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## Decolorization of Reactive Dyes, Part VI: Eco-Friendly Approach of Reactive Dye Effluents Decolorization Using Geopolymer Cement Based on Metakaolin backed by slag

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**Abstract**

In order to reduce pollution and preserve the environment, we conducted this study to remove the color from the dyeing baths residues from the liquid waste of reactive yellow dye 145, using two different types of geopolymer based on metakaolin or metakaolin mixed with slag by 70 to 30%, and the results showed that this method promising color reduction as an inexpensive and economical method

***Keywords***:Reactive dyes, Metakaolin, Slag..

## 1. Introduction

The production of Portland cement contributes significantly to the world's carbon dioxide ($CO_2$) emissions, accounting for 5% to 7% of all industrial and energy-related $CO_2$ emissions [1]. We now need to create environmentally friendly binders and cut back on the use of cement. One substitute for standard Portland cement (OPC) is geopolymer. According to studies, geopolymers share the same mechanical characteristics as Portland cement binders, but their manufacture has an 80 percent lower carbon and 30 percent lower energy footprint, which might drastically cut their $CO_2$ emissions [2,3].

Geopolymers can be produced chemically by reacting an alkaline solution with alumino silicate minerals that are high in alumina ($Al_2O_3$) and silica ($SiO_2$), like clay, or kaolin, fly ash, bottom ash [4] as well as synthetic silico-aluminate (pure $Al_2O_3$-$SiO_2$) [5]. Geopolymers come in a wide variety of forms, including Polysialate (Si:Al=1, [-Si-O-Al-O-]), Polysialatesiloxo (Si:Al=2, [-Si-O-Al-O-Si-O-]), and Polysialatedisiloxo (Si:Al=3, [-Si-O-Al-O-Si-O-]) [6]. The mechanical strength and setting of the geopolymer are significantly influenced by the concentrations of silica and alumina, respectively [5].

Many geopolymer-based materials are appealing because of their high thermal stability, high early strength, excellent durability, freeze-thaw opposition, low rate of chloride diffusion rate, resistance to abrasion, thermal stability, minimal shrinkage, low cost, and environmentally friendly nature.

Additionally, they reduce the mobility of dyes and heavy metal ions contained in geopolymer materials. Additionally, they lessen the movement of heavy metal ions and dyes present in geopolymer materials. Geopolymers are therefore recommended for industrial uses such as green concrete, construction materials, fire-resistant substance, and adsorption substances [7–15]. Many recent studies have developed geopolymer materials to eliminate dye [14] and heavy metals [16] from sewage in order to solve the environmental concerns that industrial regions faced. Many studies have recently focused on the ability of geopolymers to produce low-cost sorbents for removing colour (dyes), dangerous metals, and detergents from wastewater [17-19].

When kaolin is properly dehydrated at the right temperature (600 $^oC$- 900 $^oC$), it yields metakaolin (MK), which is an anhydrous aluminium silicate.MK is frequently used to create geopolymers. The MK-based geopolymers exhibit thermally insulated

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characteristics [20,21]., as well as greater compressive strength and bonding strength[22].Due to the superior mechanical qualities of MK-based geopolymers, numerous researchers have combined other materials with the system to lower costs, retain outstanding performance, and promote resources usage [23,24].

Jindal et al., [25] suggested that MK-based geopolymers have a wide range of uses, such as a fireproof building material, a protective coating for the surfaces of different materials, including metals, sustainability concrete, or environmentally friendly materials. Another important application area is the use of MK-based geopolymers as "self-cleaning" building materials [26].

Various research demonstrated that geopolymers made from metakaolin as a basic material can be considered good adsorbents for different water wastes, including heavy metals and dyes. [27-29].

Elapasery M.A. et al. [30-33] proposed that metakaolin-based geopolymer cement can be used to remove the color of the reactive dye remaining in the dyeing bath instead of leaving this dangerous waste without treatment.

When iron ore and limestone flux are mixed to form pig iron in the blast furnace, the resulting material is known as "ground-granulated blast furnace slag" (GGBFS).Many researchers have looked at the mixing of slag with industrial waste materials such as fly ash (FA), cement kiln dust (CKD), and silica fume (SF) for production of different geopolymer cement [34-37].35 to 45 percent of the dye used during the dyeing process remains in the waste water. Reactive dyes are the most widely used because of their several benefits, such as their stable structure, bright colour, and functionality in moderate settings [30,31]The dye is removed using chemical and physical decolorization processes, including precipitation, adsorption, oxidation, reduction, coagulation, and electrolysis [38,39].

In this investigation, we compare the adsorption of different mixes incorporating metakaolin and slag-based geopolymer by adsorption of metakaolin-based geopolymer only to obtain the best mix that can remove, in a large proportion, the color of the reactive yellow 145 dye residual in the dyeing process instead of dumping this dangerous waste without treatment.

## 2. Materials and Methods

### 2.1. Materials

**A- Reactiveyellow 145** was utilized for the decolorization studies. The structures of thisdyesis shown.

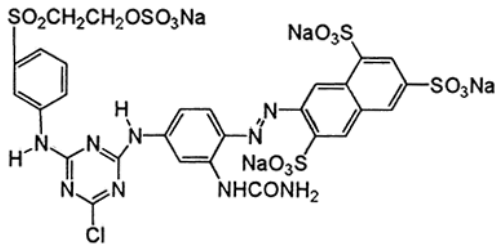


**Figure 1.** C.I. Reactive Yellow 145

**b- Preparation of Hydrolyzed Reactive Dye**

A 3 mL/L sodium hydroxide solution (33%) and a 5 g/L sodium carbonate solution were added, and the reactive dyestuff was heated at 80 °C for two hours while being stirred. The hydrolyzed dye was then cooled and neutralized using diluted sulfuric acid [30].

**c-Metakaolin(MK)** was delivered from Hemts Construction Chemical Company, Cairo, Egypt. Granulated blast furnace slag (GGBFS) was provided by The Egyptian Iron & Steel of Helwan Company, Cairo, Egypt. Those both substances (raw substances)were the sources of alumina and silica utilized to prepare samples of geopolymers. Sodium hydroxide (NaOH) and industrial liquid sodium silicate were used to prepare the alkaline activator solution. The 99% pure NaOH flakes were obtained from EL-Goumhoria chemical company, Cairo, Egypt while the Commercial liquid sodium silicate (LSS) was delivered from Silica Egypt Company, Burg Al-Arab, Alexandria, Egypt. The X-ray fluorescence (XRF) spectrometer for the chemical compositions of Metakaolin (MK)and Granulated blast furnace slag (GGBFS) were determined as shown in Table(1) respectively. The XRF data showed that silica ($SiO_2$) and alumina ($Al_2O_3$), which together make up about 95% of MK, are its principal constituents.

### 2.2. Geopolymer synthesis

#### 2.2.1. Specimens preparation

To produce geopolymer cements, Alkaline activators contained sodium hydroxide (NaOH) and sodium silicate (Na2SiO3) in liquid form.. NaOH pellets were dissolved in distilled water to create a concentration of 10M NaOH, which was then allowed to cool at room temperature. The liquid sodium silicate and sodium hydroxide solutions were combined at a ratio of 2.5:1 to create a clear gel.The geopolymer samples were created from 100 % GGBFS, 100% MK and by substituting metakaolin with 30 % of GGBFS, as shown in Table 2.

Preparation of greopolymer pastes were occurred by mixing the raw materials of each sample with the alkaline activator until a homogenous paste was obtained. Then rapidly, the fresh mixes obtained were poured into a stainless-steelcubic mould possessing a dimension of 25x25x25 mm. The mould is then briefly vibrated to remove all air bubbles and enhance paste

compaction and then left at room temperature under relative humidity (100% R.H.) for 24 hours. Following this period, the cubes were demolded and cured under tap water in a tight plastic container at ambient temperature for 7 days. Finally, after the curing regime, the specimens were removed and crushed. The resultant crushed specimens were stirred with a hydration stopping solution of alcohol/acetone (1:1) to stop further hydration[40,41].

**Table1:Chemical composition of metakaolin and granulated blast furnace slag as determined by XRF analysis in mass (%).**

| Type | Oxide constituents (%) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | $P_2O_5$ | $TiO_2$ | Cl - | L.O.I | Total |
| MK | 64.80 | 30.10 | 0.55 | 0.52 | ---- | 0.13 | 0.10 | ----- | 0.06 | 2.70 | ----- | 0.73 | 99.69 |
| GGBFS | 32.86 | 7.02 | 1.14 | 42.56 | 11.58 | 2.50 | 0.29 | 0.15 | ---- | ---- | ---- | 0.93 | 99.03 |
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | $P_2O_5$ | $TiO_2$ | Cl - | L.O.I | Total |
| MK | 64.80 | 30.10 | 0.55 | 0.52 | ---- | 0.13 | 0.10 | ----- | 0.06 | 2.70 | ----- | 0.73 | 99.69 |

**Table 2: Mix composition of the investigated mixes, liquid/solid (L/S) ratio and setting times**

| Mix | MK (%) | GGBFS (%) | $Na_2SiO_3$: NaOH ratio | L/S ratio |
|---|---|---|---|---|
| MK | 100 | 0 | 2.5 : 1 | 0.56 |
| MK-S2 | 70 | 30 | 2.5:1 | 0.47 |

### 2.2.2. Water of consistency:

The Vicat apparatus is used to determine the standard water of consistency in accordance with ASTM specifications [42]. The amount of liquid needed to create a paste with a standard consistency is the same amount needed to create a paste that allows the vicat plunger (10 mm in diameter) to settle to a point 5 to 7 mm from the bottom of the vicatmould.

### 2.3 Adsorption experiments

A specific amount of the adsorbent was shaken with 50 mL of the dye solution at 30 °C and 140 rpm. Filtration was used to separate the sample solutions' supernatant. When utilizing SHIMADZU spectrophotometry to assess absorbance at maximum wavelength (max = 475 n m for reactive yellow 145 dye), concentration was calculated using the calibration curve. By using a mass balance relationship, the amount of dye that was adsorbed onto the adsorbent, qe (mg/g), was estimated.

$$qe = ( C_o - C ) V/W \quad (1)$$

Where V is the solution volume in litres (L), $C_o$ is the initial dye concentration in milligram's per litre (mg/L), C is the equilibrium liquid-phase dye concentration in milligram's per litre (mg/L), and W is the weight of the adsorbent (g).

$$\text{Removal efficiency\%} = 100 X (q_e / C_o ) \quad (2)$$

## 3. Results and discussion

### 3.1 Factors affecting on the adsorption

#### 3.1.1 Effect of pH

If we look closely at Figure 2, we find that it gives us many evidences to show the change in the removal efficiency% when using the dye residues of the reactive dye yellow 145 at the changing pH of the adsorption bath. In order to determine the optimal pH value for geopolymer mixtures based on metakaolin MK or metakaolin mixed with slagMK-S2 dye-reated, the optimal pH value was studied, starting from a value of 2 to a value of 10. The results in Figure 2 clearly indicate that with all wastewaters, the percentage efficiency of color removal decreases with increasing pH. The highest decolorization efficiency was at pH 2 for both geopolymer mixtures (MK and .MK-S2). The maximum values were 0.5% for MK and 17% for MK-S2.

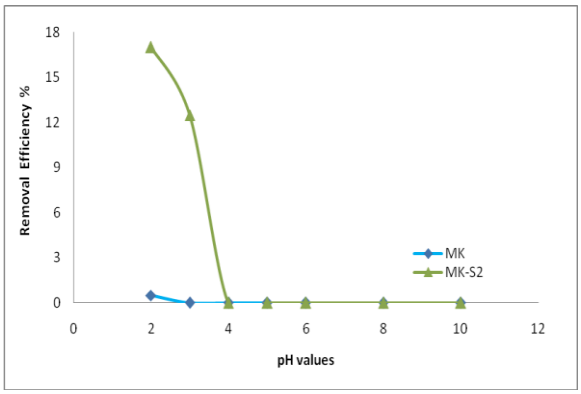


**Figure 2.** Effect of pH on dye removal efficiency % (Time 2hrs, Temperature 30°C, wt. of adsorbent 0.01g, concentration of dye 10 mg/L)

#### 3.1.2 Effect of adsorbent dose

It is worth noting here to point out that the results presented in Figure 3 show the relationship between the results of the adsorption concentration on the removal efficiency%. The adsorption of the dye under study was investigated at different concentrations (0.01–0.1 g/50 ml) of geopolymer cement, for 2 h using the dye waste of reactive yellow 145. The dye

concentration was (10 mg/L) at pH 2 for MK and MK-S2. The results show conclusively that an increase in the removal efficiency % occurs with a decrease in the weight of the adsorbent. We can say that the highest removal efficiency % was 2.5% at 0.02g/50 ml for MK and also 17% at 0.01g/50 ml for MK-S2.

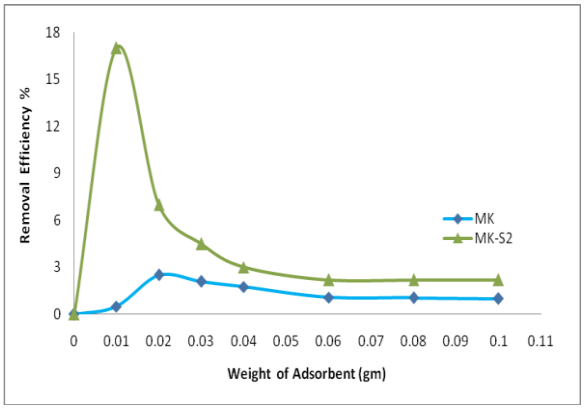


**Figure 3.** Effect of adsorbent weight on the removal efficiency % (Time 120 min, Temperature 30 $^{o}$C, concentration of dye 10 mg/L, pH 2)

### 3.1.3 Effect of time

When studying and determining the optimal duration of reaction of the reactive dye with geopolymer cement materials, the removal of the dye was studied at different time periods ranging from one to five hours. We can also say here that the results shown in Figure 4 show in a very specific scientific way that the % color removal efficiency increases by prolonging the adsorption time until it reaches a period of 4 hours, achieving a maximum color removal efficiency of 7% for the MK geopolymer mixture, while the color removal efficiency was 17% At two hours for the geopolymer mixture MK-S2, then the removal efficiency % decreases.

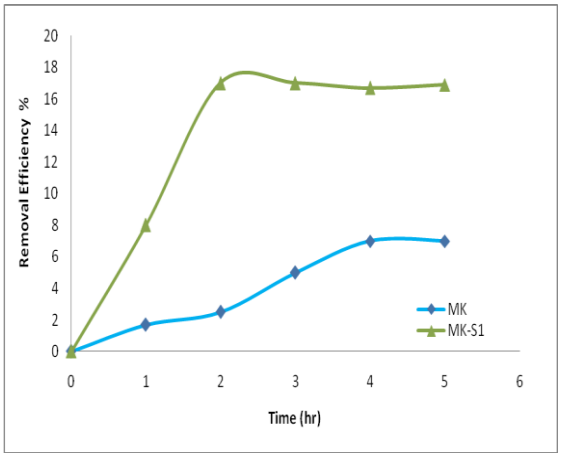


**Figure 4.** Effect of time on the removal efficiency % (Weight of adsorbent 0.02g, for Mk and 0.01 for MK-S2, Temperature 30 $^{o}$C, concentration of dye 10 mg/L, pH 2)

### 3.1.4 Effect of Dye concentration

The results shown in Figure 5 give us a clear and complete scientific perception of the relationship between the effect of the dye concentration and the color removal efficiency % when using the constant weight of the cured geopolymer mixture MK and MK-S1 with both time and optimum pH. The absorption of the dye was studied with different concentrations of the dye (1.25 - 100 mg/L). We reached the highest percentage of color removal, where the color removal efficiency was 15% for MK cement and the color removal efficiency was 25.8% for MK-S2 geopolymer cement. Also, the percentage of color removal efficiency decreases as we increase the dye concentration.

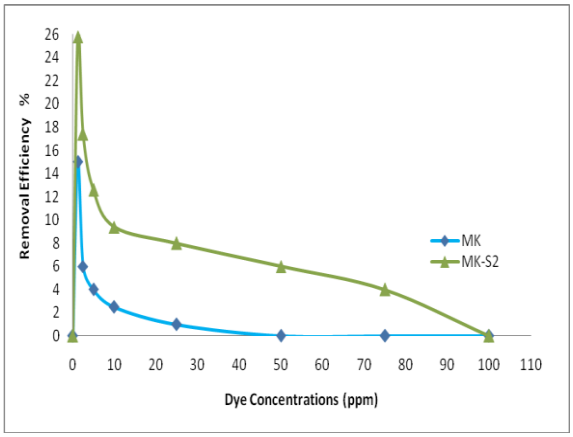


**Figure 5.** Effect of dye concentration on the removal efficiency %(Weight of adsorbent 0.01g, Time 2hrs and pH 2 , for MK-S2 Weight of adsorbent 0.02 g , Time 4hrs and pH 2 for MK, Temperature 30 $^{o}$C for all)

## 4. Conclusion

We can clearly summarize what we have reached, that this work gives the green light on the possibility of using a geopolymer based on metakaolin MK or a mixture between metakaolin and slag at a rate of 70 to 30% MK-S2 to remove the residual color in the dyeing baths of the reactive yellow dye 145. We can also say that the maximum absorption capacity of the reactive yellow 145 dye under study using slag-based geopolymer and metakaolin was better than geopolymer based on metakaolin only, meaning that the presence of 30% slag was useful in removing color.

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