NPK Fertilizer with Slow Release Fly Ash

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ABSTRACT

Fly ash is a residual solid combustion carried by gas. The content of fly ash is similar to zeolite. Hence, fly ash can be used instead of zeolite for fertilizer carrier. Slow release test result was revealed that N element has higher release level. The activity plant test was conducted on chili plant using parameter variation of fertilizer composition and plant height. Based on the research result, triple phosphate-fly ash fertilizer in comparison 2:1 gave the best result.

Key word: fertilizer, slow release, fly ash, NPK, granule

INTRODUCTION

Clean water availability is a basic needs and becomes a limited resource on the earth due to industrial include fertilizer which produce residual products that can pollute the water. In some areas, where water is abundant, clean water can be limited due to people behavior that produces pollutants. Fly ash can be a workaround to reduce residual from fertilizer [1].

Fly ash is residual solid combustion carried along with exhaust gases and captures by air controller. Fly ash contains many silica and alumina mineral which both have pozzolan properties that have no adhesive properties. Fly ash compose of Al₂O₃, SiO₂, Fe₂O₃, CaO, MgO, Na₂, and SO. By using fly ash granules, silica can react with water and afford adhere properties [2].

Fly ash can be applied for construction. It is also used as a mineral in PCC (Precipitated Calcium Carbonate) for almost 60 years which is the largest use of single fly ash. This also can be used as a material for Portland cement production and a mixture of Portland-pozzolan cement. In addition, fly ash has also been used as a replacement mineral filler on asphalt mixture which consists of particles less than 0.075 mm (200mesh). It used to fill the void in a paving mixture and increase binding cohesion (cement asphalt) and stability of asphalt mixture. Therefore, fly ash has potential for daily life [3].

Srivastava (2016)[4] revealed that fly ash can be used as a potential nutrient supplement for degraded soil to overcome the solid waste. Fly ash is applied in degraded soil to achieve enhanced fertility in order to minimize the accumulation of toxic metal in plant and without affecting soil quality.

Zeolite is a mineral aluminosilicate hydrate. Zeolite has a unique structure by the presence of alumina groups (AlO₄) and silica group (SiO₄) interconnected by oxygen atoms form a three-dimensional framework [5]. Fly ash and zeolite has similar content. Both zeolite and fly ash contain Al₂O₃, SiO₂, Fe₂O₃, CaO, and MgO which can be as super-absorbent with
low cost of production. Zeolite usually used as fertilizer carrier and also fly ash according to the theory[6].

Before fly ash is used, it requires to be activated with sodium hydroxide. The reaction of a solid aluminosilicate using a highly concentrated aqueous alkali hydroxide produces a synthetic alkaline aluminosilicate material. It depends on the raw materials and processing condition used, alkali-activated binders may feature high compressive strength, low shrinkage, acid and fire resistance, and low thermal conductivity [7]. The alkali activation of fly ashes produces the dissolution of the glassy component (aluminosilicate) and formation from dissolved species of amorphous alkaline aluminosilicate gels. The amorphous compound formed displays the same characteristics of zeolites precursors during alkaline activation of metakaoilin[8]

Fertilizer that mostly applied nowadays is the type of NPK, which is macro element for plant. However, recently the manufacture of NPK fertilizer modified with granules form due to powder or liquid form can easily solved in water prior to applying to the plant. It can cause growth of water hyacinth (Eichhornia crassipes) in the water because of high concentration of fosfor[9].

Fertilizer using slow release form can optimize nutrient absorption to the plant with time and amount that needed by plant. Fly ash slow release can maintain nutrient in soil. Moreover, slow release fertilizer more efficiently used than using conventional method[10].

**EXPERIMENT**

**Chemicals and instrumentation**

Materials used are grey fly ash from Paiton Indonesia. Urea, Potassium Chloride (KCl), triple phosphate, sodium hydroxide 48%, ethanol 70%, distilled water, molasses, and chili plant. Instrument used are XRF and XRD brand: Analytical, type: Expert Pro.

**Procedure reaction**

**Preparation and fly ash activation**

100 grams of fly ash in a beaker glass 500 mL was added by 300 mL of sodium hydroxide 48% by varying concentration of 4M and 1M and stirred for 6 hours. The precipitate was filtered and washed using distilled water until filtrate pH reached 7. The precipitate was dried in an oven under 100°C.

**Preparation of NPK fly ash slow release**

Each of urea, triple phosphate, and potassium chloride fertilizer was mixing with fly ash by ratio of 1:1, 1:2, and 2:1 in 250 mL beaker glass. Then the mixture was added by ethanol 70% 5 mL and stirred for 2 hours. The precipitate was filtered and dried in an oven at 80°C.

**Granulation**

NPK fly ash slow release fertilizer on 250 mL of beaker glass was added a drops of molasses. The mixture was shaken until granules are formed. Granule dried on an oven at 100°C.
NPK Fertilizer Activity Test Fly Ash Slow Release on Chili Plant

5-days-old chili plants grown in tray and fertilized in the soil. After one week, chili plant transferred to polybag and fertilized again. Measurement of height and number of leaf’s plant is conducted every once a week starting from planting of chili plant.

Fly ash Characterization with XRF Analysis

XRD analysis was performed on grey fly ash and fly ash activated in concentration of 4 M and 1 M samples to determine the oxide composition and presence of compounds with specific structure in samples at an angle 10°-90°. This analysis was conducted in Mineral Laboratory and Advance Material Central Laboratory, University of Malang.

Testing of slow release of NPK Fly Ash Fertilizer

The slow release test of NPK fly ash fertilizer was conducted by 2 methods, such as Atomic Absorption Spectrometry (AAS) and spectrophotometry using Kjeldal-Nessler solvent. This test was undertaken by varying immersion solution for 6 hours and 12 hours in each concentration of 1 M and 4 M.

RESULT AND DISCUSSION
XRF Characterization

Primary components of fly ash are silica (SiO$_2$), alumina (Al$_2$O$_3$), iron oxide (Fe$_2$O$_3$) with varying amounts of carbon, calcium, magnesium, and sulphur. There are two general classes of fly ash, class F (contain less than 7 wt.% of CaO) and class C (contains more 5-30 wt.%). In general, fly ash has a hydrophilic surface and porous structure [11].

![Figure 1. XRF of grey fly ash non activation](image-url)
Figure 2. XRF result of yellow fly ash non activation

Table 1. XRF result of grey and yellow fly ash

<table>
<thead>
<tr>
<th>Composition</th>
<th>Concentration (%)</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Grey</td>
</tr>
<tr>
<td>Fe</td>
<td>47.49</td>
</tr>
<tr>
<td>Si</td>
<td>22.6</td>
</tr>
<tr>
<td>Al</td>
<td>9.7</td>
</tr>
<tr>
<td>K</td>
<td>2.9</td>
</tr>
<tr>
<td>Ca</td>
<td>12.8</td>
</tr>
</tbody>
</table>

The highest component of both fly ash is Fe (47%). Si and Al component of grey fly ash has much higher concentration than yellow fly ash. The more of Si component may lead to increase the available cavities carrying fertilizer. K (2.9%) and Ca (12.8%) component on grey fly ash are less than yellow fly ash (12% and 29.4%). However, Ca and K is not major component needed by plant, therefore it only has little effect to the final result. Hence, in this research used grey fly ash as a carrier of NPK fertilizer.
Figure 3. Grey fly ash after activated by 4 M of sodium hydroxide

Figure 4. Grey fly ash after activated by 1 M of sodium hydroxide
Table 2. XRF Result of activated fly ash

<table>
<thead>
<tr>
<th>Composition</th>
<th>Concentration (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1M</td>
</tr>
<tr>
<td>Fe</td>
<td>54.49</td>
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<tr>
<td>Si</td>
<td>17.9</td>
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<tr>
<td>Al</td>
<td>7.4</td>
</tr>
<tr>
<td>K</td>
<td>2.58</td>
</tr>
<tr>
<td>Ca</td>
<td>11.1</td>
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Activation using sodium hydroxide was aimed to dissolve Si and Al components without dissolving metal contained in fly ash as micro unsure that required by plants. Based on the XRF result, there are Si and Al component dissolved in activation process. Before activation, composition of Si is 22.6%, but after activation it decreased to 17.9% and 18.8% in concentration of 1 M and 4 M, respectively. Al content before activation is 9.7%, after activation it decreased to 7.4% and 8.2%, in concentration of 1 M and 4 M, respectively. Fly ash pores clogged by Si and Al will open, indicated with decreased of Al and Si. Hence, the pores of fly ash are loose, lead to increase the carrying ability of fly ash. Activated fly ash has fast setting and rapid strength development properties and it is also used for the immobilization of toxic waste [12].

XRD Characterization

Figure 5. XRD of fly ash before activation and after activation
Based on theory, fly ash has quartz structure of SiO$_2$ (30.1%), mullite structure of Al$_6$Si$_2$O$_{13}$ (31.6%), and hematite structure of Fe$_2$O$_3$ (24.1%). According to the XRD results compared to JCPDS, fly ash comprised quartz form of Si, hematite and magnetite form of Fe, and calcite form of Ca.

Table 3. Comparison JCPDS of SiO$_2$ Quartz and XRD of Fly Ash

<table>
<thead>
<tr>
<th>JCPDS SiO$_2$</th>
<th>XRD Result Fly Ash Non Activation</th>
<th>XRD Result Fly Ash 1M Activation</th>
<th>XRD Result Fly Ash 4M Activation</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>Intensity 20</td>
<td>Intensity 20</td>
<td>Intensity 20</td>
</tr>
<tr>
<td>26.65</td>
<td>100</td>
<td>26.699</td>
<td>26.799</td>
</tr>
<tr>
<td>50.14</td>
<td>14</td>
<td>50.177</td>
<td>50.250</td>
</tr>
<tr>
<td>68.13</td>
<td>7</td>
<td>68.155</td>
<td>67.993</td>
</tr>
</tbody>
</table>

Table 4. Comparison JCPDS of Fe$_2$O$_3$ hematite and XRD of fly ash

<table>
<thead>
<tr>
<th>JCPDS Fe$_2$O$_3$</th>
<th>XRD result fly ash non activation</th>
<th>XRD result fly ash 1M activation</th>
<th>XRD result fly ash 4M activation</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>Intensity 20</td>
<td>Intensity 20</td>
<td>Intensity 20</td>
</tr>
<tr>
<td>33.15</td>
<td>100</td>
<td>33.393</td>
<td>33.494</td>
</tr>
<tr>
<td>35.61</td>
<td>71</td>
<td>35.674</td>
<td>35.677</td>
</tr>
<tr>
<td>43.52</td>
<td>3</td>
<td>43.118</td>
<td>43.212</td>
</tr>
<tr>
<td>62.45</td>
<td>30</td>
<td>62.691</td>
<td>62.757</td>
</tr>
</tbody>
</table>

Based on XRD result, there is no significant change of 20 between activation and nonactivation of fly ash. This shows that activated alkali does not change the crystal structure. Comparison of SiO$_2$ quartz and Fe$_2$O$_3$ hematite JCPDS and XRD of fly ash indicate the same value of 20 which means fly ash comprised SiO$_2$ with quartz structure and Fe$_2$O$_3$ with hematite structure. Ayanda (2012)[13] reported the diffractogram of fly ash showed that FA consists mainly of crystalline minerals mullite (Al$_6$Si$_2$O$_{13}$) and quartz (SiO$_2$) with large characteristic peak of quartz (SiO$_2$).

**Manufacture of Slow release fertilizer**

The activated fly ash was mixed by using potassium chloride, urea, and triple phosphate in ratio of 1:1, 1:2, and 2:1. The granulation done was conducted using molasses as adhesive. Fertilizer fly ash-urea granule has a different size and easily breakable in drying treatment. This is due to the texture of mixed fertilizer fly ash-urea is very dry, and also due to the
content of CO$_2$ inside urea. Thus, in the making of granules is not easy and need for compaction using hand.

The mixture of fly ash fertilizer using potassium chloride produces granule form which has different size. Physically it is not easily destroyed compared to fly ash-urea fertilizer. The texture of this fertilizer mixture is slightly wet, therefore when it mixed using molasses it is easier to be granule form.

The mixture of fly ash-triple phosphate produces a product that has a uniform shape when it is granulated. Physically, it is not easily broken. Texture of fly ash-triple phosphate is slightly wet. Hence, it was easy in process of granulation using a drops of molasses. This is due to the Ca content of triple phosphate (Ca(H$_2$SO$_4$)). Potassium content has high density properties. In addition, the content of phosphor and calcium has low solubility to water which caused this structure hard.

**NPK Fertilizer Activity Test Fly Ash Slow Release on Chili Plant**

Each fertilizer variation was applied to chili plant once a week which conducted one variation for six chili plants. The result of plant growth are obtained based on the high plant from week 1 to week 5.

![Graph showing plant height correlation](image)

**Figure 6.** Correlation between plant height and fly ash-triple phosphate 1M and 4M fertilizer for 5 weeks

Based on the difference of composition of triple phosphate fertilizer, fertilizer with 2:1 ratio using fly ash 4M has better average plant height of 16 cm. While, fly ash 1M gave the height of 14.750 cm. Ratio of 2:1 has better result due to the increasing number of phosphor element carried by fly ash, therefore, the elements will be more received by the plant which showed from the increase of plant height.
Based on the addition of the different fertilizer, fly ash-triple phosphate fertilizer has highest average height increase of 6.539 cm and 7.226 cm. The element of phosphor is a living cell compound associated with nucleic acid and adenosine three phosphate (ATP). Chili still applied in its growth phase, therefore, it needs phosphor to strengthen the stem and accelerate the growth of roots and cell division, while urea and potassium elements are not needed yet. Thus, plant growth with addition of triple phosphate become faster. It can be concluded that triple phosphate-fly ash fertilizer is successfully conducted in plant growth phase. It due to phosphor element is needed by plants during growth phase, therefore it has better test result.

**Slow release Test on Chili Plant**

**Figure 7.** Correlation between plant height and various fertilizer for 5 weeks

**Figure 8.** Slow release result of fly ash 4M in immersion for 6 and 24 hours
Figure 9. Slow release result of fly ash 4M in immersion for 6 and 24 hours

Fly ash 4M with immersion for 6 hours in aquadest, N element released 12.42 ppm, while P element released 0 ppm. However, fly ash with immersion for 24 hours, P element released still 0 ppm, while K element only released 0.14 ppm. In addition, slow release test for 1M with immersion for 6 hours in aquadest, N element easily release with 6.85 ppm, while P released 0 ppm. However, fly ash in 24 hours immersion, P element released 117 ppm, while K element only 0.11 ppm.

The lower concentration, it may lead to control easily the release of nutrients according to time and the number of plants required which can hold nutrients in the soil. Hence, fly ash can be applied as a good fertilizer carrier. The undetectable elements of release speed are due to the absence of fertilizer release in 6-24 hours. It signifies better release ability. Thus, P and K element have better release capabilities than N.

CONCLUSION

The activation of fly ash using sodium hydroxide can open or enlarge the pore size. 2:1 ratio of triple phosphate with 4M fly ash has better result due to the more phosphor element carried by fly ash. Thus, the elements received by plants are also increasingly shown from plant growth. The slow release test result indicated that N element has higher release level than P and K.

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