



## Preparation and Optimization of Coal Fly Ash-Based Geopolymer and Its Application as an Adsorbent for Basic Yellow 2 and Methylene Blue Dyes

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methylene blue.

**ABSTRACT.** The preparation of geopolymer from coal fly ash (CFA) solid waste of Jepara PLTU has been carried out by activating CFA using a 10 M NaOH solution and Na<sub>2</sub>SiO<sub>3</sub>. Characterization of CFA and geopolymer using X-ray fluorescence (XRF), X-ray diffractometer (XRD), Scanning Electron Microscopy (SEM), and Fourier Transform Infra-Red (FTIR) spectroscopy. The adsorption process was studied under parameters including pH, geopolymer mass, contact time, and initial adsorbate concentration. The optimum conditions for the adsorption process of basic yellow 2 dye with fly ash-based geopolymer were optimum at solution pH of 11; adsorbent mass of 0.15 g; contact time of 120 minutes; and initial concentration of 80 ppm, while methylene blue adsorption was at solution pH of 11; adsorbent mass of 0.15 g; contact time of 120 minutes; and initial concentration of 60 ppm. Coal fly ash, a solid waste product from the Jepara PLTU, can be used to produce geopolymers through geopolymerization in the presence of activators such as NaOH and Na<sub>2</sub>SiO<sub>3</sub>. The adsorption capacities of these geopolymers for basic yellow 2 and methylene blue dye solutions are 26.55 and 19.825 mg/g, respectively.

To date, coal has been one of the most abundant alternative energy sources (Gollakota *et al.*, 2019) and is considered economical compared to petroleum. Steam Power Plants (PLTU) use coal as fuel to generate steam, which then powers the electricity grid. Despite its attractiveness as an alternative energy source, coal use produces polluting exhaust gas, fly ash, bottom ash, and boiler slag, which can pose environmental problems (Alterary and Marei, 2021). Gaseous pollutants, such as CO<sub>2</sub>, NO<sub>x</sub>, CO, and SO<sub>2</sub>, are generally removed from flue gases using purification techniques before being released into the atmosphere. Coal fly ash (CFA) is a pozzolanic material based on silica or silica-alumina. CFA compounds are silica-based or silica-alumina-based pozzolanic materials. Its main components are organic and inorganic materials, including amorphous and crystalline phases (Alterary and Marei, 2021). If CFA is not recycled in solid waste processing, it will be temporarily stockpiled in the landfill. Although no longer classified as Hazardous and Toxic waste (B3), CFA can harm the environment over a relatively long period. Therefore, direct disposal without prior treatment is prohibited; thus, it is not allowed to dispose of directly without treatment (Acar and Atalay, 2013).

The use of CFA has been widely adopted in the concrete industry as a cement additive, in the ceramic industry for wastewater treatment, zeolite synthesis, and soil stabilization, and in the synthesis of fly ash nanoparticles to improve their properties. One method for converting fly ash into other materials involves its conversion into geopolymers, which consist of an amorphous alumina-silicate framework (Al-Zboon *et al.*, 2011).

Geopolymers are novel inorganic aluminosilicate polymer products with a three-dimensional (3D) network structure similar to zeolite. Composed of a tetrahedral network of Al and Si, geopolymer can be produced through geopolymerization. Synthesizing geopolymers from CFA will be significantly more profitable because, in addition to utilizing industrial solid waste, it enables the production of more environmentally friendly materials (Temuuji

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*et al.*, 2009). In this study, geopolymers were synthesized from ash waste from a coal-fired power plant (PLTU) in Jepara, Indonesia, a process that other researchers have not reported for use as a dye adsorbent.

## RESEARCH METHODS

### Geopolymer Synthesis

Geopolymers were produced from CFA and alkaline activators. The CFA sample used in this study was from PLTU Jepara. 40 g of CFA was added to 400 mL of water, and the mixture was filtered and baked in an oven at 110 °C for 2 hours. The alkaline activator was synthesized from sodium silicate powder and sodium hydroxide (Merck). An alkaline silicate activator was prepared by mixing 10 M NaOH and a Na<sub>2</sub>SiO<sub>3</sub> solution in a 1:1 ratio. After the alkali silicate activator was formed, CFA was added in a 2:1 solid/liquid ratio. The mixture was stirred for 10 minutes until a homogeneous geopolymer paste was obtained. The paste was then poured into a cylindrical container and stored at room temperature for 24 hours. The geopolymer was then stored at 60 °C for 6 hours. The geopolymer was milled and sieved to obtain a particle size fraction less than 250 μm. Prior to application, the geopolymer samples were characterized using XRD, XRF, FTIR, and SEM.

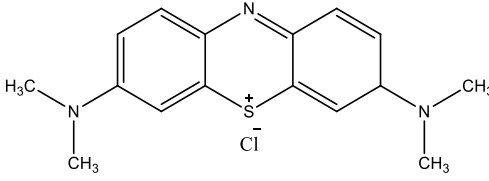
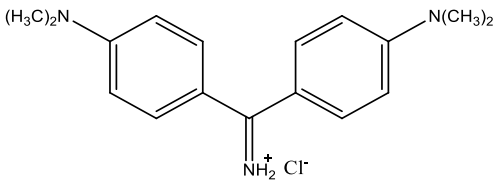
### Characterization

Characterization of ash and ash-based geopolymers was carried out to determine their physicochemical properties. Chemical composition was analyzed by XRF using a Baxs S2 Ranger spectrometer and by XRD using a Bruker D8 Advance diffractometer. The functional group was identified by Fourier transform infrared spectroscopy (FTIR) on KBr pellets. Pellet samples were analyzed with a Shimadzu FTIR spectrophotometer, model 820431 PC, in the wavenumber range of 400 – 4000 cm<sup>-1</sup>. Morphological and structural analyses were performed using a scanning electron microscope (SEM). Adsorbate concentrations were analyzed with a Hitachi UH5300 UV-Vis Spectrophotometer.

### Cationic Dye

In this study, the dyes used in all experiments were basic yellow 2 (BY2) and methylene blue (MB). Their chemical formulas and some other specific characteristics are presented in Table 1. A 100 mg/L solution of MB and BY2 was prepared by dissolving 0.1 g of each dye in distilled water in a 1000 mL volumetric flask.

**Table 1.** Name and structure of adsorbate dyes.

Adsorbate Name	λ max (nm)	Relative molecular mass (g/mol)	Structure
Methylene blue C <sub>16</sub> H <sub>18</sub> C <sub>1</sub> N <sub>3</sub> S	664	319.852	
Basic yellow 2 C <sub>17</sub> H <sub>22</sub> C <sub>1</sub> N <sub>3</sub>	432	303.834	

### Adsorbent Performance Test

The adsorption performance tests were carried out in an Erlenmeyer flasks containing the desired adsorbent mass and 50 mL of dye solution. The mixture was stirred at 200 rpm. The adsorption conditions are shown in Tables 2 and 3.

**Table 2.** MB adsorption condition.

Variable applied	Initial concentration (ppm)	Number of Adsorbents (g)	Contact Time (minutes)	pH
Adsorbate Concentration (ppm)	30	0.15	120	5
	40			
	60			
	80			
Number of Adsorbents (g)	60	0.10	120	5
		0.15		
		0.20		
		0.25		
Contact Time (minutes)	60	0.15	30	5
			60	
			90	
			120	
pH	60	0.15	120	3
				5
				9
				11

**Table 3.** BY2 adsorption condition.

Variable applied	Initial concentration (ppm)	Number of Adsorbents (g)	Contact Time (minutes)	pH
Adsorbate Concentration (ppm)	40	0.15	120	5
	60			
	80			
	100			
Number of Adsorbents (g)	80	0.10	120	5
		0.15		
		0.20		
		0.25		
Contact Time (minutes)	80	0.15	30	5
			60	
			90	
			120	
pH	80	0.15	120	3
				5
				9
				11

To vary the pH, 0.1 M NaOH and 0.1 M HCl solutions were used. Adsorption tests were conducted to determine the %Removal and the amount of dyes adsorbed on the geopolymer. After each adsorption test, the samples were centrifuged at 2500 rpm for 10 minutes to separate the solid phase from the liquid phase, and the dye concentration was determined from the UV-Vis absorbance spectrum using a calibration method. The concentrations of the solution before and after adsorption were measured using a Hitachi UH5300 UV-Vis spectrophotometer. The percentage of adsorbate lost during adsorption corresponds to the removal efficiency. This efficiency can be calculated using Equation 1.

$$\text{The removal of efficiency (\%)} = \frac{(C_o - C_e)}{C_o} \times 100\% \quad (1)$$

Description:

$C_o$  = initial concentration (mg/L)

$C_e$  = concentration when equilibrium is reached (mg/L)

To compare adsorption capacity, the amount of adsorbate adsorbed by the adsorbent is expressed in mg/g. The pH of the MB solution at 60 ppm and the BY2 solution at 80 ppm were adjusted to 11 with 0.1 M NaOH. Then, 50 mL of the dye solution was mixed with 0.15 g of the adsorbent. The mixture was stirred for 120 minutes at a speed of 200 rpm. The solution was then transferred to a centrifuge tube to separate the solid and liquid phases, resulting in a clear solution. This solution was then analyzed by UV-Vis spectroscopy. At equilibrium, the adsorption capacity can be calculated using Equation 2.

$$q = \frac{v}{m} (C_o - C_e) \quad (2)$$

Description:

$q$  = adsorption capacity (mg/g)

$C_o$  = initial concentration (mg/L)

$C_e$  = concentration when equilibrium is reached (mg/L)

$v$  = adsorbate volume (L)

$m$  = adsorbent mass (g)

## RESULTS AND DISCUSSION

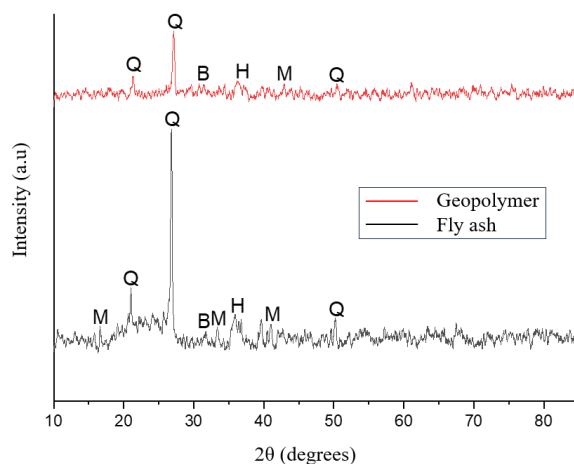
### Characterization

The chemical composition of fly ash and geopolymers, determined in the study, is presented in Table 4. It appears that CFA contains the oxides SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and CaO. The total percentages of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> exceed 70% of the sample weight, which, according to Ahmaruzzaman (2010), is characteristic of type F fly ash. After the addition of the activating solution (geopolymerization), geopolymers with a Si/Al ratio greater than 2 show a polysilox-disilox form (-Si-O-Si-Si-O-O) (Embong *et al.*, 2016).

**Table 4.** Chemical composition of fly ash and geopolymers.

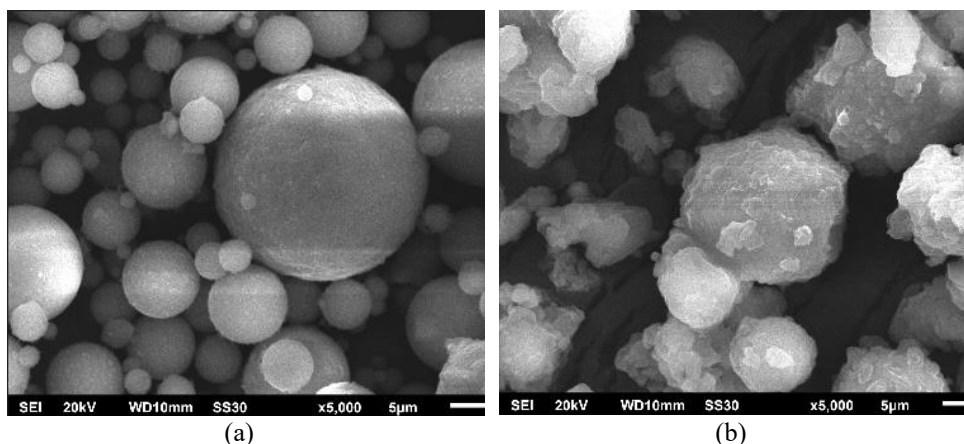
Oxide Components	Rate (%)		Components	Rate (%)	
	Fly Ash	Geopolymer		Fly Ash	Geopolymer
SiO <sub>2</sub>	42.1	39.5	Si	30.4	27.8
Al <sub>2</sub> O <sub>3</sub>	16	13	Al	12	9.7
Fe <sub>2</sub> O <sub>3</sub>	26.3	31.27	Fe	37.5	42.35
CaO	7.67	7.83	Ca	9.74	9.52
K <sub>2</sub> O	2.86	2.96	K	4.11	4.08
TiO <sub>2</sub>	1.71	1.85	Ti	1.89	1.96

The decrease in SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> content before and after activation is due to the dissolution and partial loss of silicon during activation. The SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> present in fly ash are not completely soluble in alkaline solutions; only the amorphous phases of Si and Al are more soluble (Xu and Van Deventer, 2002). The XRD patterns of fly ash and geopolymer are shown in Figure 1. The XRD results indicate that quartz (SiO<sub>2</sub>) is the dominant crystalline phase. In addition to quartz, mullite, hematite, and belite are also observed. The peak intensities are higher for fly ash than for geopolymer, indicating that the addition of an activator can reduce these intensities. Some peaks in mullite also disappear, likely due to the reactive structure of alumina silicates (Lecomte *et al.*, 2006). The narrow peaks broaden to form peaks of much lower intensity, which is characteristic of amorphous materials. Overall, these results indicate that the crystalline phase of alumina-silicate transforms into an amorphous structure during geopolymerization (Al-Zboon *et al.*, 2011).



**Figure 1.** Diphractograms of fly ash and geopolymers (Q=Quarsa, M=Mullite, B=Belite, H=Hematite).

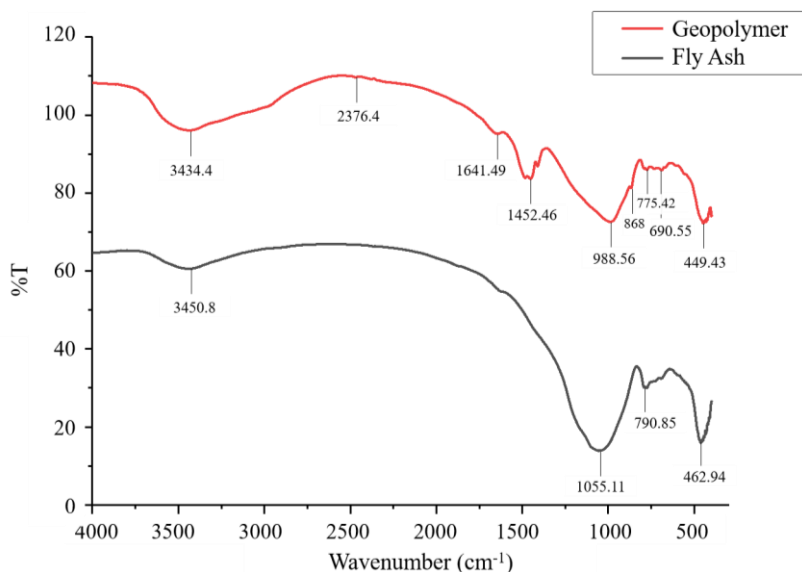
In addition to determining the crystal structures of fly ash and geopolymers by XRD, their surface textures were analyzed by SEM. [Figure 2](#) illustrates the structure of fly ash and geopolymer. The ash surface is smooth and spherical, while the geopolymer surface is rougher and more porous. During activation, the activating solution continuously interacts with the fly ash particles, roughening their surface and enlarging their pores, thereby increasing their adsorption capacity. The circular shape of the particles indicates the presence of aluminosilicate (Si–Al) in the fly ash.



**Figure 2.** The morphology of the magnification is 5000× (a) fly ash and (b) geopolymer.

The IR spectra are shown in [Figure 3](#), and the results of the functional group analysis of ash and geopolymer are presented in [Table 5](#). The IR spectra show absorption bands at 3450.8  $\text{cm}^{-1}$  for ash and 3434.4  $\text{cm}^{-1}$  for geopolymer, indicating stretching vibrations of the –OH group. The absorption band of 2376.4  $\text{cm}^{-1}$  indicates stretching vibrations of the Si–H bond in the geopolymer. The absorption at 1641.49  $\text{cm}^{-1}$  shows a weak peak, which may be due to the CO bending strain of unburned carbon in fly ash. The band at 1452.46  $\text{cm}^{-1}$  is associated with the presence of sodium carbonate, produced by the reaction of residual sodium with atmospheric carbon dioxide, and indicates the formation of geopolymers (Novais *et al.*, 2019).

The absorption bands observed at 1055.11  $\text{cm}^{-1}$  and 790.85  $\text{cm}^{-1}$  for ash, and at 988.56  $\text{cm}^{-1}$  and 868  $\text{cm}^{-1}$  for the Si–O and Al–O functional groups, indicate network formation within the geopolymer structure, i.e., a reorganization of the Si and Al environments during geopolymer formation (Liu *et al.*, 2016). The peaks appearing at 775.42  $\text{cm}^{-1}$  and 690.55  $\text{cm}^{-1}$  for the geopolymers are attributed to the Si–O–Si and Si–O–Al vibrational modes, respectively, confirming the presence of mullite and quartz (Criado *et al.*, 2005). The band at 462.94  $\text{cm}^{-1}$  is due to the symmetrical bending vibration of the Si/Al–O bond in the fly ash, while the band at 449.43  $\text{cm}^{-1}$  can be attributed to the Si/Al–O bond in the geopolymer (Siyal *et al.*, 2018). This observation is also supported by XRD results, which highlight the presence of an amorphous phase and the formation of new reaction products.



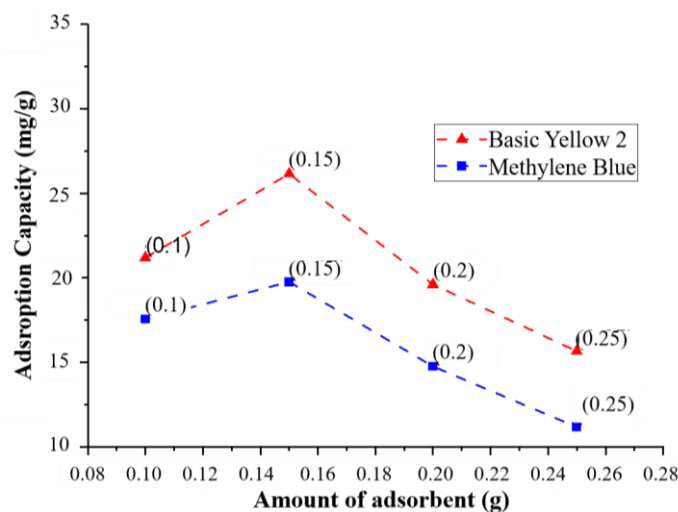
**Figure 3.** Infrared spectrum of ash and geopolymers.

**Table 5.** The infrared spectrum interpretation.

Functional Groups	Wavenumber (cm <sup>-1</sup> )	
	Fly Ash	Geopolymer
-OH	3450.8	3434.4
Si-H Stretching	-	2376.4
C=O Stretching	-	1641.49
O-C-O Stretching	-	1452.46
Si-O Stretching	1055.11	988.56
Al-O Stretching	790.85	868
Si-O- Si Bending	-	775.42
Si-O-Al Bending	-	690.55
Si/Al-O Bending	462.94	449.43

**Adsorption Ability Test**

The adsorption results are shown in Figure 4. The adsorbed amount increased for geopolymer amounts of 0.1 and 0.15 g. This increase was due to higher specific surface area and greater availability of free space for the dye (Acisli *et al.*, 2020). Conversely, an increase in the amount of adsorbent reduced the amount of adsorbate for an adsorbent amount of 0.25 g. This is explained by the concentration gradient between the solute concentration in the solution and the concentration on the adsorbent surface, which causes a decrease in the amount of adsorbent (Padmavathy *et al.*, 2016). The optimal adsorption amount was obtained at a total geopolymer amount of 0.15 g.



**Figure 4.** The relationship curve of the number of adsorbents to the amount of adsorbent (mg/g).

### Optimal Effect of pH on the Adsorption of MB and BY2

The effect of pH on the adsorption of MB and BY2 by geopolymers was studied by varying the initial pH of solution from 2 to 11. The results are shown in Figure 5. According to Figure 5, maximum dye adsorption by geopolymers was observed at pH 11. In acidic media, the presence of acid increases the number of protons in the solution, which decreases the probability of dye binding by the geopolymer. At low pH, the functional groups of coal ash-based geopolymers are partially positively charged due to protonation by  $H^+$  ions. This can reduce the adsorption capacity of cationic dyes due to repulsion between the adsorbent's active sites and the adsorbate, and to competition between free  $H^+$  ions and dye cations for binding to the adsorbent's active sites (Tumin *et al.*, 2008). An increase in pH is accompanied by an increase in the adsorbed amount. This is because the geopolymer surface tends to ionize, releasing  $H^+$  ions in an alkaline environment, which makes it negatively charged and attracts the positively charged cationic dyes.

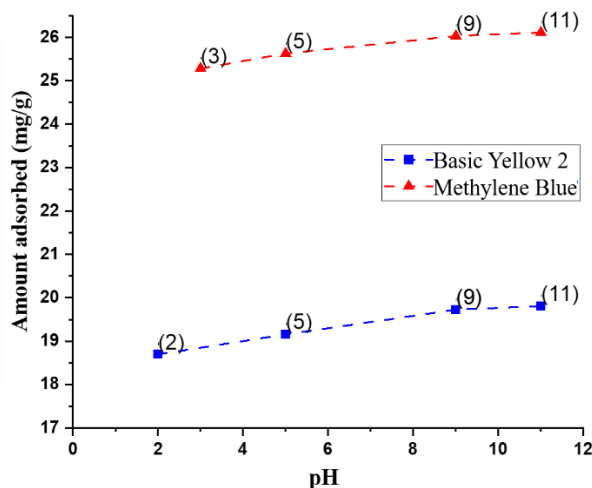


Figure 5. The pH relationship curve with the amount of adsorption (mg/g).

The point of zero charge ( $pH_{PZC}$ ) was determined by measuring the final pH after adsorption. The difference between the initial and final pH ( $pH_i - pH_f$ ) was plotted graphically as a function of the initial pH ( $pH_i$ ), and the point where  $pH_i - pH_f = 0$  was considered as the  $pH_{PZC}$ . The results are shown in Figure 6.

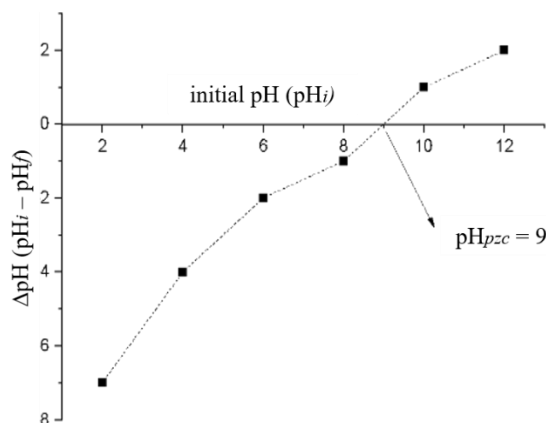


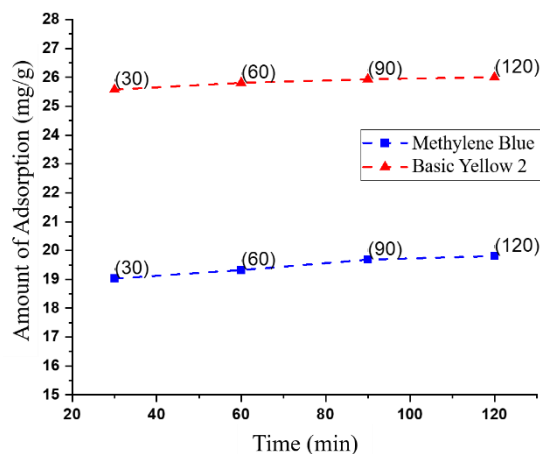
Figure 6. Zero charge point graph for geopolymers.

According to Figure 6, the  $pH_{PZC}$  of the geopolymer is determined to be 9. At  $pH < 9$ , the surface becomes positively charged; at  $pH > 9$ , the surface becomes negatively charged. These results indicate that as the pH of the system increases and the concentration of  $H^+$  ions decreases, the number of negatively charged sites increases while the number of positively charged sites decreases.

### Optimum Contact Time of MB and BY2 Adsorption

The effect of contact time on the adsorption of the aqueous phase on geopolymers was studied in the time range of 30 to 120 minutes. The results are shown in Figure 7. It can be observed that the adsorption of MB and

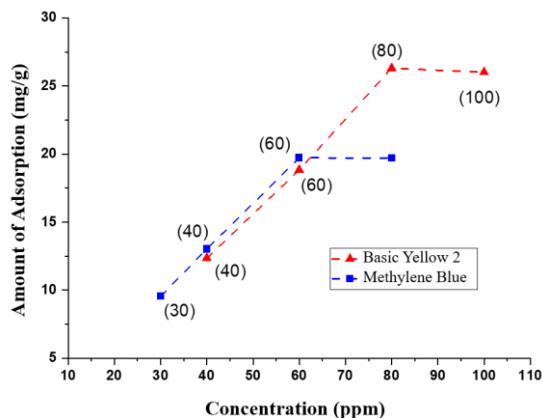
BY2 by geopolymers increases with time. This is explained by the fact that the longer the contact time, the more geopolymer particles collide with and interact with the dye solution, thereby increasing its adsorption capacity. The optimal contact time for the dye solution is 120 minutes.



**Figure 7.** The contact time relationship curve to the amount of adsorption (mg/g).

### Optimum Concentration of MB and BY2 Adsorption

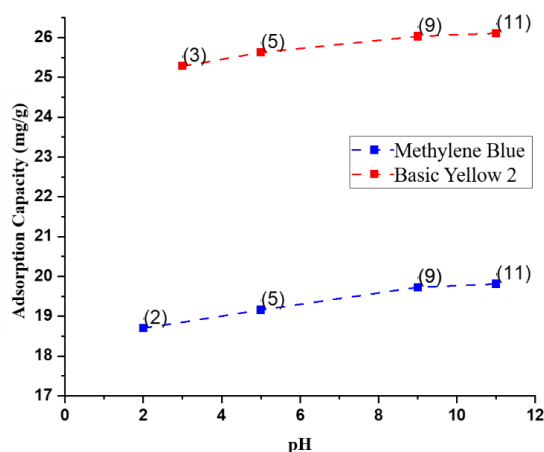
The initial concentration variation treatment aims to determine the optimal concentration required for optimal adsorption of MB and BY2 dyes by geopolymers. The effect of various concentrations on the adsorbed amount is illustrated in Figure 8. The adsorption amount increases with the initial dye concentration, from 30 to 60 mg/L for MB and from 40 to 80 mg/L for BY2. After maximum adsorption, the adsorbent sites are completely occupied by dye molecules, so there are no sites available for further adsorption. The dye concentration is related to the number of active sites on the adsorbent surface. If the number of active sites is large enough relative to the amount of dye, the adsorption efficiency will be high until the number of active sites is equal to the amount of dye.



**Figure 8.** The concentration relationship curve with the amount of adsorption (mg/g).

### Determining the Optimum pH for the Adsorption of MB and BY2

One factor that affects adsorption is pH, which depends on the acidity or basicity of the medium, and affects adsorption due to the varying cationic nature of dye. Variations in pH can modify the surface charge distribution of the adsorbent and dye due to protonation and deprotonation reactions of functional groups. Therefore, determining the optimal pH aims to assess the effect of acidity on the adsorption of methylene blue and basic yellow 2. The effect of pH on the adsorption capacity of methylene blue and basic yellow 2 by geopolymers is shown in Figure 9.



**Figure 9.** The pH relationship curve with the adsorption capacity (mg/g).

According to [Figure 9](#), the maximum dye adsorption capacity by geopolymer adsorbents was observed at pH of 11, with values of 19.8 and 26.1 mg/g for MB and BY 2, respectively. In acidic media, the presence of acid increases the proton concentration of the solution, which decreases the probability of dye binding by the geopolymer. At low pH, the functional groups of coal ash-based geopolymers are partially positively charged due to protonation by  $H^+$  ions. This can reduce the adsorption capacity of cationic dyes due to repulsion between the active sites of the adsorbent and adsorbate, and competition between free  $H^+$  ions and dye cations for binding to the active groups of the geopolymer. Therefore, the adsorption capacity increases with pH. This occurs because the geopolymer surface tends to ionize, releasing  $H^+$  ions in an alkaline environment, so that the surface becomes negative and interacts with the positively charged cationic dye.

## CONCLUSION

Coal fly ash from the PLTU Jepara (Indonesia) thermal power plant can be used to produce geopolymers through geopolymerization, in the presence of activators such as NaOH and  $Na_2SiO_3$ . The optimal conditions for the adsorption of Basic Yellow 2 by geopolymers are pH 11, an initial concentration of 80 ppm, an adsorbent amount of 0.15 g, and a contact time of 120 min. Similarly, the optimal conditions for the adsorption of methylene blue are pH 11, an initial concentration of 60 ppm, an adsorbent amount of 0.15 g, and a contact time of 120 min. The adsorption capacities of geopolymers for Basic Yellow 2 and methylene blue solutions are 26.55 and 19.825 mg/g, respectively.

## CONFLICT OF INTEREST

There are no conflicts of interest in this article.

## AUTHOR'S CONTRIBUTION

EH: Conceptualization, Supervision, and Analysis; EP and YH: Analysis, Writing, and Review; KDN: Review; AM and IDR: Experiment and Investigation.

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