OPTIMIZATION OF THE USE OF FLY ASH AS AN ADDITIVE PORTLAND COMPOSITE CEMENT (PCC)

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ABSTRACT

Research has been carried out on the optimization of the use of Lahat Fly Ash as an Additive Portland Composite Cement (PCC) which aims to determine the optimum conditions for adding Lahat fly ash to produce cement with physical and chemical requirements in accordance with SNI 7064:2014 and discover the ideal composition of cement with fly additions Lahat ash from various cement compositions. The quality analysis in making PCC cement is chemically in the form of Insoluble Residue (IR), Loss of Ignition (LOI), and free lime (FCaO) as well as the quality of physics in the form of Blaine, setting time, and cement mortar compressive strength. The results obtained after the process of making PCC cement with the addition of 14% Lahat fly ash, namely PCC cement in optimum conditions with physical and chemical requirements in accordance with SNI 7064:2014 where the ideal composition of PCC cement manufacture is 14% Lahat fly ash, clinker 62%, 3% gypsum, 18% limestone, and 3% pozzolans in making PCC cement. Therefore, Lahat fly ash can be used as an alternative mixture in making PCC cement.

Keywords: Lahat fly ash, PCC cement, the chemical quality of cement, physical quality of cement
INTRODUCTION

In Indonesia, there are various kinds of energy sources that can be used for multiple purposes, one of which is coal. Coal is widely used by the industry and the Steam Power Plant (PLTU) as boiler fuel to produce steam as a heating medium or power plant because besides being able to save operational costs also the availability of coal is quite abundant [1]. In 2017 national coal production in Indonesia reached 461 million tons [2]. The use of fuel in the production process can harm environmental quality. The environmental impact caused is an increase in the amount of solid waste from the remaining coal combustion, which is categorized as toxic and hazardous waste [3]. From coal combustion, solid pollutants can be produced in the form of ash, one of which is fly ash with an amount of about 80 - 90% of the total ash produced [4].

Fly ash is one of the waste products from coal combustion in a steam power furnace, which is round, smooth, and pozzolanic. The content of fly ash consists mainly of silica oxides (SiO2), aluminum (Al2O3), iron (Fe2O3), and calcium (CaO), as well as small amounts of potassium, sodium, titanium, and sulfur [5]. From the content, fly ash can be used as a mixture of cement, because it has a compound component of SiO2, Al2O3, Fe2O3 which is the main compound needed in cement other than CaO. The composition of the content of SiO2, Al2O3, Fe2O3 is similar to pozzolanic material which makes it possible to be used as a mixture of elements in the manufacture of cement, especially Portland Composite Cement (PCC) [6].

PCC cement is made from grinding together with portland cement slag and gypsum with one or more chemical ingredients, or the result of mixing between portland cement powder with other inorganic ingredients. These inorganic materials include blast furnace slag, pozzolan, silicate compounds, limestone, with a total mineral content of 6% - 35% of the PCC cement mass [7].

The best fly ash addition for the manufacture of composite Portland cement (PCC cement) is 25% because the addition of fly ash as much as 25% obtained a cement incandescent level of 1.78%, cement fineness of 4601.26 cm²/gr and Mortar compressive strength at seven days is 330 kg/cm². All results of the analysis meet SNI 2049:2015 [6]. Additions of additives such as those that occur in the type of PCC cement using fly ash and trass additives can increase the compressive strength of the cement. The value of compressive strength from the ratio of fly ash and trass additives (1:1) is higher in value than fly ash and trass additives (0:1). Addition of fly ash and trass additives on PCC cement can replace its role as a clinker so that the addition of clinker to the cement becomes less and the raw material is economical [8].

In this study, PCC cement will be made with the addition of fly ash from the Lahat area with variations in the acquisition of Lahat fly ash by 6%, 10%, and 14% of the PCC cement mass by testing physical and chemical properties according to SNI 7064:2014.
RESEARCH METHODS

FIGURE 1. Research Flow Chart
The analytical method used in carrying out the analysis of this research is based on the established standards, namely SNI 2049: 2015 concerning Portland Cement and SNI 7064:2014 concerning Portland Composite Cement. The flow chart in this study is explained in FIGURE 1.

In this study, PCC cement was made using a mini meal. The raw materials used in the study are clinker (70%, 66%, and 62%), gypsum (3%), limestone (18%), pozzolans (3%) as the main ingredients and Lahat fly ash (6%, 10% and 14% as additional ingredients. The clinker used comes from PT Semen Baturaja (Persero) Tbk. Baturaja Factory 1. Before making PCC cement, we first test the clinker and fly Lahat ash to be used. Clinker testing is done using XRF (X-Ray Fluorescence). The quality of the clinker tested is determined by chemical compounds, especially Tricalcium Silicate ($C_3S$), and Dicalcium Silicate ($C_2S$). Meanwhile, testing of Lahat fly ash is determined by the content of Loss of Ignition (LOI) and moisture content. After the clinker quality requirements and Lahat ash fly are fulfilled, analysis of Insoluble Residue (IR), Loss of Ignition (LOI), Freelime (FCaO), Blaine, residue (sieve residue) 45µm, and compressive strength are performed.

The content of $C_3S$ and $C_2S$ is determined by the following calculation [9].

\[
\begin{align*}
C_3S & = 4,071CaO - 7,602SiO_2 - 6,718Al_2O_3 - 1,4297Fe_2O_3 \\
C_2S & = 2,8675SiO_2 - 0,7544C_3S
\end{align*}
\]

The content of Insoluble Residue (IR), Loss of Ignition (LOI), Freelime (FCaO), Blaine, residue (remaining sieve) is 45µm, and compressive strength is determined by the following calculation [10].

\[
\begin{align*}
%IR & = \frac{(B-B_1)}{w} \times 100\% \\
%LOI & = \frac{(W_1-W_2)}{W} \times 100\% \\
%FCaO & = \frac{E(V-B)}{W} \times 100\% \\
S & = F\sqrt{T} \\
SA & = \frac{W_1}{W} \times 100\% \\
KT & = \frac{F}{w}
\end{align*}
\]

Information:

- $C_3S$ = Tricalcium silicate (%)
- $C_2S$ = Dicalcium silicate (%)
- $CaO$ = Calcium oxide (%)
- $F CaO$ = Freelime (%)
- $SiO_2$ = Silica oxide (%)
- $S$ = Blaine (cm$^2$/g)
- $Al_2O_3$ = Alumina oxide (%)
- $Fe_2O_3$ = Iron oxide (%)
- $IR$ = Insoluble Residue (%)
- $B$ = Deposition weight (g)
- $LOI$ = Loss of Ignition (%)
- $SA$ = Remaining sieve (%)
- $F$ = Factor (380.85)
- $T$ = Time (s)
\[ B_1 = \text{Blank sediment weight (g)} \quad \bar{F} = \text{Pump press force (N)} \]
\[ KT = \text{Compressive strength (kg/cm}^2 \text{ or MPa)} \quad W = \text{Example weight (g)} \]
\[ W_1 = \text{Sample weight (g)} \quad A = \text{Sample surface area (mm}^2 \text{)} \]
\[ W_2 = \text{The weight of the remaining incandescent sample (g)} \]
\[ E = \text{Ammonium acetate factor } (E = 0.0089 \text{ g/ml}) \]
\[ V = \text{Ammonium acetate volume needed to sample (ml)} \]
\[ B = \text{Ammonium acetate volume needed to blank form (ml)} \]

**RESULTS AND DISCUSSION**

**Clinker Quality**

The results of clinker testing using XRF are presented by TABLE 1.

**TABLE 1. Clinker Test Results.**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Content (%)</th>
<th>Compound</th>
<th>Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO(_2)</td>
<td>21.61</td>
<td>SO(_3)</td>
<td>0.82</td>
</tr>
<tr>
<td>Al(_2)O(_3)</td>
<td>5.04</td>
<td>LSF</td>
<td>94.33</td>
</tr>
<tr>
<td>Fe(_2)O(_3)</td>
<td>4.07</td>
<td>SM</td>
<td>2.37</td>
</tr>
<tr>
<td>CaO</td>
<td>65.18</td>
<td>AM</td>
<td>1.24</td>
</tr>
<tr>
<td>MgO</td>
<td>1.08</td>
<td>C(_4)AF</td>
<td>12.39</td>
</tr>
<tr>
<td>K(_2)O</td>
<td>0.42</td>
<td>C(_3)A</td>
<td>6.47</td>
</tr>
<tr>
<td>C(_3)S</td>
<td>61.39</td>
<td>C(_2)S</td>
<td>15.65</td>
</tr>
</tbody>
</table>

The largest content in clinker is LSF compound of 94.33%, which is one of the compounds that affect the quality of clinker. LSF for good clinkers contains around 93 - 98% with free lime < 2% [9]. LSF is obtained from a comparison of all CaO content contained in a mixture of raw materials with a proportion of other oxides. Meanwhile, the lowest content in clinker is K\(_2\)O compound of 0.42%.

In addition to LSF compounds, the quality of clinker also depends on C\(_4\)AF, C\(_3\)A, C\(_3\)S and C\(_2\)S compounds which later affect the quality of PCC cement. C\(_4\)AF plays a role in determining the color of cement generally ranges from 9 - 11% and is formed at 1100 - 1200\(^\circ\)C during the clinkerization process. C\(_3\)A is formed at a temperature of 1100 - 1200\(^\circ\)C. The C\(_3\)A content in clinkers varies between 7% - 9%. This content plays a role in determining the initial compressive strength of cement. C\(_3\)S is the main component in the clinker, which is formed at temperatures of 1200 - 1450\(^\circ\)C and generally ranges from 57% - 63%. This C\(_3\)S gives the initial cement strength (before 28 days) and can affect the final strength of the cement. C\(_2\)S generally ranges from 15 - 35% and averages 25%. C\(_2\)S is formed at a temperature of 800 - 900\(^\circ\)C and gives ultimate strength to cement [9].
Lahat Fly Ash Content

The content was tested from Lahat fly ash, namely LOI and moisture content. The results of the Lahat fly ash test are presented in TABLE 2.

**TABLE 2.** Test Results of Lahat Fly Ash.

<table>
<thead>
<tr>
<th>Test Material</th>
<th>Loss of Ignition (%)</th>
<th>Moisture Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lahat Fly Ash</td>
<td>3.23</td>
<td>0.47</td>
</tr>
</tbody>
</table>

LOI contained in Lahat fly ash is 3.23%. This content still meets the standards of PT Semen Baturaja (Persero) Tbk, which is a maximum of 5%. LOI on fly ash can indicate unburned carbon (carbon in the fuel that does not change to CO or CO\textsubscript{2} during the combustion process). Unburned carbon in fly ash can cause losses to cement, including water/cement ratios tend to be higher so compressive strength tends to be low, air content in cement is higher, and cement color becomes darker.

Moisture content is the mass ratio of water contained in sample fly ash with a dry mass of fly ash. The moisture content contained in Lahat fly ash is 0.47%. This content still meets the standards of PT Semen Baturaja (Persero) Tbk, which is a maximum of 1%. Moisture content in fly ash affects the value of mortar compressive strength because the moisture content of the fly ash can be considered as the first charge of water in the process of making cement, thus causing the value of mortar compressive strength to increase.

Insoluble Residue

The Insoluble Residue (IR) test results are presented in TABLE 3.

**TABLE 3.** Insoluble Residue (IR) Test Results.

<table>
<thead>
<tr>
<th>Proportion of Fly Ash (%)</th>
<th>Standard Insoluble Residue SNI 7064:2014 (%)</th>
<th>Insoluble Residue (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td></td>
<td>7.82</td>
</tr>
<tr>
<td>10</td>
<td>-</td>
<td>8.75</td>
</tr>
<tr>
<td>14</td>
<td></td>
<td>11.97</td>
</tr>
</tbody>
</table>

IR, one of the impurities that remain after the cement, is reacted with hydrochloric acid and sodium carbonate. IR is limited to preventing the mixing of portland cement with other natural ingredients which cannot be limited to physical requirements [11]. The IR test results show that the higher the proportion of Lahat fly ash used (14%), the higher the IR content of 11.97%.
The addition of 4% proportion of Lahat fly ash causes an increase in IR of 11.89% and if the ratio of Lahat fly ash is added by 8%, then an IR increase of 53.07%.

**Loss of Ignition**
The Loss of Ignition (LOI) test results are presented in TABLE 4.

**TABLE 4. Loss of Ignition (LOI) Test Results.**

<table>
<thead>
<tr>
<th>Proportion of Fly Ash (%)</th>
<th>Standard Loss of Ignition SNI 7064:2014 (%)</th>
<th>Loss of Ignition (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>9.65</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>-</td>
<td>9.56</td>
</tr>
<tr>
<td>14</td>
<td></td>
<td>9.60</td>
</tr>
</tbody>
</table>

LOI is the level of lost incandescent of cement or substance that will be released as gas when heated or burned. The LOI is required to prevent minerals from decomposing during the spawning, where this process can cause damage to the rock after a few years [11]. On the proportion of 6% Lahat fly ash produces an LOI content of 9.65%, the ratio of 10% Lahat fly ash produces an LOI of 9.57% and the proportion of 14% Lahat fly ash can create an LOI of 9.60%.

**Freelime**
The results of the FCaO test (see TABLE 5) show that the FCaO content contained in PCC cement with the proportion of Lahat fly ash that varies does not exceed the standard of PT Semen Baturaja (Persero) Tbk. (maximum 2%) and the resulting level is equal to 0.71%. This means that the proportion of Lahat fly ash that varies does not affect the amount of FCaO content produced. The frelime level, which is hydrated free lime, provides Ca(OH)2 if given a high temperature. This makes the volume of free lime more abundant so that it can cause the development of capacity during binding or expansion. High expansion can cause cracks and damage to cement when hardened. FCaO is formed because the particle size of the kiln feed is not sufficiently smooth, clinker combustion is less than perfect, the CaO content is too high in the kiln feed and decomposition of clinker minerals during the cooling process [12].
### TABEL 5. Freelime (FCaO) Test Result.

<table>
<thead>
<tr>
<th>Proportion of Fly Ash (%)</th>
<th>Standard Free Lime Internal PTSB (%)</th>
<th>Free Lime (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>max. 2</td>
<td>0.71</td>
</tr>
<tr>
<td>10</td>
<td>0.71</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>0.71</td>
<td></td>
</tr>
</tbody>
</table>

**Blaine**

The results of the Blaine test (see TABLE 6) show that the proportion of 6% Lahat fly ash with a grinding time of 120 minutes resulted in cement fineness of 4073.50 cm²/gr where the residual (residual sieve) of 45 μm was 12.36 %. In the proportion of 10% Lahat fly ash with 110 minutes of grinding time the cement fineness was produced by 3209.10 cm²/gr where 45 μm residue was 12.94%, and for the proportion of 14% Lahat fly ash with 100 minutes grinding time, cement fineness was produced as big as 5086.9 cm²/gr where the residual 45 μm is 10.10%.

### TABLE 6. Blaine Test Results of PCC Cement with Addition of Lahat Fly Ash.

<table>
<thead>
<tr>
<th>Proportion of Fly Ash (%)</th>
<th>Grindling Time (minute)</th>
<th>R45μm</th>
<th>Blaine (cm²/gr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>120</td>
<td>12.36</td>
<td>4073.50</td>
</tr>
<tr>
<td>10</td>
<td>110</td>
<td>12.94</td>
<td>3209.10</td>
</tr>
<tr>
<td>14</td>
<td>100</td>
<td>10.10</td>
<td>5086.90</td>
</tr>
</tbody>
</table>

Based on this research, the most refined PCC cement was obtained from PCC cement grain with the addition of Lahat fly ash proportion of 14%. The finer the particles or grain of cement, the higher the compressive strength. This is because more surface area reacts with water and mixes with aggregate. The fineness of the cement is the physical property of cement, the finer the cement granules, the faster the hydration process of the cement so that the strength of the mortar will be faster. The finer the cement grain, the more time cement needs to harden more quickly.

**Press Strength**

The results of the observation of PCC cement compressive strength with the addition of Lahat fly ash are presented in TABLE 7.
TABLE 7. Observation Results of PCC Cement Compressive Strength with Addition of Lahat Fly Ash.

<table>
<thead>
<tr>
<th>Proportion of Fly Ash (%)</th>
<th>3 Days</th>
<th>7 Days</th>
<th>28 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>150.92</td>
<td>230.86</td>
<td>325.08</td>
</tr>
<tr>
<td>10</td>
<td>160.70</td>
<td>221.28</td>
<td>306.93</td>
</tr>
<tr>
<td>14</td>
<td>164.38</td>
<td>243.30</td>
<td>351.59</td>
</tr>
</tbody>
</table>

The results of the three days, seven days and 28 days compressive strength have met the SNI 7064: 2014 standard, where the standard for three days compressive strength is 130 kg/cm², seven days compressive strength of 200 kg/cm², and 28 days compressive strength at 280 kg/cm². The following is the relationship between the addition of the proportion of Lahat fly ash to the compressive strength of PCC cement mortar indicated by FIGURE 2.

FIGURE 2. Graph of the Relationship between the Proportion of Lahat Fly Ash and Compressive Strength

In FIGURE 2, it can be seen that the relationship between the addition of the proportion of Lahat fly ash and the PCC 3-day cement mortar compressive strength is directly proportional or linear when the value of the ratio of Lahat fly ash is higher, the resulting compressive strength is more significant. However, at 7 days and 28 days compressive strength the graph does not show a linear relationship, because there is a decrease in compressive strength when the proportion of Lahat fly ash is raised 4% or the ratio of Lahat fly ash is 10% and a surge occurs when the rate of Lahat fly ash is increased 4% again.
Setting Time
The setting time is determined if the cement paste has undergone a setting (which has thickened) and hardening (which has hardened) for several hours. In physically testing the quality of PCC cement the setting time is carried out by 2 testing stages, namely initial setting time and final setting time. Initial setting time is when the dough starts to occur until a certain stiffness begins to occur where the dough has begun to not workable. Meanwhile the final setting time is the time the dough starts to occur until full stiffness occurs. The initial setting time usually ranges from 2 - 5 hours and the final setting time is 3 - 6 hours [11]. The results of observations of setting time on PCC cement that have been made show that by increasing the proportion of Lahat fly ash which can increase the initial setting time, while the final setting time is getting faster (see TABLE 8).

<table>
<thead>
<tr>
<th>Proportion of Fly Ash (%)</th>
<th>Initial Setting Time (minute)</th>
<th>Final Setting Time (minute)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>122</td>
<td>272</td>
</tr>
<tr>
<td>10</td>
<td>123</td>
<td>253</td>
</tr>
<tr>
<td>14</td>
<td>134</td>
<td>245</td>
</tr>
</tbody>
</table>

CONCLUSIONS
In this study the proportion of Lahat fly ash which can produce PCC cement in optimum conditions with physical and chemical requirements in accordance with SNI 7064: 2014 is the addition of Lahat fly ash by 14%, where the frelime value produced does not exceed the standard and value of blaine, compressive strength, the initial setting time reaches the highest results and the final setting time produces the lowest value. Therefore, the ideal composition in making PCC cement is adding Lahat fly ash as much as 14%, 62% clinker, 3% gypsum, 18% limestone, and 3% pozzolan, thus Lahat fly ash can be used as an alternative mixture in the manufacture of cement PCC.

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