Characterization of PCC Cement by Addition of Napa Soil from Subdistrict Sarilamak 50 Kota District as Alternative Additional Material for Semen Padang

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Abstract. The study has been conducted to determine characteristics of the portland composite cement by the addition of napa soil from Sarilamak subdistrict, 50 Kota District as an alternative additional material at PT. Semen Padang. Napa soil is a natural material highly containing silica and alumina minerals so that it can be one of material in producing cement. This study aims to determine the effect of napa soil on the quality of portland composite cement. Napa soil used in the variation compositions 0%, 4%, 8%, 12% and 16%, for control of cement used 8 % of pozzolan and 0 % of napa soil. Determination of cement quality by testing cement characteristics include blaine test, sieving, lost of ignition or LOI, insoluble residue, normal consistency, setting time and compressive strength. Cement was characterized using XRF. Fineness of cement decreases with the addition of napa soil. Lost of Ignition of cement decreased, while the insoluble residue increased with the addition of napa soil. Normal consistency of cement increasing, so does initial setting time and final setting time of cement. While the resultant compressive strength decreases with the addition of napa soil on 28 days, 342, 325, 307, 306, and 300 kg / cm2.

1. Introduction
Proportional amount will result the ability in binding fine and coarse aggregate to be a material called concrete the quality of good concrete depends on the quality of the cement. In processing of producing cement, it using additive and additional material to get good quality and to minimize the defrayment. The additional material used in the producing of cement is gypsum that has function as retarder in hardening of cement, and pozzolan. Pozzolan can be divided into two, they are natural pozzolan and synthetic pozzolan Natural pozzolan is a pozzolan that comes from nature, meanwhile synthetic pozzolan is synthesized by combustion of clay, husk and coal forms fly ash [1]. Pozzolan contains silica and alumina that when it is mixed by water and free CaO will form cement compound [2].

Napa soil is natural material that is used by West Sumatra people as medicine of stomachache and diarrhea [3]. Napa soil highly contains SiO2 and Al2O3, the presentation is SiO2 63.20 % and Al2O3 16.55% [4]. Thus, napa soil can be used as alternative material as source of silica and it is hoped that it can be used as potential inorganic material as adsorbent, catalyst and additional material in cement industry [5].

In West Sumatera, there are several areas that lies napa soil, they are district of 50 Kota, district of Tanah Datar, district of Pesisir Selatan, and district of Solok. Seeing the abundance of napa soil in west Sumatra so the research about the effect of the utilization of napa soil as alternative additional material in producing cement is very interesting to be done. Thus, in this research the writer will discuss about
the effect of the utilization napa soil from Sarilamak subdistrict, 50 Kota District as alternative additional material in producing cement.

2. Experimental

2.1. Materials and Instruments
The instrument used in this research are crusher brands ME-100 JAW CRUSHER 5”X8”, mini mill, mixer machine Toni Technik ToniMI, mixer knife, vicat tools Zwick Roell Toni SET, furnace, analytical scale Sartorius BL 210 S, alpine air jet 200LS-N, cube mold, gloves, pressing equipment, oven brand Carbolite CWF 1300, crusher, ring tablet, blaining tool brand Toni Technik Toni Trol, glass equipment.

Characterization of cement using X-ray fluorescence (xrf).

The materials used in this research are napa soil from subdistrictsarilamak, district 50 kota, clinker, gypsum, lime stone, ottawa sand, herzog pill, NaOH brands Merck, HCl brands KGaA, MM indicator, NH4NO3.

2.2. Methodology

2.2.1 Preparation of cement sample. The chunk of napa soil is crushed in crusher, then it is dried for a day. Sample is made by mixing clinker, gypsum, lime stone and pozzolan for control cement, and clinker, gypsum, lime stone and napa soil with the composition in Table 1 after being grinded using mini mill. The cement of napa soil is ready to be tested.

| Table 1. The Composition of Cement (%) |
|---------------------------------------|
| Sample             | Napa Soil | Clinker | Gypsum | Limestone | Pozzolan |
| Cement Control    | -         | 76      | 4      | 12        | 8        |
| Cement 1          | 4         | 76      | 4      | 16        | -        |
| Cement 2          | 8         | 76      | 4      | 12        | -        |
| Cement 3          | 12        | 76      | 4      | 8         | -        |
| Cement 4          | 16        | 76      | 4      | 4         | -        |

Based on Table 1, The amount of composition of napa soil is varied. It is done because napa soil is the testing variable. Meanwhile the lime stone is the component that it does not influence enough to the quality of cement. The amount of sample must be 5000 g, so the composition of lime stone is possibly reduced to make the amount of sample in producing cement keeps 5000 g.

2.2.2 Characterization of cement. Characterization of cement done based on SNI 2049: 2015.

Blaine Analysis
2.8948 g sample is entered to blaining tool brand Toni Technik, then the fineness of the cement can be known.

Sieving Analysis
20 gram of sample is placed on 45µ sieve, the machine is switched on and set the timer to 3 minutes after three minutes, the rest of the sample found on the sieve is weighted, then the percentage can be calculated.

\[
\% \text{rest} = \left(\frac{\text{mass of rest}}{\text{mass before sieving}}\right) \times 100 \%
\]

Lost of Ignition (LOI) Analysis
Analysis of LOI is aimed to determine the containing of water and CO2 that lost when the cement ignited. 1.000 gram of cement is ignited in temperature of 1000°C for 15 minutes, then it is placed in desiccator, then the LOI can be calculated.

\[
\% \text{LOI} = \left(\frac{\text{dish+sample before being ignited}-\text{after being ignited}}{\text{mass of sample}}\right)
\]
**Insoluble Residue Analysis**

1 g of sample is placed in 250 ml beaker glass and added 25 ml of HCl and aquadest until 100 ml. Then the solution is boiled and filtered, then it is rinsed by aquadest. The filtering paper that containing the precipitation is placed in the beaker glass which containing 100 ml NaOH 1 %. The filter paper is broken then boiled. Then it is added by two drops indicator of red methyl and added by drops of HCl until the color of solution become red. That solution is filtered by filtering paper and rinsed by hot water and NH4NO3 solution to make sure that the filtering paper is perfectly rinsed. Filtering paper and its content is placed in crucible that has been measured its weight then it is placed in furnace in 1000 °C for 30 minutes, then calculate the insoluble part of cement.

\[
\text{Insoluble Part} = \frac{\text{mass of precipitate}}{\text{mass of sample}} \times 100\%
\]

**Normal Consistency Analysis**

650 gram of sample is placed in the mixing dish and it is added by 156 ml of water. Cement is added into the water then mix it for 30 seconds then stop mixing for 15 seconds. The paste that sticks on the bowl is collected, mix it again for 1 minutes, stop mixing and collect the paste that sticks on the dish. Make the shape of the paste to be the ball shape and push the ball through the vicat ring.

**Setting Time Analysis**

650 gram cement is placed based on its normal consistency. Cement is added by water to form a paste. The paste of cement is prepared to normal consistency analysis. After being printed, the analysis is done by penetrating test using vicat pin with 1 mm diameter in every 10 minutes. The first setting is marked when the pin penetrates more than 25 mm. The final setting is marked when the vicat pin can not penetrate the sample.

**Compressive Strength Analysis**

740 gram of sample, 2035 gram of standard sand and 410 ml of water. Cement is placed in the dish and it is added by water. Sample is mixed using mixer with low velocity (140±5) radiants per minutes for 30 seonds. Then the sabd is slowly added with low velocity. The mixing is stoped then the velocity is increased until (285±10) radiants per minutes for 30 seconds. The mixer is stopped and the mixer :eft for 1.5 minutes. For the first 15 seconds, take the mixture that stick in the surface of the dish, then the dish is covered by its cover to prevent the water evaporation of the paste. The mixer is switched on for 60 seconds with medium velocity, then it is stopped for 90 seconds, then it is run for 15 seconds. The mixture is poured down to each cube, then it is slowly pressed to ± 4 mm for 32 times for one sample. Then the cube mold is fully fulfilled and pressed for 32 times. The excess of the mixture on the surface of the mold is cutted using the knife and the surface of the sample is softened using the spoon. After being molded, the sample is placed in the humid room for 20-24 hours and the surface of the sample is conditioned to have contact with the humid air, but it prevented to have contact with water. After one day, the sample is take out from the mold, the sample is soaked in the water containing calcium oxide and it is placed in the anti-rust humid room until the definite time for analyzing the sample. The compressive strength analysis is done for the sample in the age of 3 days, 7 days and 28 days. The sample is cleaned by the dry cloth. The compressing of sample is done by using compressive tools that has been calibrated.

**XRF Analysis**

XRF analysis is done to know the chemical composition of sample, they are napa soil cement and napa soil. 30-gram sample is added by 2 pellets of herzog pill, then is grinded at it is ready for XRF analysis to get the information about chemical composition of the sample.

3. Results and Discussion

3.1. *The Effect of Utilizing of Napa Soil to The Chemical Composition of Cement*

The Effect of Utilization of Napa Soil to The Chemical Composition of Cement Presented in Table 2.
Table 2. The Result of XRF Analysis to The Cement with Variation of Napa Soil Composition.

| Composition of Napa Soil (%) | SiO₂ (%) | Al₂O₃ (%) | Fe₂O₃ (%) | CaO (%) | MgO (%) | SO₃ (%) | Other (%) |
|-----------------------------|----------|-----------|-----------|---------|---------|---------|----------|
| 0                           | 22.00    | 6.10      | 3.29      | 59.36   | 0.91    | 1.79    | 6.55     |
| 4                           | 20.79    | 5.25      | 3.03      | 61.09   | 0.90    | 1.84    | 7.10     |
| 8                           | 23.41    | 5.55      | 3.11      | 58.92   | 0.89    | 1.79    | 6.33     |
| 12                          | 25.57    | 5.59      | 3.03      | 57.35   | 0.86    | 1.71    | 5.89     |
| 16                          | 28.36    | 5.94      | 3.11      | 54.71   | 0.85    | 1.67    | 5.36     |

In the Table 2. It can be seen that it happens the changing of mineral composition of cement by addition of napa soil. The more % of napa soil addition, there will be an increasing of content of SiO₂, Al₂O₃, and Fe₂O₃ while the content of CaO, MgO, and SO₃ decrease. It happens because napa soil is a natural material that highly contains alumina and silica. The highest content of SiO₂ is found in napa soil that contain 16 % of napa soil, it has 28.36 % of SiO₂. CaO, SiO₂, and Al₂O₃ are the most chemical composition contained in the cement. The content of SO₃ is limited in determining the cement quality. Because, if its content is more than the qualified standard, it will make the setting time of cement become longer. According to SNI 7064:2014 [6] SO₃, the maximum content of SO₃ is 4 %.

3.2. The Effect of Utilizing of Napa Soil to Blaine Analysis.

Blaine analysis is conducted to determine the fineness of cement particle. The effect of utilizing of napa soil to blaine analysis can be seen in the Table 3.

Table 3. Result of Blaine Anlaysis of Cement

| Variation of Napa Soil Composition (%) | The Fineness of Cement Particle (cm²/g) | The Fineness of Cement Particle (m²/kg) |
|---------------------------------------|----------------------------------------|-----------------------------------------|
| 0                                     | 4012                                   | 401.2                                   |
| 4                                     | 4027                                   | 402.7                                   |
| 8                                     | 3977                                   | 397.7                                   |
| 12                                    | 3934                                   | 393.4                                   |
| 16                                    | 3974                                   | 397.4                                   |

In Table 3, it can be seen that the fineness of cement particle produced by the addition of napa soil still qualifies the standard of SNI 7064:2014, it is minimally 280 m²/kg. The curve of correlation between napa soil composition and the fineness of cement particle can be seen in Figure 1.

Figure 1. Curve of Correlation between Napa Soil Composition and The Fineness of Cement Particle.
Based on the curve above, it can be seen that the more quantity of napa soil in the cement the fineness of the cement will be smaller, it means that the cement particle produced is getting coarse. However, in the addition 16 % of napa soil for the cement, it increases the fineness of the cement particle. It happens because of unconstant grinding time of cement.

3.3. The Effect of Utilizing of Napa Soil to Sieving Analysis.
The effect of utilizing of napa soil to sieving analysis can be seen in the Table 4.

| Variation of Napa Soil Composition (%) | Rest on The Sieve |
|---------------------------------------|-------------------|
| 0                                     | 13.71             |
| 4                                     | 15.16             |
| 8                                     | 15.67             |
| 12                                    | 15.86             |
| 16                                    | 15.98             |

From the data above, it can be seen that the rest on the sieve (%) for control cement is 13.71 %, it is much smaller than the cement that contain napa soil. It means that the particle size of control cement is smaller than napa soil cement. The curve of corelation between napa soil composition and sieve analysis can be seen in Figure 2.

![Figure 2. Curve of Corelation Between Napa Soil Composition and % 45 µ sieving Analysis](image)

Based on Figure 2, it can be seen that the more quantity of napa soil composition in cement, the more percentage of sieving analysis. It means that the percentage of cement that can pass the sieve is getting smaller, because the particle size is getting bigger, it makes the surface area smaller, it makes the compressive strength of the cement is getting lower.

3.4. The Effect of Utilizing of Napa Soil to Lost of Ignition Analysis
The effect of utilizing of napa soil to lost of ignition analysis can be seen in the Table 4.

| Variation of Napa Soil Composition (%) | Lost of Ignition (%) |
|---------------------------------------|----------------------|
| 0                                     | 6.48                 |
| 4                                     | 7.65                 |
| 8                                     | 6.32                 |
| 12                                    | 4.85                 |
| 16                                    | 3.37                 |
From the data above, it can be seen that 0% addition of napa soil result 6.48% of lost of ignition, this value is almost the same with the lost of ignition in 8% of napa soil addition, it is about 6.32%. The curve of correlation between napa soil composition and lost of ignition analysis can be seen in Figure 3.

![Figure 3. Curve of Corelation Between Napa Soil Composition and Lost of Ignition (%)](image)

*Figure 3. Curve of Corelation Between Napa Soil Composition and Lost of Ignition (%)*  
Based on the figure above, it can be seen that the more quantity of napa soil composition decreases the percentage of lost of ignition of cement. It is caused by the most component of napa soil is SiO$_2$, and the value of lost of ignition is influenced by the content of CaO (CaCO$_3$) in the limestone. In this study the percentage of limestone decreases as the increasing of percentage of napa soil.

3.5. The Effect of Utilizing of Napa Soil to Insoluble Part Analysis

The effect of utilizing of napa soil to the value of insoluble part analysis can be seen in Table 5.

| Variation of Napa Soil Composition (%) | Insoluble Part (%) |
|---------------------------------------|--------------------|
| 0                                     | 7.72               |
| 4                                     | 4.94               |
| 8                                     | 8.11               |
| 12                                    | 11.76              |
| 16                                    | 15.19              |

From the data in the table 5, it can be seen that the value of insoluble part analysis of 0% of addition of napa soil (pozzolan 8%) is 7.72%, it almost has the same value with 8% of addition of napa soil, it is 8.11%. The curve of correlation between napa soil composition and the value of insoluble part analysis can be seen in Figure 4.

![Figure 4. Curve of Corelation Between Napa Soil Composition and The Value of Insoluble Part Analysis](image)
Based on the figure above, it can be seen that the increasing of napa soil composition in cement increases the value of insoluble part of cement. It is caused by the the impurities content such as organic material in limestone and napa soil.

3.6. The Effect of Utilizing of Napa Soil to Normal Consistency Analysis
Normal consistency analysis is done to determine the quantity of water needed to form cement paste. The effect of utilizing of napa soil to normal consistency analysis can be seen in Table 6.

| Variation of Napa Soil Composition (%) | Normal Consistency (%) |
|---------------------------------------|------------------------|
| 0                                     | 25.23                  |
| 4                                     | 24.62                  |
| 8                                     | 24.92                  |
| 12                                    | 25.54                  |
| 16                                    | 25.85                  |

Based on the data in table 6, based on Table 6, 0 % of napa soil addition (pozzolan 8 %) results the lowest value of normal consistency, it is 25.23 %, so it needs less water in producing the cement paste. The curve of correlation between napa soil composition and the value of normal consistency can be seen in Figure 5.

![Figure 5. Curve of Correlation Between Napa Soil Composition and Value of Normal Consistency](image)

Based on Figure 5 it can be seen that the increasing of napa soil composition in cement increases the value of normal consistency of cement. It means that the more quantity of water needed in producing the cement paste. The value of normal consistency is influenced by the particle size of cement that make the water distribution happen difficultly in cement because the low surface area of cement, thus it needs more water.

3.7. The Effect of Utilizing of Napa Soil to Setting Time Analysis
The setting time of cement is the time needed for cement hardening process.

| Variation of Napa Soil Composition % | Setting Time |
|--------------------------------------|--------------|
|                                      | Outset (minutes) | Final (minutes) |
| 0                                     | 142           | 231            |
| 4                                     | 120           | 209            |
| 8                                     | 122           | 211            |
| 12                                    | 126           | 215            |
| 16                                    | 128           | 217            |
Based on Table 8 it can be seen that 0% of napa soil addition results 142 as the highest value of outset setting time and 231 minutes as final setting time. Outset setting time is started when the mixture of cement made that is indicated by penetrating of vicate for less than 25 mm. Final setting time is started when the mixture of cement made and form full strength [7], it is indicated when the vicate can not penetrate the surface of harden cement. Curve of corelation between napa soil composition and cement setting time can be seen in Figure 6.

**Figure 6.** The Curve of Corelation Between Napa Soil Composition and Cement Setting Time

Based on Figure 6, it can be seen that the increasing of napa soil composition in cement increases the cement setting time. The composition of gypsum is same in each samples so that the influence can not be significantly seen.

The particle size of cement also influences the cement setting time. The bigger particle size of the cement the longer setting time of the cement. The setting time of the cement that contains napa soil still fulfill the standard of the cement SNI 7064:2014, the maximal outset setting time is 45 minutes and 375 minutes for the final setting time.

**3.8. The Effect of Utilizing of Napa Soil to Compressive Strength Analysis**

Compressive strength analysis is done in the day of 3, 7, and 28. The effect of utilizing of napa soil to the compressive strength of the cement can be seen in Table 9.

| Napa Soil Composition (%) | Compressive Strength (kg/cm²) |
|---------------------------|-------------------------------|
|                           | Day 3 | Day 7 | Day 28 |
|---------------------------|-------|-------|--------|
| 0                         | 195   | 262   | 342    |
| 4                         | 197   | 256   | 325    |
| 8                         | 186   | 248   | 307    |
| 12                        | 182   | 246   | 306    |
| 16                        | 181   | 236   | 300    |

From the data above, it can be seen that the compressive strength of the cement containing 4, 8, 12, and 16% of napa soil tend to decrease than the compressive strength of control cement, curve of corelation between napa soil composition and compressive strength is in Figure 7.
Figure 7. The Curve of Corelation Between Napa Soil Composition and Compressive Strength of Cement

Based on the data in Figure 7, it can be seen that the increasing of quantity of napa soil in the cement decreases the compressive strength of the cement. It is caused by the content of SiO$_2$ in cement increases as the increasing of napa soil composition. The compressive strength of napa soil cement is still qualified for the cement standard SNI 7064:2014, the minimal compressive strength of day 3, 7 and 28 are 130, 200, and 280 kg/cm$^2$.

4. Conclusion

Based on the result of study that has been conducted, there are several conclusions. The addition of napa soil causes the high level of SiO$_2$, Al$_2$O$_3$, Fe$_2$O$_3$ in cement, while the others composition in cement such as CaO, MgO, and SO$_3$ decrease. The utilization of napa soil as alternative material as addition material in producing cement increases the normal consistency, the insoluble part and the setting time, while the rest on sieve, the loss of ignition and the compressive strength decreases.

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