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# Characterization and Application of Greener-Activated Coal Fly Ash for Phosphate Adsorption in Donan River Water Samples

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**Abstract:** The Karangkandri PLTU (Steam Power Plant) Industry in Cilacap is one of the large industries in the Cilacap industrial area which has abundant coal burning waste in the form of fly ash. The excess fly ash from PLTU Karangkandri can be used for adsorption of polluted phosphate ions in the Donan River. The aim of this research is to determine the characterization of fly ash as an adsorbent using FTIR (Fourier Transform Infra-Red), XRD (X-Ray Diffraction) and XRF (X-Ray Fluorescence) instruments and its ability to adsorb phosphate ions in polluted water sample. The first stage of this research was to take water samples from the Donan River at the 3 most polluted points. The second stage was to activate fly ash using wet grinding and hydrothermal methods as well as characterization of fly ash using FTIR, XRD and XRF instruments. Then the effectiveness of the fly ash adsorbent in adsorbing phosphate ion tested by using the spectrometric method. The analysis was carried out with the influence of mass variations in the adsorbent of 50, 100, 150, and 200 grams per 500 ml of water sample. This research found that activated fly ash by greener activation can removed 82,8% phosphate ion with 0,5g fly ash, removed 72.4% phosphate ion with 1 gram fly ash, removed 77.6% phosphate ion with 2 grams of fly ash. And the best one is 5 grams of fly ash can remove 93.2% phosphate ion.

**Keywords: adsorption; fly ash; phosphate; hydrothermal**

## Introduction

Sources of clean water on earth are decreasing and are predicted to experience scarcity by 2050, this is due to the large amount of technological growth that is not balanced with correct enviromental management [1]. It is estimated that 80% of waste is directly returned to the environment without any processing [2] and rivers are one of the water sources that are mainly affected by the disposal of this waste [3]. River pollution has a negative impact on human health and environmental health [4].

The adverse impacts caused by river pollutant waste can be in the form of B3 liquid waste, namely hazardous waste whose quantity, concentration, physical and chemical properties can cause disease for humans and animals living around the river environment[5].

The Donan River is the largest river that surrounds the Cilacap City, its flow passes through a large industrial area, namely PT. Pertamina RU IV Cilacap, PT. Holcim

Indonesia Tbk Cilacap, and home industries [6]. The Donan River empties into Segara Anakan before heading to the sea, so it is important to research the status of its water quality standards every year in order to preserve the biota and maintain the health of the water environment [7]. In 2024 the level of pollution in the Donan River will increase to moderately polluted compared to the previous year, the water quality parameters BOD, COD, TSS, TDS for oil and detergent are heavily polluted, then the presence of heavy metal ions and phosphate does not meet class III water quality standard[8]. This pollution causes the biota population in the waters of the Donan River to be polluted [9],[10].

The Research Team have also carried out an introduction regarding the presence of pollution in several water quality standard parameters (BOD, COD, Dissolved Oxygen, Phosphate, nitrate, Pb and Cd) with sampling data can be seen in the Table 1.

**Table 1.** Sampling results of several water parameters (mg/l) in the Donan River

| Water Parameters (mg/l) | Site point (7°42'43.11 "S 108°59'26.29"T) | Class (III) water quality standards |
|---|---|---|
| BOD | 12 | 6 |
| COD | 40.5 | 40 |
| DO | 7,6 | 3 |
| **Phosphate** | **1,343** | **1** |
| Nitrate | 4,24 | 10 |
| Pb | 0.184 | 0,05 |
| Cd | 0.0066 | 0,01 |

Based on the data in Table 1, preliminary research carried out by researchers shows that there is phosphate ion pollution in the Donan River which is classified as quite polluted because it does not meet the threshold for class III water quality standards. As is known, phosphate is an important element for plant growth [11], but increasing phosphate concentrations in rivers causes excessive algae growth and causes a reduction in dissolved oxygen in the water, resulting in ecosystem damage [12], [13]. Therefore, it is important to maintain river environment from phosphate pollution.

On the other hand, PLTU Cilacap has abundant fly ash waste which can be used as an adsorbent to absorb phosphate ions [14] in Donan river water samples. Before being used as a fly ash adsorbent, it is activated using wet grinding and hydrothermal methods, this method is ecofriendly method compared with conventional acid-based method, for example using sulfuric acid or HCl which often produce hazardous resiues and corrosion issues, this methode also produce little waste which functions effectively to remove impurities in fly ash so that it can carry out more optimal adsorption [15]. Fly ash is ground to a size of 250 mesh, then soaked with NaOH, this aims to reduce the size of the fly ash and remove impurities in the fly ash [16][17]. Hence, this research focuses on the characterization of greener-activated fly ash derived from coal combustion waste and its application as a sustainable adsorbent for reducing phosphate contamination in Donan River water sample.

## Materials and Methods

### Materials

*X-Ray Diffraction* (XRD) data for powder samples were collected on Philip Analytical X-Ray B.V., Shimadzu IRPRestige-21 spectrophotometer in the wave number range of 4000-400 $cm^{-1}$ , X-Ray Fluorescence (XRF) analysis was performed using a *Bruker S8 Tiger* sequential wavelength-dispersive spectrometer equipped with a Rh anode X-ray tube, operating at 4 kW for multi-elemental analysis of solid samples Mortar, Oven (*GallenKemp*), *Furnace* (*Vulcan*$^{TM}$ *seri A-300*), 200 Mesh-Sieve, *Hotplate Magnetic Stirrer* (Rexim RSH-IDR As One), *Magnetic Stirrer* (Spinbar), *Sibata Waterbath Shaker* (WS-120), analytical balance (*Mettler AE 200*), three neck flask with condenser, water pump, mercury thermometer, desiccator, flash point determination device (Clevand BBS product type BAP-243), and other research glassware according to work procedures.

The materials used include fly ash obtained from PLTU Karangkandri Cilacap, Donan river water samples, NaOH 1M, distilled water, aquabides, filter paper, High Density Polyethylene (HDPE) 1000ml.The tools used in this research include an oven, 250 mesh sieve, erlenmeyer glass, beaker, measuring cup, analytical balance, watch glass, pH meter and spatula, pipette, volume pipette, grinder, magnetic stirrer, and furnaces.

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### Sampling and analysis for the presence of phosphate ions

Based on the preliminary analysis showing phosphate contamination in the Donan River, further research was conducted to explore the potential of fly ash as an adsorbent material. Donan River water samples were taken from the three most polluted points[18] . In the next stage, 1 kg of coal fly ash is washed using distilled water, then in the oven for 180 minutes at a temperature of $100^{o}$C [19] . Testing for the presence of phosphate ion was carried out using the Spectrometry method at BKLPP Yogyakarta.

### Fly ash adsorbent activation

Activation of coal fly ash is carried out by first sieving the fly ash with a 250 mesh sieve to obtain a homogeneous particle size, then soaking it with NaOH[20] . Next, the fly ash dried and heated in a furnace at a temperature of $550^{o}$C[21].

The mixture then filtered and washed with aquabides until the filtrate was neutral (pH=7) [22]. The activated fly ash (AFA) solid then dried at room temperature. The coal fly ash adsorbent is ready to be used and characterized by FTIR, XRD and XRF to determine its constituent structure [22]. The characteristics of fly ash before and after activation was carried out using FTIR, XRD and XRF methods at Gadjah Mada University, Yogyakarta

### Testing the effectiveness of fly ash and the effect of variations in its mass on the adsorption of Donan River water samples contaminated with metal phosphate ions

The test begins by taking 500 ml of each metal-contaminated Donan River water sample solution, adding a buffer solution at the optimum pH, then adding activated fly ash with mass variations of 50, 100, 150 and 200 grams. The mixture was stirred for 60 minutes at room temperature, then filtered and the filtrate obtained was analyzed to determine the reduction in phosphate ion concentration using spectrometry. For the control solution at each mass variation, the same procedure was carried out but without the addition of adsorbents [23]. All of the method can be showed in Figure 1.

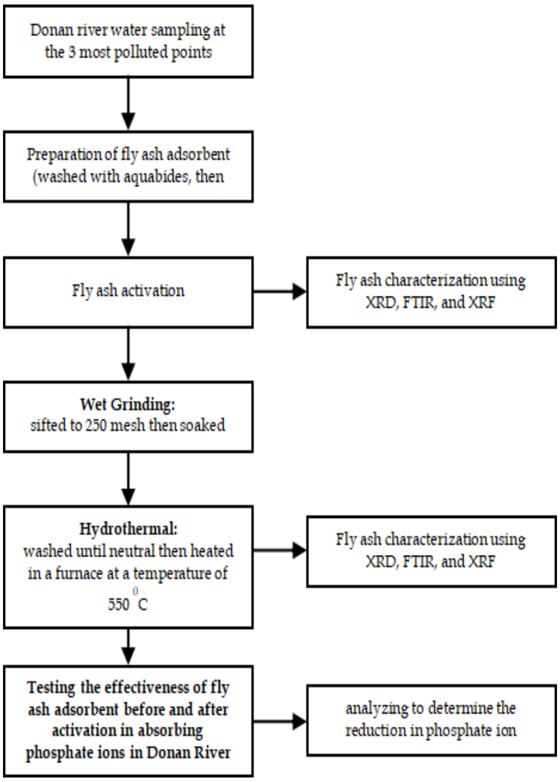

**Figure 1.** Diagram of the method

## Results and Discussions

### Analysis of Phosphate Pollution Levels in the Donan River

The study began with sample collection at several sampling points along the Donan River. The locations for monitoring the water quality of the Donan River were as follows: a natural water source at a location with minimal or no pollution (Point 1), polluted water sources at locations affected by waste contamination (Points 2 and 3), and the area where the river flows into the sea.

In this case, the natural water source was sampled at Point 1, located at 7°43'43.59"S, 108°59'15.99"E (Saliwangi Village, Tritih Kulon, North Cilacap). The polluted water sources were sampled in the industrial and urban areas of Cilacap, specifically at Points 2 and 3, located at 7°42'43.11"S, 108°59'26.29"E and 7°40'43.15"S, 109°0'8.55"E, respectively. The water samples were tested at BPPTKL Yogyakarta. The sampling results from these three points are shown in Figure 2.

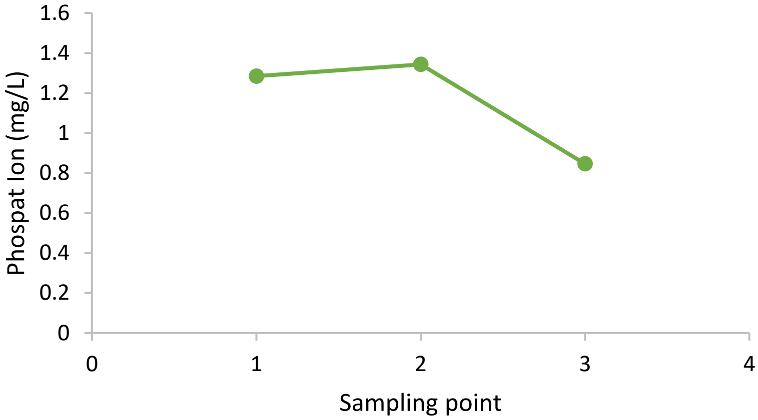

**Figure 2.** The graphic of contamination phosphate in three sites of Donan River

From the data, it can be observed that phosphate ion pollution in the Donan River in sampling point 1 count 1.285mg/L, point 2 showed 1.343 mg/L and point 3 showed 0,846mg/L, The phosphate ion in sampling point 2 has exceeded the permissible threshold for Class III water quality standards. Phosphate is an essential element for plant growth [11]; however, an increase in phosphate concentrations in rivers leads to excessive algae growth, which reduces dissolved oxygen levels in the water and causes ecosystem damage [24]. Some algae species produce toxins harmful to other aquatic organisms, and their proliferation can cover the water's surface, thereby reducing sunlight penetration and dissolved oxygen levels in the water. This highlights the importance of preventing phosphate pollution in aquatic environments [25] .

## Adsorbent Characterization

In this study, the focus is on managing phosphate waste. The researchers created synthetic phosphate waste with a concentration equivalent to the highest level of phosphate pollution in the Donan River, measured at 1.343 mg/L. the water sample adsorbed using fly ash activated through a greener activation method. This process involved soaking the fly ash to NaOH, followed by heating it in a furnace at 750°C [26].The mixture was subsequently filtered and washed with aquabidest until the filtrate reached a neutral pH (pH = 7) after the addition of NaOH. The activated fly ash solids were dried at room temperature. The prepared coal fly ash adsorbent was then was formed. The bending vibration of OH shifted from 1644.4 cm$^{-1}$ to 1640.4 cm$^{-1}$ after activation with NaOH. There was also a shift in the Si-O-Si bond from 1014.6 cm$^{-1}$ to 1096 cm$^{-1}$, and the

Fe-O-Si bond shifted from 693.4 cm⁻¹ to 697.6 cm⁻¹. This indicates that NaOH activation caused changes in the bonding structure of the fly ash components

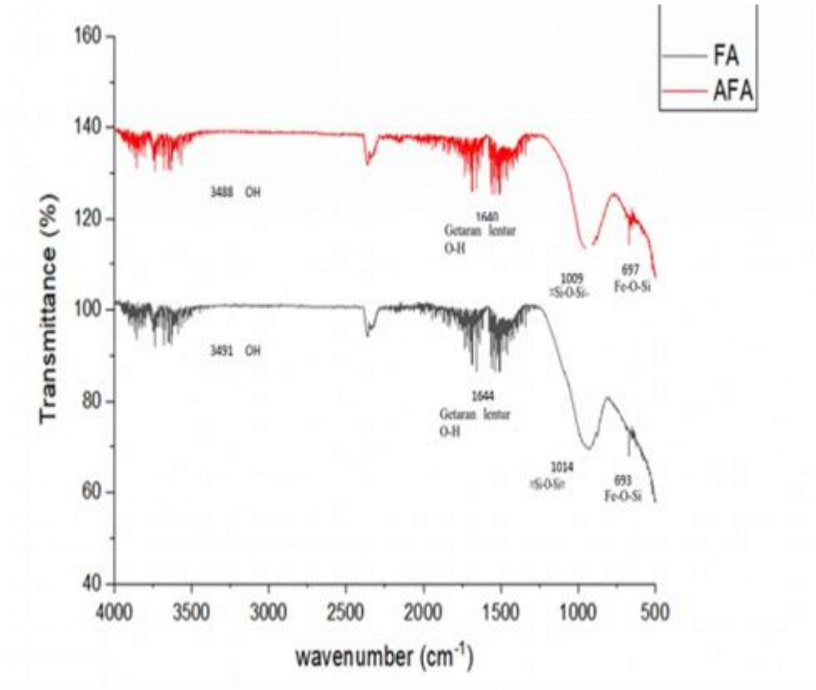

**Figure 3. FTIR spectra of fly ash before and after activation**

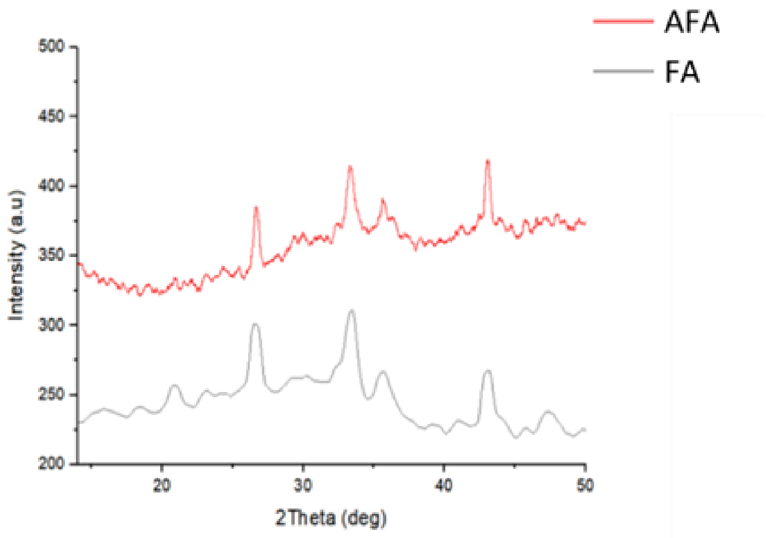

**Figure 4**. The XRD difraktograms before and after Greener Activation

Activation with NaOH alters both the physical and chemical structure of coal fly ash, increasing its surface area and porosity. This enhancement allows the material to adsorb more substances or contaminants [22]. However, an excessive NaOH concentration can reduce the adsorptive capacity of fly ash, making 1 M NaOH the most effective concentration for fly ash activation. The OH wavenumber was 3749.66 cm⁻¹ for raw fly ash (FA) and 3746.8 cm⁻¹ for the activated sample. The structural differences between NaOH and hydrothermal activation were analyzed using XRD, with the results can be seen in Figure 4.

The difraktograms of before and after still appear, specifically the peaks at 2θ 26.67° and 33.33° with d-spacing of 3.34 and 2.68, which correspond to quartz peaks. Meanwhile, the mullite peak appears at 43.042° with d-spacing of 1.828 and 2.297. This indicates that NaOH activation does not destroy the main structure of the fly ash components. NaOH activation decreases the crystallinity of the fly ash, making it more amorphous. This more amorphous structure has more active sites, thereby increasing the adsorption capacity of the fly ash for pollutants [27], [28]. And then the last characterization by XRF shows Table 2.

**Table 2.** The constituent compounds of coal fly ash

| Compounds | FA | AFA |
|---|---|---|
| $SiO_2$ | 33.46 | 26.97 |
| $Al_2O_3$ | 15.49 | 16.32 |
| $Fe_2O_3$ | 11.55 | 12.10 |
| CaO | 19.23 | 21.02 |
| MgO | 7.32 | 7.62 |
| $SO_3$ | 0.76 | 0.17 |
| $K_2O$ | 0.96 | 0.77 |
| $Na_2O$ | 1.98 | 3.06 |

The analysis using XRF shows that activation leads to a reduction in $SiO_2$. The alkaline hydrothermal process applied to fly ash can transform the main component of quartz and amorphous phases into zeolite-like material containing sodalite, mullite, and zeolite minerals [20]. The activation and hydrothermal treatment open up the active sites of the fly ash. The increae of active sites observed in AFA pimaly refers to the formation and exposure of basic surface functional groups (-O and -OH) and exchangeable cations resulting from alkaline hydrothermal activation. These sites enhance the surface reactivity toward anionic species such as phosphate ions, the transformation from amorphous $SiO_2$ and $Al_2O_3$ phases into zeolite-like aluminosilicate frameworks (containing sodalite and mullite) creates more porous structures with higher surface area and accessible active centers[29]. The amount of $Na_2O$ in the activated fly ash increased, providing evidence that the activation process has been successfully carried out.

After the fly ash was activated, it was contacted with synthetic water contain phosphate ion waste, where the sample water was adjusted to 1.343 mg/L. A 500 mL water sample was contacted with each ratio of activated fly ash (AFA) of 0.5, 1, 2, and 5 grams. The water and AFA adsorbent were mixed manually for 5 minutes, the results can be seen in Figure 5.

From the Figiure 5, it is clearly seen that by using AFA, 1.343 mg/L of phosphate ions can be adsorbed and leaving only 0.09 mg/L in the water sample. This indicates that the maximum adsorption occurs at 5 gram of fly ash which can adsorbed 93.2% of the phosphate ion contamination.

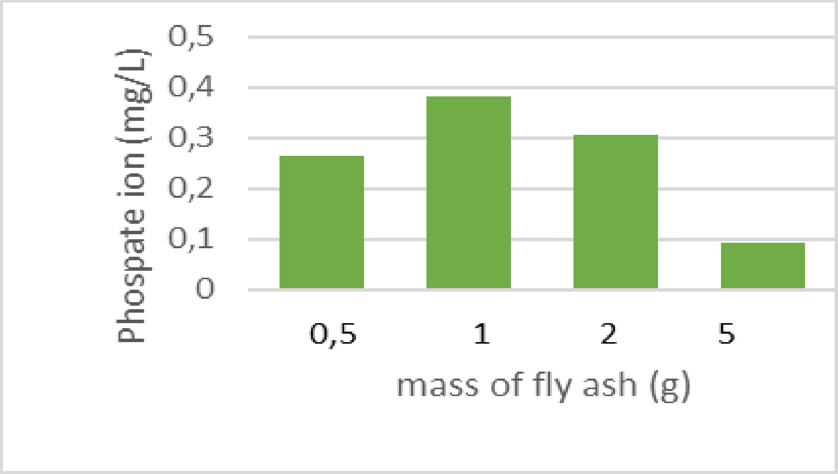

**Figure 5.** The adsorption of phosphate using AFA in various mass

**Table 3.** The calculation adsorption of phosphate using AFA in various mass

| Mass of Adsorbent (g) | Remaining phosphate (mg/L) | Adsorbed phosphate (mg/L) | Adsorbed phosphate (%) |
|---|---|---|---|
| 0.5 | 0.23 | 1.113 | 82.8 |
| 1 | 0.37 | 0.973 | 72.4 |
| 2 | 0.3 | 1.043 | 77.6 |
| 5 | 0.09 | 1.253 | 93.2 |

Other results show that the activated fly ash can remove 82,8% of phosphate ions with 0.5 grams of fly ash, 72,4 % with 1 gram of fly ash, and 77,6% with 2 grams of fly ash. The phosphate ion sample was present in 500 mL of waste water. The adsorption performance of AFA obtained in this study, which achieved 93.2% phosphate removal at an initial concentration of 1.343mg/L is comparable or even higher than previous studies. Earlier works utilizing acid base activation or fusion hydrothermal modification generally produced phosphate removal efficiencies ranging 80% to 90% but often involved the use of strong acid such as $H_2SO_4$ or HCl, which generated hazardous residues and require more complex processing conditions[30][31]. In contrast, the greener NaOH based activation method used in this research produced similar or better adsorption efficiency while minimizing chemical waste and energy input. Furthermore the enhanced adsorption capacity of AFA can be attributed to the increase formation of surface active sites and the transformation of amorphous $SiO_2$ and $Al_2O_3$ phases into zeolite like aluminosilicate structures with hgher porosity and reactivity[29]. These findings demonstrate that the activation process applied in this study is both effective and environmentally sustainable for phosphate removal from aquatic system.

## Conclusion

This research found that phosphate pollution in the Donan River has exceeded the permissible threshold for Class III water quality standards with amount 1.343mg/L. Then activated fly ash by greener activation can removed 82,8% phosphate ion with 0,5g fly ash, removed 72.4% phosphate ion with 1 gram fly ash, removed 77.6% phosphate ion with 2 grams of fly ash. And the best one is 5 grams of fly ash can remove 93.2% phosphate ion. However, the characterization of FTIR, XRD and XRF showed there were some differences of fly ash before and after the activation.

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