Using Geopolymer Composites for Heat and Corrosion Resistant Pipes

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Abstract. This research studied the use of carbon fiber reinforced geopolymer composites as corrosion resistant pipe material. Geopolymer material is a general term for material synthesized from fly ash. Fly ash is an industrial by-product (coal combustion residue) with high silica content (SiO$_2$) and Alumina content (Al$_2$O$_3$). In addition there are also other elements in a relatively small percentage. This material will compound like cement if activated with Sodium Silicate. This study aims to obtain the optimum composition for high quality mechanical properties. The method used is to synthesize material with various elemental compositions. Furthermore, a mechanical test (bending test) is performed to see the effect of each element to be optimized. Besides that, a physical test in the form of SEM-EDX was also conducted to see the microstructure. Materials with optimum composition have been fabricated as pipes with centrifugal casting process. Mechanical testing of pipe samples is done by hoop tensile strength test. The test results show that the strength of geopolymer composites can achieve 58 MPa.

1. Introduction
Pipeline is transportation mean to drain fluid that is commonly used in industry. Nowadays, steel pipe is mostly used in industry field. However, in a certain condition, it is needed a pipe that is resistant to corrosion and heat temperature, especially it is in geothermal industry. One of the problems that appears while using steel pipe is corrosion rate and deposits of crust in the pipes used. This case can make the company get lossed. In Indonesia, PERTAMINA company have to spend USD 6-7 for steel pipe treatment used [1].

Geopolymer is one of the materials that is resistant to heat temperature and corrosion. Geopolymer is the synthetic result from basic material that contains a lot of bonding elements of alumina (Al), silica (Si) and oxide. Geopolymer is made of coal industry waste thish are fly-ash dust and others materials that contain silica. The material will become strong material if it is activated with activator like sodium Hidroksida and sodium Silicate [2].

The aim of this research is to know the mechanic characteristic of geopolymer pipe as an alternative pipe. The success of this research is hoped which can answer the problem that is faced in pipe, like the level of corrosion and resistant to heat temperature. Therefore, it can impact to efficiency and low treatment cost. This reasearch is also hoped can facilitate in resolving fly-ash waste and give point plus to the waste industry.
The composition of geopolymer composite that is added the element of silicon, dioxide and sodium silicate that will be molded as the standard of ASTM C 1161 [3], and then it will be tested by using Bending Three Point method. After getting the best competition, geopolymer composite will be molded on a pipe mould. The pipe mould will be powered by mover bike. The mover bike will give centrifugal force, so geopolymer material that is in pipe mould will be formed like the mould. After becoming geopolymer pipe [4-7].

The optimization of geopolymer composite by adding the element of silicon dioxide and sodium silicate in making geopolymer pipe by using centrifugal casting molding, it is hoped that it can increase the power of geopolymer composite pipe which is resistant to corrosion and heat temperature.

2. Materials and Experiment

Materials used in the manufacture of geopolymers are Fly Ash (FA), Kaolin (K), Calcium Oxide (CaO) and Carbon Fiber (CF). This material mixture is then added with SiO₂, NaOH and Na₂SiO₃ which functions as activator. Fly ash comes from PLTU Tarahan Sector III Lampung Selatan Indonesia and kaolin used commercial type. The chemical composition of FA and K is investigated by XRF (X-Ray Fluorences) testing and the respective content is shown in Table 1. Silica fume used is a commercial type from PT. NORMET Indonesia and Carbon Fiber medium type (1.56 kg / m³).

| Materials | Si | Al | Fe | Ti | Zr | Mn | Sb | Zn | Sn | Ni | Pb | Ga |
|-----------|----|----|----|----|----|----|----|----|----|----|----|----|
| Fly-ash   | 48,2 | 26,0 | 22,1 | 2,53 | 0,49 | 0,25 | 0,20 | 0,10 | 0,07 | 0,06 | 0,02 |
| Kaolin    | 57,6 | 37,2 | 3,82 | 1,23 | 0,1 | - | - | 0,04 | - | 0,04 | - | 0,02 |

2.1 Design of Matrix Composition by Taguchi Method

Composite Geopolymer is made with 2 stages: activator preparation and mixing of solid binder. In preparation for making the activator to be done first is to dissolve the crystalline NaOH into a solution of 14M by adding a solution of Aquades. The ratio of Na₂SiO₃ and NaOH ratio is 0.3. The carbon fiber is cut 1,5 cm and then weigh all the material according to the predetermined variation.

There are 4 materials that will be varied by the taguchi method [8]: Silicon dioxide, kaolin, calcium oxide (CaO), and carbon fiber. Silicon dioxide, kaolin, and CaO use the best composition performed by Jamiatul Akmal [2], SiO₂ 27% wt, Caolin 51% wt, and CaO 0,5 wt%, where as the carbon fiber is 13%.

Composition are respectively: A, B, C, D,E and F, as shown in Table 2. Table 3 is an orthogonal table showing the mixing of factors with varying levels. There are 9 mixtures to be tested to obtain the optimum mixture.
Table 3. Taguchi orthogonal matrix L9(3^4)

| No | SiO₂ (gr) | K (gr) | Na₂SiO (gr) | NaOH (gr) |
|----|----------|--------|-------------|-----------|
| 1  | 27       | 55     | 15          | 15        |
| 2  | 27       | 55     | 20          | 10        |
| 3  | 27       | 51     | 22.5        | 7.5       |
| 4  | 30       | 55     | 20          | 7.5       |
| 5  | 30       | 53     | 22.5        | 15        |
| 6  | 30       | 51     | 15          | 20        |
| 7  | 33       | 55     | 22.5        | 20        |
| 8  | 33       | 53     | 15          | 7.5       |
| 9  | 33       | 51     | 20          | 15        |

The next step is mixing the powder materials: Silicon dioxide, kaolin, CaO and CF until evenly distributed. Then add activator solution, with the ratio of powder to the activator solution is 0.7. After stirring evenly, put the mixture into a 90x6x8 mm mold and vibrate at a frequency of 54000 Hz for about 30 minutes. The sample is then stored in an incubator with a temperature of 30-35 °C for ≥ 28 days.

2.2 Manufactures pipes with centrifugal casting machines

Geopolymer composites with the best composition are then molded into pipes with a centrifugal casting machine, as shown in Figure 1a. Figure 1b shows the molded pipe.

![Figure 1. (a) Centrifugal machine testing, (b) The molded pipe](image)

2.3 Experiment Testing

Rectangular bars with size (90x6x8) mm subjected to the three point bending test to determine the strength and flexural modulus (based on ASTM C 1161), as shown in Fig. 2. The test machine used is MTS Landmark (capacity 50 kN) with loading speed 1.0 mm / min. Flexural strength can be determined by Eq. (1).

\[ \sigma_f = \frac{3P_mS}{2WD^2} \]  

(1)

where \( \sigma_f \) is flexural strength (MPa), \( P_m \) is ultimate load (N), \( S \) is the span of the sample, \( W \) is the specimen width and \( D \) is the specimen thickness. The flexural modulus was computed using the initial slope of the load-displacement curve, \( \frac{\Delta P}{\Delta x} \), using the Eq. (2)

\[ E_f = \frac{S^3}{4WD^3} \left( \frac{\Delta P}{\Delta x} \right) \]  

(2)
Figure 2. Schematic of the three point bend test

3. Result and discussion
There were 9 samples tested and each sample consisted of 3 specimens. Figure 3 shows the tested sample and the cross-sectional view broken. Flexural strength of each specimen is shown in Table 4.

Figure 3. Deformation specimens after testing

The influence of factor A, B, E and factor F to SN Ratio are shown in Figures 4 and 5, respectively. Factor E and F shows the effect of Na$_2$SiO$_3$ and NaOH addition on flexural strength, to optimum at S3 level, with 27%wt SiO$_2$, 51%wt kaolin, 5%wt CaO and 13%wt CF addition. Compositions are shown in Table 4.

Table 4. Flexural strength of the samples (MPa)

| Mix | A  | B  | C  | D  | E  | F  | Flexural Strength (MPa) | Average |
|-----|----|----|----|----|----|----|--------------------------|---------|
|     |    |    |    |    |    |    | Spesimen 1 Spesimen 2 Spesimen 3 |         |
| S1  | 27 | 55 | 5  | 13 | 15 | 15 | 15.26 9.34 4.38 | 9.66    |
| S2  | 27 | 53 | 5  | 13 | 20 | 10 | 7.83 31.57 9.15 | 16.18   |
| S3  | 27 | 51 | 5  | 13 | 22.5 | 7.5 | 64.87 49.60 60.00 | 58.16   |
| S4  | 30 | 55 | 5  | 13 | 20 | 7.5 | 19.84 24.51 12.11 | 18.82   |
| S5  | 30 | 53 | 5  | 13 | 22.5 | 15 | 7.63 20.89 12.02 | 13.51   |
| S6  | 30 | 51 | 5  | 13 | 15 | 20 | 23.56 6.48 26.61 | 18.88   |
| S7  | 33 | 55 | 5  | 13 | 22.5 | 20 | 18.03 17.45 30.24 | 21.91   |
| S8  | 33 | 53 | 5  | 13 | 15 | 7.5 | 34.34 44.74 43.40 | 40.83   |
| S9  | 33 | 51 | 5  | 13 | 20 | 15 | 34.72 25.75 32.34 | 30.94   |
Figure 4. Effect plot for SN ratios

Figure 5. Effect plot for Means

Table 5. The 5 optimum samples and their compositions

| Mix Number | SiO\textsubscript{2} (gr) | K (gr) | CaO (gr) | CF (%) | Na\textsubscript{2}SiO\textsubscript{3} (gr) | NaOH (gr) |
|------------|----------------|-------|----------|--------|--------------------------|-----------|
| S3         | 27             | 51    | 5        | 13     | 22,5                     | 7,5       |
| S6         | 30             | 51    | 5        | 13     | 15                       | 20        |
| S7         | 33             | 55    | 5        | 13     | 22,5                     | 20        |
| S8         | 33             | 53    | 5        | 13     | 15                       | 7,5       |
| S9         | 33             | 51    | 5        | 13     | 20                       | 15        |

Mix S3 is the best specimen with composition: Silicon dioxide (27% wt), kaolin (51% wt), CaO (5% wt), and carbon fiber (13% wt), with flexural strength 58,16 MPa. Figure 6 shows the stress-displacement curve on the geopolymer composite for 5 optimum samples. The curves appear to be 2 segments: linear segments and non-linear segments. This shows a similar trend with steel performance. In the first segment the curve is relatively straight and no plastic damage has
occurred. In the second segment, after reaching the maximum strength the curve drops suddenly and then horizontally until the specimen breaks out debonding between the matrix and fiber. This curve also explains that the addition of carbon fiber (CF) can significantly increase flexural strength and modulus. Figures 7 and 8 respectively show flexural strength and modulus strength.

![Figure 6. The stress and displacement curves of the 5 optimum samples](image)

![Figure 7. The average flexural strength of 5 optimum samples](image)

![Figure 8. The flexural modulus of 5 optimum samples](image)
Back Scatter Electron (BSE) Images for samples with different compositions of Na$_2$SiO$_3$ and NaOH are shown in Figure 9. Surface morphology for all samples shows a low density and there are holes with even distribution. The low density level is caused by the process of compacting that is not optimal when molding samples, only by the vibration process. This becomes the input for further research so that compaction is done with pressurized molding process.

The composition of the elements for each sample is shown in Table 6. In this picture is shown spectrum 1 and spectrum 2 for each sample, indicating the points analyzed. In Figure 9a, the points shown are matrix (without fiber), there is an element of carbon with a small percentage (15% -22%). This carbon comes from carbon tape which is added as an electron intermediate during the scanning process Figure 9b shows the analyzed points on carbon fiber (CF) with relatively high carbon content (about 81-95%). Likewise, Figure 9c and Figure 9d, each showing the same analysis form as Figure 9a and Figure 9b.

**Figure 9.** BSE image for samples with different compositions of Na$_2$SiO$_3$ and NaOH (a) Na$_2$SiO$_3$ 0 gr and NaOH 30 gr (b) Na$_2$SiO$_3$ 15 gr and NaOH 15 gr (c) Na$_2$SiO$_3$ 20 gr and NaOH 10 gr, (d) Na$_2$SiO$_3$ 22.5 gr and NaOH 7.5 gr.

**Table 6.** Result Point Analysis Sample

| Elemen | CaO=0 (% Atom) | CaO=0 (% Atom) | CaO=4% (% Atom) | CaO=4% (% Atom) |
|--------|----------------|----------------|-----------------|-----------------|
| C K    | 15.50          | 94.51          | 18.51           | 75.80           |
| O K    | 59.08          | 52.98          | 52.76           | 13.86           |
| Na K   | 17.71          | 16.75          | 25.72           | 9.09            |
| Mg K   | -              | -              | -               | -               |
| Al K   | 2.58           | 0.11           | 0.74            | 3.49            |
| Si K   | 13.96          | 0.28           | 1.75            | 6.05            |
| S K    | 0.78           | 0.05           | 5.77            | 0.17            |
| Ca K   | 0.40           | 1.93           | 0.04            | 0.07            |
| Fe K   | 1.15           | -              | 0.04            | -               |
| Totals | 100.00         | 100.00         | 100.00          | 100.00          |
4. Conclusion
Geopolymers with the variation of Na$_2$SiO$_3$ and NaOH have a significant effect on increasing the strength of the geopolymer and changing the brittle properties to be more ductile. The relatively good geopolymer composite can achieve flexural strength of 58.16 MPa with the content: Silicon dioxide (27% wt), kaolin (51% wt), CaO (5% wt), and carbon fiber (13% wt), with flexural strength 58.16 MPa. The Sodium hidroksida and Sodium silicate content still has the potential to be increased because point F3 is not the optimum level.

Acknowledgements
This work was supported by the DIPA-BLU, LPPM Universitas Lampung, 2019.

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