both people and animals [8]. There are several different methods for removing heavy metals and dyes from wastewater, including chemical, biological, and physical ones [9,10]. Since it is easy to use, inexpensive, and highly effective, the adsorption method is frequently used in the treatment of wastewater [11]. Many different substances, including clays, activated carbon, charcoal, resins, zeolites, etc., are frequently utilized as adsorbents to remove industrial pollutants. The study of the adsorption characteristics of synthetically made highly porous frame work materials [12] and geopolymers has become increasingly popular recently [13].

Geopolymers sorption properties are correlated with both their mesoporosity and cation exchange capacity. Alkali cations balance negatively charged

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sites created by an exchange of silicon for aluminum in the network and are now open to transport within the geopolymer structure [14]. Geopolymers are inorganic polymers produced by alkali activating aluminosilicate minerals. In the synthesis of geopolymers, potassium hydroxide and sodium hydroxide, with sodium and potassium silicate, are often employed alkali activators [15]. The aluminosilicate materials are dissolved by strong alkali solutions, releasing free tetrahedral  $\text{AlO}_4$  and  $\text{SiO}_4$ . After the water has evaporated, the tetrahedral groups of  $\text{SiO}_4$  and  $\text{AlO}_4$  link together alternately to form polymorphic precursors ( $-\text{SiO}_4-\text{AlO}_4-$ ,  $\text{SiO}_4-\text{AlO}_4-$ , or  $-\text{SiO}_4-\text{AlO}_4-\text{SiO}_4-\text{SiO}_4-$ ). An amorphous semi-crystalline geopolymer is created by sharing all the oxygen atoms between the two tetrahedral where the negative charge on aluminum is balanced by cations from alkali metals.[16]. Aluminosilicate minerals are divided to by-product such as cement kiln dust, granulated blast furnace slag, fly ash and natural product including metakaolin, bentonite.

Bentonite is an aluminum phyllosilicate clay formed by weathering of volcanic ash [17]. Bentonite has been employed extensively in the field of geopolymers due to its small particle size, high specific surface area, and strong dispersibility. [18]. The chemical composition and mineralogy have a significant impact on the properties of bentonite clays. There are two different categories for bentonite clays namely calcium and sodium bentonite. Calcium bentonites are the non-swelling form while sodium bentonites are the swelling type. The calcium form of bentonite is a widely distributed around the world. [19]. Bentonite is considered as an appropriate and good adsorbent. Due to the abundance of active sites on the outside surfaces as well as in the spaces between the sheets. In addition to larger surface area and higher adsorption capacity of it [20]. Ying Yang et al., [21] prepared porous geopolymers (PGs) by adding a pre-swelled bentonite into a geopolymer paste. The pore volume increased from 0.035 mL/g to 0.626 mL/g and from 0.175 mL/g to 0.708 mL/g, respectively, when the bentonite slurry content increased from 0% to 60% at a water-to-powder ratio of 0.45 and 0.55. This resulted in an improvement in the thermal insulation performance.

Granulated blast furnace slag (GBFS) used as a source of silica and alumina to produce geopolymers [22].GGBS originally exists in an amorphous state when molten slag swiftly hardens during the production of iron [23].

In keeping with our earlier research [24-28] that attempts to remove any remaining dyes from dyeing baths, especially when reactive dyes are adopted, our objective in this study is to do the same by utilizing

geopolymer cement based on bentonite to remove the color of any remaining reactive yellow 145 dye in the dyeing bath.

In this paper, both bentonite clay (BT) and granulated blast furnace slag (GBFS) were utilized as starting raw material for the synthesis of geopolymer adsorbents. We looked at how the inclusion of slag affected the microstructure and adsorption capability of the bentonite/slag-based geopolymers. Additionally, the adsorption mechanisms of  $\text{Cu}^{2+}$  and reactive yellow 145 were investigated.

## 2. Materials and Methods

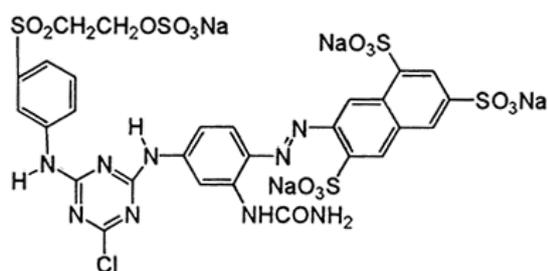
### 2.1. Materials

The main raw materials are bentonite, slag. Pure pellets of sodium hydroxide and  $\text{Na}_2\text{SiO}_3$  are used as activating solution. Bentonite was procured from Sphinx Milling Station, Alexandria, Egypt. Bentonite clay was heated in a lab furnace to 800 °C at a rate of 5 °C per minute, then it was soaked for 2 hours at that temperature before being cooled in the furnace. To obtain the desired size (less than 200mesh), the heat-treated bentonite clay is ground and sieving. Granulated blast furnace slag with a Blaine surface area of  $4700 \pm 50 \text{ cm}^2/\text{g}$  was acquired from the Egyptian iron & steel of Helwan Company. Sodium hydroxide (NaOH) pellets with 99% purity are supplied by ELGoumhouria chemical company, Cairo, Egypt, while sodium silicate liquid (SSL) is acquired from Silica Egypt company, Alexandria, Egypt. Table (1) displays the major chemical composition of the raw materials analysed by X-ray fluorescence.

For adsorption investigation, reactive yellow 145 and  $\text{Cu}(\text{NO}_3)_2$  are used. Copper nitrate obtained from Merk, Germany. The structural formula of reactive yellow 145 is shown in Fig. (1). Deionized water was used to prepare all the solutions.

**Table (1):** Chemical composition of raw materials (wt.%)

Oxides	BT	GBFS	SSL
<b>SiO<sub>2</sub></b>	<b>48.52</b>	<b>32.86</b>	<b>32.8</b>
<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>18.81</b>	<b>1.14</b>	--
<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>9.68</b>	<b>7.02</b>	--
<b>Na<sub>2</sub>O</b>	<b>0.14</b>	<b>0.29</b>	<b>11.7</b>
<b>CaO</b>	<b>17.04</b>	<b>42.56</b>	--
<b>MgO</b>	<b>3.97</b>	<b>11.58</b>	--
<b>SO<sub>3</sub></b>	<b>0.07</b>	<b>2.5</b>	--
<b>K<sub>2</sub>O</b>	<b>1.77</b>	<b>0.15</b>	--
<b>H<sub>2</sub>O</b>	--	--	<b>55.5</b>
<b>Total</b>	<b>100</b>	<b>98.1</b>	<b>100</b>



**Figure 1.** Chemical structure of Reactive Yellow 145

### 2.1.1. Preparation of reactive dye

The reactive dye was made by adding a 3 mL/L (33% NaOH) solution and a 5 g/L sodium carbonate solution, and then heating the mixture at 80 °C for 120 minutes while stirring. After the hydrolysed dye had cooled, diluted H<sub>2</sub>SO<sub>4</sub> was used to neutralize it [29].

### 2.1.2. Preparation of geopolymer

The mix compositions of the samples that were prepared are listed in Table (2). The preparation process can be divided into four parts. To obtain alkaline activator, NaOH (12 M) and sodium silicate with ratio 1:2.5 were first mixed for 30 minutes by a magnetic stirrer. The second part involved progressively adding bentonite and slag to the alkaline solution created in the previous step. The freshly prepared pastes are then placed in stainless steel cube-shaped molds with a one-inch diameter and cured for 24 hours at 70 °C to reach the final setting and hardness. Then the cubes were demolded and hydrated for seven days at 100% relative humidity. The following step involved crushing the cured samples and adding them to the ethanol/acetone (1:1) stopping solution on an electric magnetic stirrer for 30 minutes. The residue is filtered and washed with ethanol, repeatedly washed with HCl and distilled water to remove the excess alkali liquor, and then dried for 24 hours at 50°C. Following drying, materials were ground and sieving to a mean particle diameter of 100 μm then stored in a desiccator.

**Table (2):** Mix composition of mixes Mix name

Mix name	BT	GBFS	NaOH (M)	NaOH/SSL ratio	L/S ratio
BS 1	70	30	12	2.5	0.25
BS 2	50	50	12	2.5	0.21
BS 3	30	70	12	2.5	0.19

## 2.2. Methods

### 2.2.1. Adsorption test

In this study, effects of slag content, pH level, adsorbent dosage, and starting mass concentration of the bentonite-based geopolymer on the reactive

yellow 145 dye effluent and heavy metal (Cu<sup>2+</sup>) solution and contact time on the adsorption properties of the samples were investigated for each solution separately and systematically. In the experiment to explore the best mix, 0.5 gm of adsorbent is added into 30 mL solution of dye and Cu<sup>2+</sup> separately with pH = 7 for 12 hours. HCl can be used to adjust the solution's pH value (2–6). To investigate the impact of various pH values, experiments are conducted under same conditions. To examine amount of adsorbent, different mass of adsorbents (0.05, 0.1, 0.3, 0.5 g/30mL) are added into and initial mass concentration of each solution. The best dosage of adsorbent is added into 30mL solution with different initial mass concentrations (145, 290, 440, 580 mg/L for reactive yellow & 200, 400, 600, 800 mg/L for Cu<sup>2+</sup>) to ascertain the effects of solution concentration. Finally, in the experiment of time contact, adsorbents are added to 30 mL each solution of reactive yellow and Cu<sup>2+</sup> respectively, and shocked in oscillator for 30min, 60min, 180min and 360 min. Reactive yellow and Cu<sup>2+</sup> solution concentrations are determined using a UV spectrophotometer (Spectrophotometer V-670) and atomic absorption spectrophotometer, respectively. The adsorption capacity (mg/g) and removal efficiency (%) are calculated by equations (1) and (2), respectively [24,30].

$$\text{Adsorption capacity } q_e = (C_o - C) V/W \quad (1)$$

$$\text{Removal efficiency } R = (C_o - C) / C_o \times 100 \quad (2)$$

C<sub>o</sub> (mg/L) and C (mg/L) are initial and equilibrium dye concentrations of the reactive yellow 145 dye and Cu<sup>2+</sup> solution respectively. V (L) is the reactive yellow 145 dye and Cu<sup>2+</sup> solution volume, and W (g) is the geopolymer weight.

## 3. Results and discussion

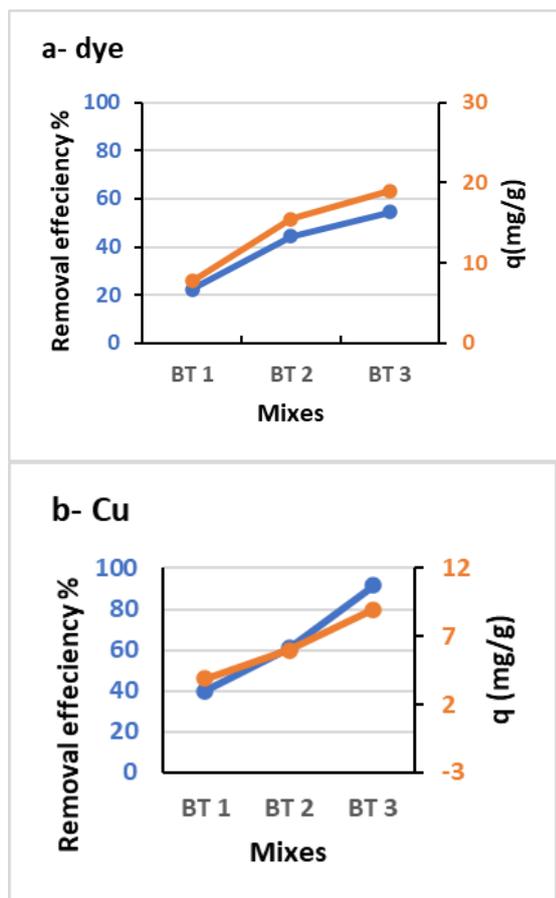
### 3.1. Effect of slag content

The adsorption properties of dye effluent and Cu<sup>2+</sup> onto bentonite/slag based geopolymers with different slag contents are shown in fig. (2) a and b.

The removal efficiency of reactive yellow 145 dye effluent is rises from 22.2 to 54.5% as slag content increases. Although the absorption of Cu<sup>2+</sup> fluctuates slightly between 40 and 91.5%. The BS3 adsorbent has the highest removal efficiency for both Cu<sup>2+</sup> and dye. The BS3 mix is selected for the next investigations based on the outcomes of these two aspects.

### 3.2. Effect of pH

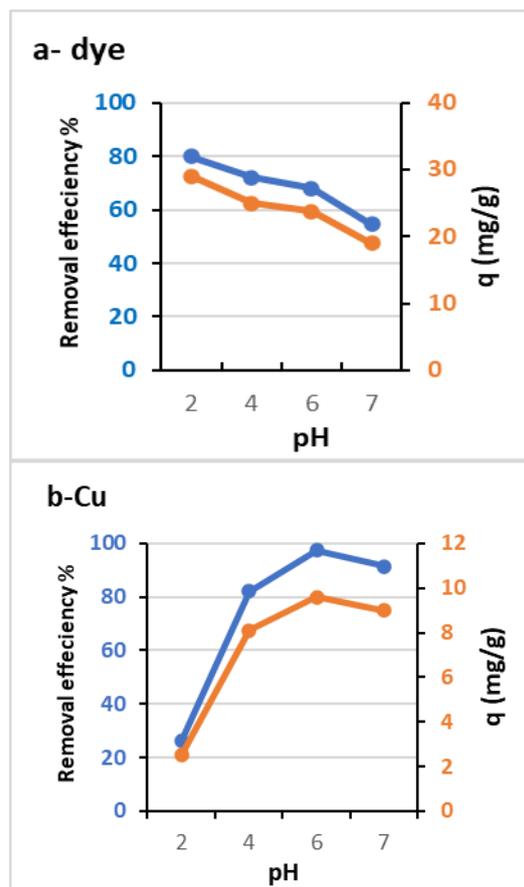
The impact of pH is investigated because it affects the characteristics of metal ions and dye in solutions. The change in the removal efficiency and adsorption of pollutants at different pH values is illustrated in Fig. (3) a & b.



**Figure (2):** Adsorption capacity and removal efficiency of bentonite based geopolymers as a function of slag content for a) dye effluent and b)  $\text{Cu}^{2+}$

The results show that the adsorption capacity of copper increases from 2.5 mg/g to 9 mg/g when the pH value increases from 2 to 6, and the removal efficiency likewise increases from 26 to 97.5%. This may be due to the high concentration of  $\text{H}_3\text{O}^+$  in low pH solutions, which compete  $\text{Cu}^{2+}$  for the available active sites on the BS3 mix and makes  $\text{Cu}^{2+}$  removal difficult [31].

As the pH value increases, the competitive force weakens and the repulsive force grows, increasing the removal rate. The adsorption capacity of  $\text{Cu}^{2+}$  diminishes when the pH of solution rises to 7. For reactive yellow 145 dye effluent, the removal efficiency declines with increasing pH value. The maximum removal efficiency % is at pH 2 with maximum values 80%. Thus, the pH value of 2 is chosen for the next experiment of dye while pH=6 is selected to the next experiment of heavy metal.



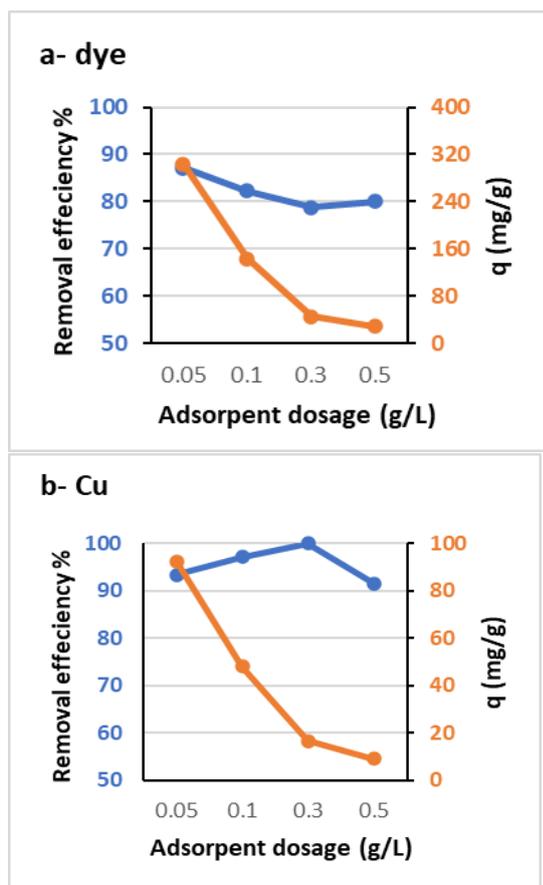
**Figure (3):** Adsorption capacity and removal efficiency as a function of pH for a) dye effluent and b)  $\text{Cu}^{2+}$

### 3.3. Effect of adsorbent dose

The effect of dosage of BS3 mix on the adsorption of reactive yellow 145 dye effluent and  $\text{Cu}^{2+}$  solution separately are shown in Fig. (4)a and b. The results indicate that as the dosage of adsorbent increase from 0.05 to 0.3 g/30mL, the removal efficiency of dye is slightly decrease from 87.2 to 78.8 and then slightly increase at dosage 0.5 g/30mL. This is because, on the surfaces of adsorbents, extra-accumulation and complex interactions may also reveal dependencies [32]. The adsorption capacity of dye is also decreases with increase the dosage up to 0.5 g/30mL.

The results also indicate that the removal efficiency of  $\text{Cu}^{2+}$  increases with adsorbent dosage up to 0.3 g/30mL. The removal efficiency of  $\text{Cu}^{2+}$  increases from 93.3% to 99.9% when the concentration of BS3 is increased from 0.05 g/L to 0.3 g/30mL. This is due to increasing adsorbent dosage creating greater accessible surface area and adsorption sites [33]. Further increase in adsorbent dosage leads to sharp decrease in removal efficiency to 91.5%.

Once equilibrium has been reached, there is no further active site available in the adsorbent. So that the amount of adsorbate that can be removed cannot be changed after equilibrium has been attained [34].



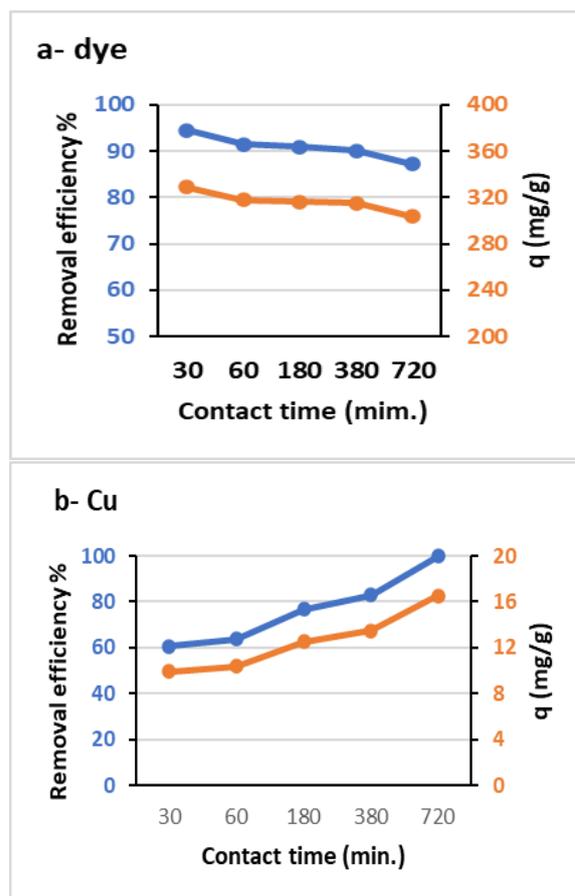
**Figure (4):** Adsorption capacity and removal efficiency as a function of adsorbent dosage for (a) dye effluent and (b)  $\text{Cu}^{2+}$

### 3.4. Effect of contact time

In the adsorption process, the adsorption time is also crucial. The effect of time on the adsorption process of reactive yellow 145 dye and  $\text{Cu}^{2+}$  by BS3 is exhibited in the Fig. (5) a&b.

We found that in the early stages of contact time, the adsorption is rapid and then begins gradually diminishing as contact time increases. After 60 min. the adsorption capabilities essentially remain constant. Over 94% of the entire quantity of dye and  $\text{Cu}^{2+}$  in the solution is absorbed after 60 min. This is because the adsorbate molecules are quickly adsorbed on the surface of the BT3 mix during the early range of contact time due to the abundance of active sites on the adsorbent surface [35]. As the adsorption time increase, adsorbent molecules move from the surface to the inside, reducing the number of effective adsorption sites on the adsorbent surface and slowing the rate of adsorption.

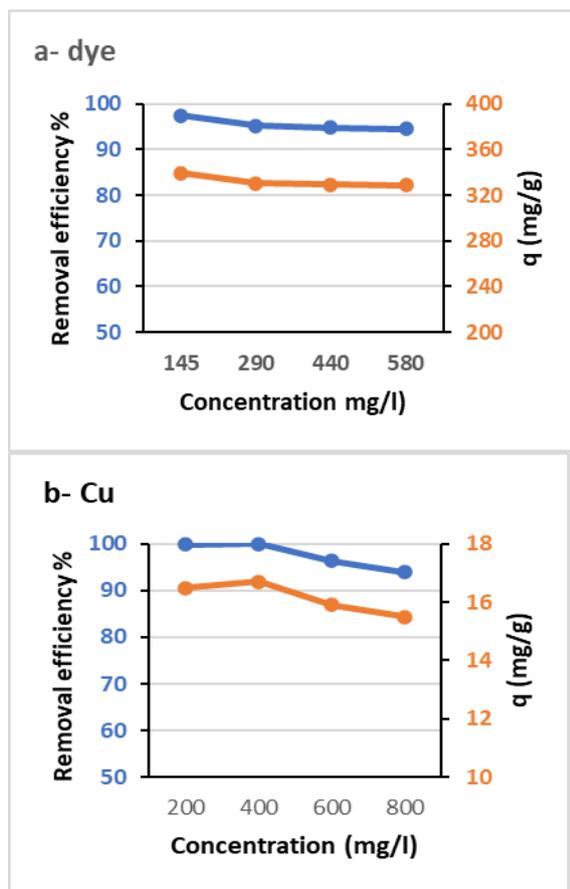
The removal efficiency of copper increases as contact time increases and reaches to maximum value (99.9%) at 720 minutes. It is found that adsorption capacity increase as time increases up to 720 minutes.



**Figure (5):** Adsorption capacity and removal efficiency as a function of time for (a) dye effluent and (b)  $\text{Cu}^{2+}$

### 3.5. Effect of initial concentration of pollutants

Figure (6) (a and b) shows the effect of dye and metal concentration on the adsorption process of cured BS3 geopolymer mixes with optimum pH, dose and time. The dye effluents concentration (145 mg/L) at pH 2 giving maximum decolorization. In addition, adsorption capacity slightly decreases with increasing dye concentration up to (580 mg/L). This phenomenon can be explained by the restricted number of active sites on the adsorbent that can accommodate as the dye concentration rises. The decolorization efficiency drops at high dye concentrations. As the initial concentration of copper solution increase from 200 to 400 mg/l, the removal efficiency is slightly increase then decrease up to 800 mg/l. Because the active sites on the BS3 were not saturated at the low copper concentration. On the other hand, the growth of the adsorption capacity was delayed at the high copper concentration due to the gradual saturation of active sites by  $\text{Cu}^{+2}$  [36].



**Figure (6):** Adsorption capacity and removal efficiency as a function of initial concentration in ppm for (a) dye effluent and (b)  $\text{Cu}^{2+}$

#### 4. Conclusion

In this study, various slag mass ratios of geopolymer adsorbents based on bentonite are synthesised by using sodium hydroxide and SSL as alkaline activator. The adsorption properties of reactive yellow 145 dye and  $\text{Cu}^{2+}$  at different factors such as pH, adsorbent dosage, contact time and concentration of pollutant are thoroughly examined. Following are the conclusions:

- 1) The maximum removal efficiency of reactive yellow 145 dye is achieved by BS3 mix (30 % BT + 70% slag) at pH value 2, 1 g/L adsorbent dosage for 30 min. and at initial dye concentration 145 mg/L.
- 2) Adsorption of heavy metal, the optimum conditions for adsorption of copper by BS3 mix are pH=6, adsorbent dosage 6 g/l for 720 min and at initial concentration 400 ppm.

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