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RESEARCH PAPER

Removal of ammonium and phosphate in the synthetic wastewater using coal fly ash adsorbent

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Abstract. The main composition and availability of fly ash have made this waste potential to be used as an adsorbent for ammonium and phosphate removal. Each composition of fly ash may have different removal efficiency performances. The objective of this research was to investigate the optimal operation condition for ammonium and phosphate removal and to determine which source of fly ash has great adsorption potential. The experiments were carried out on various initial pH, the dosage of adsorbent, and the molar ratio of ammonium and phosphate. It was found out that the optimum removal efficiencies (i.e., 8–16% of ammonium and 16–75% of phosphate) were acquired at pH 8, the molar ratio of 7:1, and using 4.5 g of fly ash. Among five different sources, Punagaya fly ash exhibits the highest potential, in which the adsorption of ammonium and phosphate reached 7.17 mg/g and 19.50 mg/g, respectively.

Keywords: adsorbent; ammonium; efficiency removal; fly ash, phosphate

1. Introduction

The waste from the coal combustion process increases with the increase of electricity demand. Overall, the waste can be divided into two categories, i.e., fly ash and bottom ash (Slamet & Imas, 2017). Fly ash is the main by-product of the coal combustion process. Due to its small particle size (diameter ranging from >0.15 mm to <0.002 mm), fly ash can be carried on by flue gas stream and then collected with filtering devices (Chen et al., 2018; Lu et al., 2009).

Fly ash contains small amounts of heavy metals (i.e., Pb, Hg, and As). However, in large quantity, it may create major problems to human health and the environment (Ahmaruzzaman, 2010; Borm, 1997; Feng et al., 2018). Prolong exposure to heavy metals may cause minor to severe illness, such as abdominal pain, respiratory disease, cardiovascular disease, genes mutation, skin and lung cancer, to comatose (Munawer, 2018). Moreover, the heavy metals may leak into the soil,

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surface water, and groundwater if not properly collected, stored, or disposed (Basappa & Dikshit, 2012; Munawer, 2018; Sherly & Kumar, 2011).

Fly ash is potential to be used as a low cost adsorbent because of its characteristic and availability in large quantity. The main chemical compositions of fly ash are silica (60–65%), alumina (25–30%), and ferric oxide (6–15%) (Ahmaruzzaman, 2010). Additionally, it has 0.989–1.5 g/cm^3 of bulk density (Kishor et al., 2009; Kumar et al., 2017), 50–60% of porosity, 35–40% of water binding capacity, negatively charged surface area, and small particle size that are beneficial to be used in gas or wastewater treatment (Ahmaruzzaman, 2010).

Previously, fly ash has been applied as an adsorbent of ammonium and phosphate in wastewater (Niu et al., 2017; Ragheb, 2013). Noteworthy, the excess of ammonium and phosphate may enhance the eutrophication process and decreasing the oxygen level in the water. The reduction of oxygen levels eventually will interfere the aquatic life and make undesirable changes in the ecosystems (Larasati & Notodarmojo, 2014). An example of industry that generates wastewater high in ammonium and phosphate concentration is the fertilizer industry (Fitriana & Warmadewanthi, 2016; Kusuma, 2019). Adsorption is one of the methods that can be used to remove ammonium and phosphate in the water. This method has a high affinity for ammonium and phosphate. It also has high removal efficiency, simple application and operation, and environmentally friendly (Uğurlu & Karaoğlu, 2011).

The performance of the adsorption process is affected by several factors such as pH, the mass of the adsorbent, and the concentration of pollutants (Niu et al., 2012). Previous research by Hermassi et al. (2017) described that fly ash could be used as a reactive sorbent of phosphate and has the potential to be further used as plantation media. In this study, the optimum adsorption of phosphate (i.e., 59.5 mg $P\text{-}PO_4/g$) occurred at pH 8. Zhang, et al. (2011a) also stated that the optimum pH for ammonium removal is pH 8. Conversely, Ji et al. (2013) revealed that pH 7.5 could achieve an optimum removal of ammonium and phosphate, particularly at an initial ammonium concentration of 100 mg/L and with fly ash adsorption capacity of 14.63 mg/g (Niu et al., 2012). The adsorption method worked by accumulating ammonium and phosphate on the surface of the adsorbent, so that the saturated adsorbent (i.e., fly ash) can be utilized further as a nutrient-enriched plantation media.

In this study, the adsorption performance of fly ash was investigated for the removal of ammonium and phosphate contained in the synthetic wastewater. The experiment was carried out on various condition of pH, fly ash dosage, and the molar ratio of ammonium and phosphate. This study aims to determine the source of fly ash that could provide optimum adsorption on ammonium and phosphate, as well as to investigate the best operation condition for ammonium and phosphate removal.

2. Material and methods

2.1. Material

Raw fly ash was collected from five power plants locations in Indonesia, i.e., Punagaya (South Sulawesi), Nagan Raya (Aceh), Jeranjang (West Nusa Tenggara), Labuhan Angin (North Sumatera), and Suge (Bangka Belitung). Prior to the experiment, each fly ash was sieved through a 200-mesh sieve before further used as an adsorbent.

Synthetic wastewater containing ammonium and phosphate was prepared by dissolving potassium dihydrogen phosphate (KH_2PO_4) and ammonium chloride (NH_4Cl) (analytical grade) into distilled water. Meanwhile, sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) were used for pH adjustment.

2.2. Fly ash characterization

The chemical characterization of fly ash was done by X-ray fluorescence (XRF, PAN-analytical MiniPal4 Spectrophotometer). While, morphological structure and element of fly ash were identified with scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX, HITACHI FLEXSEM 1000). Both XRF and SEM-EDX were operated at 14 kV, in which Helium was used as the medium.

2.3. Experimental set-up and analyses method

The adsorption of ammonium and phosphate was carried out using batch mode in 500 mL beaker glass. In each experiment, 300 mL of ammonium and phosphate solutions were agitated at a constant rate of 180 rpm for 150 minutes. The selection of time and agitation speed is based on the best outcome obtained from previous researches (Baraka et al., 2012; Niu et al., 2012; Ragheb, 2013). The effect of pH on the removal of ammonium and phosphate was investigated at pH 7, 8, and 9. The pH was kept constant during the study by adding 2N NaOH or 2N H_2SO_4. In this study, the ammonium and phosphate molar ratios that were used were 1:1 (based on the initial concentration of ammonium and phosphate in the synthetic wastewater, i.e., 100 mg/L and 533 mg/L, respectively), and the molar ratio of 7:1 (based on real fertilizer industry wastewater, which has ammonium and phosphate concentration of 780 and 533 mg/L, respectively). The effect of pH on the adsorption process was evaluated on each molar ratio. Meanwhile, the effect of adsorbent dosage was carried out using 4.5 and 7 g mass variants, at molar ratio of 7:1 and pH 8. In the end of each experiment, the supernatant was kept steady for 1 h to promote settling. The precipitates were then filtered and dried in the sunlight.

Prior analyses, supernatant were taken and diluted several times to determine the residual ammonium and phosphate. For example, at the molar ratio of 1:1, the sample was diluted 100 and 625 times for ammonium and phosphate analyses, respectively. While at molar ratio of 7:1, the sample was diluted 1000 and 625 times according to its concentration. The ammonium and phosphate concentrations were measured by UV spectrophotometer at a wavelength of 400 nm and 675 nm, respectively, according to the Nessler method. The obtained absorbance values were then inputted to the calibration curve of ammonium and phosphate, based on the equation as follows, i.e., $y = 0.2138x - 0.009$ (for ammonium), and $y = 0.7522x - 0.0029$ (for phosphate); in which y is the absorbance, and x is the concentration. The concentration obtained from those equations were then multiplied by each diluent factor to get the actual concentration. Moreover, in order to understand the chemical composition and morphological structure, each precipitate was analyzed with XRF and SEM EDX.

The removal efficiency and the amount of ammonium and phosphate adsorbed were calculated according to the following equations (1) and (2).

$$\eta = \frac{C_o - C_e}{C_o} \times 100\% \tag{1}$$

$$q_e = \frac{C_o - C_e}{m} \times \frac{V}{1000} \qquad (2)$$

Where: η is the removal efficiency (%), C_o is the initial concentration of ammonium and phosphate (mg/L), and C_e is the concentration of ammonium and phosphate at equilibrium (mg/L), q_e is the amount of ammonium and phosphate adsorbed (mg/g), m is the mass of fly ash (g), and V is the volume of the solution.

3. Result and discussion

3.1 Characterization of fly ash

The chemical composition of each fly ash is presented in Table 1. As observed, all fly ashes mainly consist of silica and aluminium oxide components, with the percentage ranging from 31.81–45.24% and 17.66–24.59%, respectively. Compared to other fly ash, Punagaya has shown the best effectivity for ammonium and phosphate removal. The morphological structures and elements of Punagaya fly ash can be seen in Figure 1. According to Figure 1 (a), Punagaya fly ash has characteristic of irregular shape particles and porous surfaces. However, after treatment, these particles were agglomerated and the nucleation appeared on particle surfaces.

Table 1. Chemical composition of fly ashes

Component	**Content (wt %)**				
	Punagaya	**Nagan Raya**	**Jeranjang**	**Labuhan Angin**	**Suge**
SiO_2	37.94	45.24	31.81	47.33	36.74
Al_2O_3	17.66	24.59	19.77	26.32	23.25
Fe_2O_3	19.55	5.9	12.14	6.25	13.55
CaO	11.05	6.84	9.41	5.03	17.6
MgO	4.65	3.12	3.76	2.89	2.26
Na_2O	0.5	1.56	0.71	2.23	0.37
K_2O	0.83	0.86	1.08	0.81	0.31
TiO_2	0.77	0.74	0.84	0.84	1.06
MnO_2	0.3	0.13	0.15	0.11	0.62
Cr_2O_3	0.01	0.01	0.01	< 0.01	< 0.01
P_2O_5	0.11	0.36	0.15	0.3	0.04
SO_3	5.04	2.31	1.83	1.52	1.33

3.2 Effect of pH

The pH value is an important parameter that affects the removal efficiency of ammonium and phosphate in the solution. The effect of pH on the adsorption process was determined at ammonium and phosphate molar ratio of 1:1 and 7:1, using 4.5 g of fly ash. The results are presented in Figure 2 and 3. Based on these figures, it is confirmed that Punagaya fly ash is more potential to be used as a removal agent for ammonium and phosphate in the solution compared to other fly ashes.

As seen in Figure 2, the removal efficiency of phosphate decrease with the increase of pH level in the solution. In addition, the removal of ammonium is not detected at all on pH 7 and 8. At pH 7, the maximum phosphate removal (69%) is obtained on Punagaya fly ash, whereas the minimum removal (8%) is acquired on Labuhan Angin fly ash at pH 7. When pH is increased to pH 9, the removal efficiency on Punagaya fly ash decreased to 45%, while the minimum efficiency on Labuhan Angin decreased to 2%. In addition, figure 2 shows that pH 7 and 8 does not cause the removal of ammonium. The undetectable removal of ammonium is possibly due to the presence of competitive ion such as K^+ that take place on the adsorption site of ammonium on the adsorbent surface (Niu et al., 2012; Zhang, et al., 2011a). In this molar ratio, the optimum removal of ammonium is achieved at pH 9, which is in the range of 10–37%.

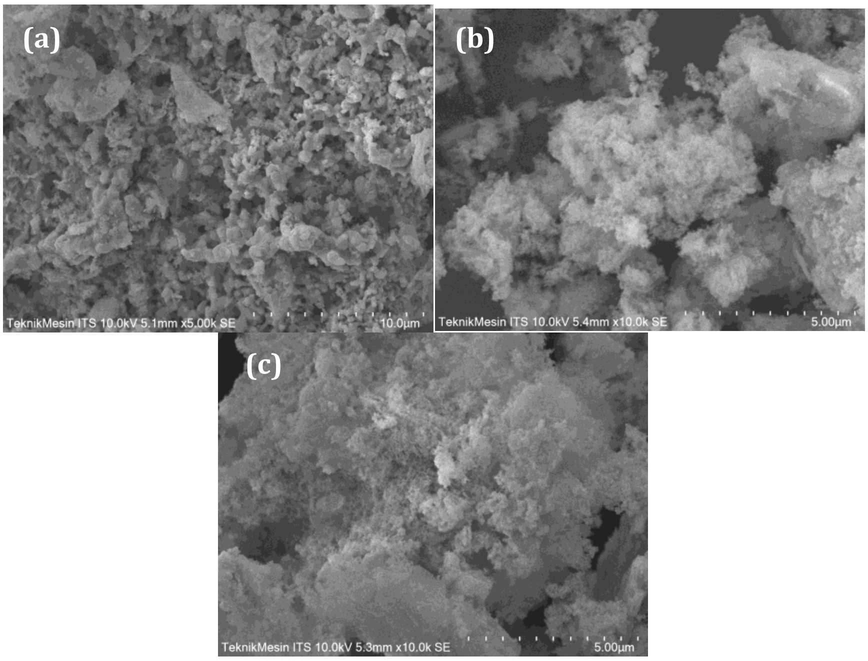

Figure 1. Morphological structure of Punagaya fly ash (a) before removal process; (b) at molar ratio of 1:1 and 4.5 g of fly ash dose; (c) at molar ratio of 7:1 and 4.5 g of fly ash dose

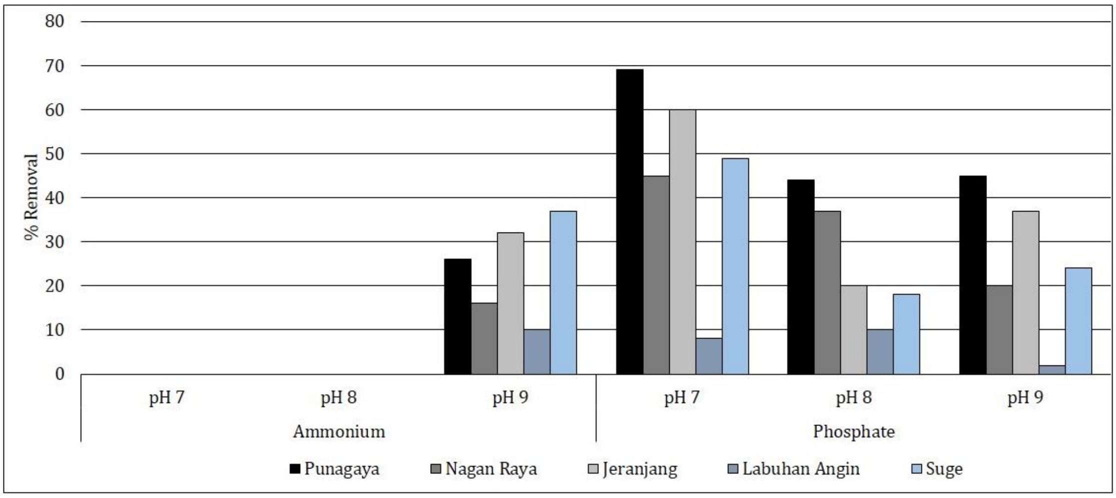

Figure 2. Effect of pH on the removal of ammonium and phosphate at molar ratio of 1:1

Moreover, Figure 3 describes that the optimal phosphate removal efficiency at molar ratio of 7:1 is obtained at pH 7 using Punagaya fly ash. In this pH level, the efficiency reaches 71%.

However, the percentage drops to 35% when pH is increased to 9. Regarding ammonium, the removal efficiency at pH 7 could only be detected on Punagaya and Labuhan Angin fly ashes. However, as pH is increased to 8, the removal of ammonium occurred on all fly ash variants. The removal efficiency of ammonium is even higher when pH is increased to 9. At the higher ammonium concentration, the driving force between ammonium ions and the surface of the adsorbent is increased, resulting in good removal efficiency at pH 7, 8 and 9.

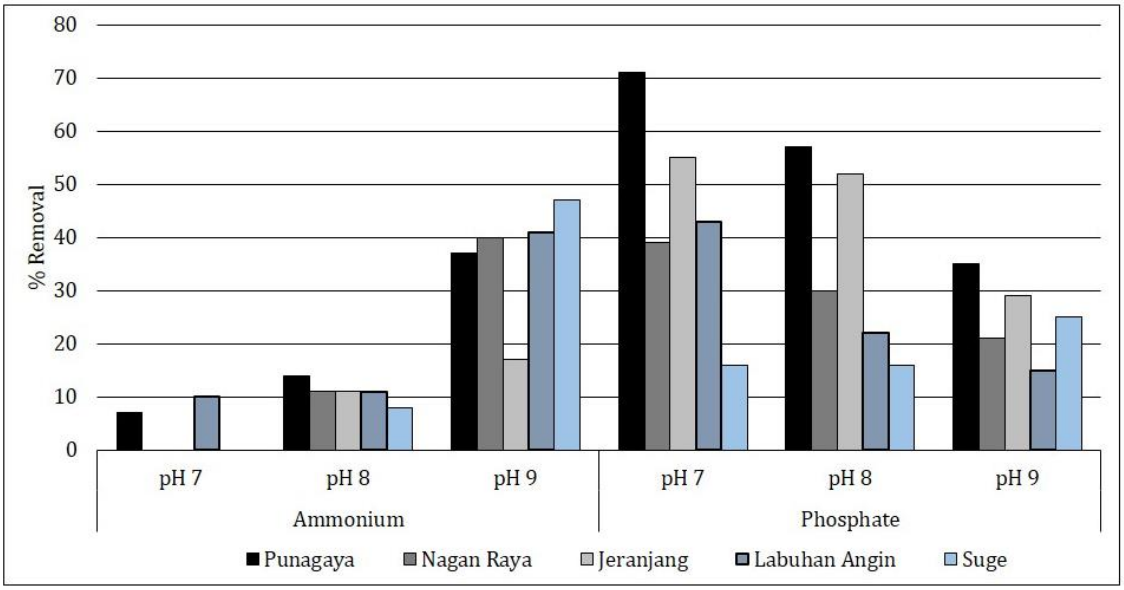

Figure 3. Effect of pH on the removal of ammonium and phosphate at molar ratio of 7:1

Ammonium and phosphate ions have different charges, i.e., ammonium ion has a positive charge (as cations) and phosphate has a negative charge (as anions). The pH value has a significant influence on the determination of species compounds and the surface charge of adsorbent. For example, H_3PO_4 is dominant at pH less than 2. Other species such as $H_2PO_4^-$ and HPO_4^{2-} are more likely to exist at pH 2–11, whereas PO_4^{3-} is dominant at pH >11 (Guaya et al., 2015). Meanwhile, at pH < 9, nitrogen is readily available as ammonium. However, as pH increases to more than 11, ammonium compounds are completely dissappear and are converted into ammonia (NH_3). Since fly ash has an alkaline pH, it is more likely to have negativecharge surface area at a high pH level and can be used to precipitate or adsorb material (Hermassi et al., 2017). Negatively charge adsorbent surface is beneficial for cation adsorption and induced ammonium removal by ion exchange and electrostatic attraction (Feng et al., 2020). The electrostatic force occurs due to attractive or repulsion force between adsorbent and adsorbate surface charge (Feng et al., 2020). As observed, ammonium removal is mainly dominated by electrostatic attraction, whereas phosphate removal is mainly occurs through ligand exchange with a hydroxyl group, precipitation with Ca^{2+} and Mg^{2+}, or complexation (Feng et al., 2020).

The effect of pH on ammonium can be described further. At pH > 9, ammonium ion is converted to a volatile non-ionized NH_3 (Myllymäki et al., 2020). It indicates that at pH 9 and molar ratio of 1:1 and 7:1, ammonium tends to evaporate than being adsorbed to the fly ash. This theory is in agreement with EDX results that do not show any nitrogen element at pH 9. It is also explained that the higher removal efficiency of ammonium in pH 9 is mainly caused by the evaporation of nitrogenous compounds rather than the adsorption process.

Table 2. Element composition in Punagaya fly ash before and after treatment

Element	Content (weight %)		
	Before treatment	After treatment	
		a	b
O	34.99	42.39	38.1
Mg	3.24	2.81	2.87
Al	8.10	7.50	6.57
Si	13.89	13.16	12.14
S	4.23	-	-
Ca	10.37	9.76	11.18
Fe	25.17	14.63	20.88
C	-	2.68	-
P	-	3.93	0.14
Na	-	1.53	0.88
K	-	1.61	1.47

Notes: (a) removal of ammonium and phosphate at molar ratio of 1:1 using 4.5 g dose of Punagaya fly ash; (b) removal of ammonium and phosphate at molar ratio of 7:1 using 4.5 g dose of Punagaya fly ash.

Table 2 clarifies that before treatment, Punagaya fly ash has no phosphorus content. However, after treatment there is 0.14–3.93% phosphorus traced in the fly ash particles, in which higher phosphorus content is achieved on molar ratio of 1:1. Moreover, at those molar ratios, the nitrogen element is undetectable.

According to previous studies, optimum removal of ammonium and phosphate can be achieved at pH 7–9. Niu et al. (2012) reported that the percentages of ammonium removal reached an optimum level (up to 85%) at pH 5–8 but then decreased to 45% when experiments were carried out at pH 10. Moreover, Ragheb (2013) evaluated the removal performance of phosphate at pH 3–12. It was shown that the maximum removal of phosphate (i.e., 83–69%) could be obtained at pH 7–9. Another study by Cheng et al. (2017) obtained 92.13% of ammonium and 90.3% of phosphate removal at pH 8 on the initial ammonium and phosphate concentration of 708.4 mg/L and phosphate 21.62 mg/L, respectively. Lastly, Hermassi et al. (2017) also reached optimum adsorption capacity at pH 8, i.e., 54.1 mg/g, and then the capacity decreased significantly to 19.1 mg/g when pH was increased to pH 9.

3.3 Effect of adsorbent dosage

The removal of ammonium and phosphate was carried out by adding 4.5 and 7 g fly ash dose into the solution. The experiments were conducted at molar ratio of 7:1 and at pH of 8. The effect of adsorbent dosage on the removal efficiency of ammonium and phosphate are shown in Table 3.

It was observed that a larger dosage of adsorbent did not provide a significant increase in removal efficiency. Based on the Table, the maximum removal of phosphate (62%) is achieved by adding 7 g dose of Punagaya fly ash. While at the same dose, the lowest removal of phosphate (21%) is obtained from Labuhan Angin fly ash. These results confirm that the higher dose of fly ash added, the more active sides are available, which then result in the more optimum elimination process (Zhang et al., 2011b).

Table 3. Effect adsorbent dosage on efficiency removal of ammonium and phosphate

Fly ash source	Initial Concentration (mg/L)		Removal (%)			
			4,5 g		7 g	
	NH_4^+	PO_4^{3-}	NH_4^+	PO_4^{3-}	NH_4^+	PO_4^{3-}
Punagaya	780	533	14	57	7	62
Nagan Raya			11	30	17	50
Jeranjang			11	52	10	58
Labuhan Angin			11	16	1	21
Suge			8	22	10	44

The ammonium removal is higher when 4.5 g dose of fly ash is added into the solution. The highest removal of ammonium and phosphate is acquired from Punagaya fly ash. Conversely, Suge and Labuhan Angin fly ash obtain the lowest removal of ammonium and phosphate efficiencies, i.e., 8 and 16%, respectively. The optimum ammonium and phosphate removal are achieved when 4.5 g dose of adsorbent is added into the solution.

Figure 4 illustrates the concentrations of ammonium and phosphate ions that can be adsorbed by 4.5 g dose of fly ash. Based on this figure, the effectiveness of fly ash to adsorb phosphate ions can be arranged into the following order: Punagaya >Jeranjang >Nagan Raya > Suge > Labuhan Angin. While for ammonium removal, the effectiveness of fly ash can be sorted by the following order: Punagaya > Labuhan Angin > Jeranjang > Nagan Raya > Suge.

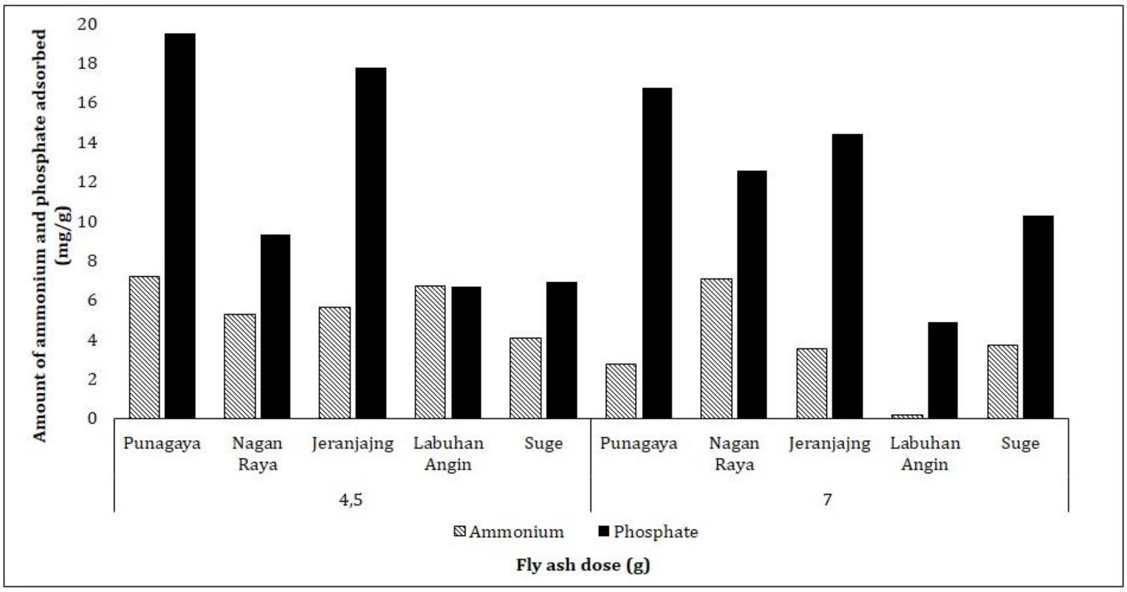

Figure 4. The ability of each fly ash source to remove ammonium and phosphate at different masses

It can be seen that Punagaya fly ash has the highest adsorption of ammonium and phosphate ions, i.e., 7.17 mg/g and 19.50 mg/g, respectively. At this concentration, the residual of ammonium and phosphate remains in the solution are 640 mg/L and 218 mg/L, respectively. In comparison, Suge fly ash has the minimum adsorption of ammonium ion, whereas Labuhan Angin fly ash has the minimum adsorption of phosphate ion, resulting in higher residual of ammonium and phosphate, i.e., 673 and 535 mg/L, respectively.

4. Conclusion

In this study, fly ashes from five location coal power plants were evaluated to understand its performance on removing ammonium and phosphate contained in synthetic wastewater. The results show that optimum efficiencies were achieved on pH 8 using 4.5 g dose of fly ash. The removal percentage of ammonium and phosphate were 14% and 57%, respectively. It is concluded that at molar ratio 1:1, the removal efficiency of ammonium and phosphate were low compared to the molar ratio of 7:1. Moreover, among five fly ashes that have been evaluated, Punagaya has the best adsorption efficiency. The ammonium and phosphate ions that can be absorbed by Punagaya fly ash were 7.17 mg/g and 19.50 mg/g, respectively.

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