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To cite this article: J Akmal *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **602** 012079

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Optimization of matrix compositions of Al_2O_3 , SiO_2 , Caolin, and CaO on the mechanical properties of a geopolymer composite with short carbon fiber

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Abstract. Geopolymer is a material synthesized from a base material with a large amount of silica and alumina. Geopolymer can be used as an affordable alternative as a substitute for Portland cement, reducing pollution, and resistant to fire under certain conditions. But pure geopolymer has not been widely used in industrial areas because overall it has low toughness and mechanical properties when compared to metals or ceramics. This research was conducted to produce geopolymer composite material with good mechanical properties and can be applied as a material in making environmentally friendly pipes, by identifying the influence of the composition of the geopolymer base material using the Taguchi method. Generate Geopolymer composites with the addition of Silica Powder and random short carbon fiber have good mechanical properties, from the results of the experiment produced the best flexural strength in T9 specimens with details of the composition of Silica Powder (27 g), Kaolin (55 g), CaO (6 g), and Carbon Fiber (13 gr) with a flexural strength of 41.46924655 MPa and flexural modulus 6 GPa.

1. Introduction

Geopolymer is a material synthesized from a base material with a large amount of silica and alumina. Geopolymer can be used as an affordable alternative as a substitute for Portland cement, reducing pollution, and resistant to fire under certain conditions. But pure geopolymer has not been widely used in industrial areas because overall it has low toughness and mechanical properties when compared to metals or ceramics.

Many studies reveal that geopolymers can be used as composite matrices to improve their mechanical properties. According to Shaikh., (2013) geopolymers that are used as a matrix reinforced with short fibers have flexural strength and good tensile strength without regard to the type of fiber [1]. This was strengthened by a study conducted by Jingkun et al. (2015) that geopolymers reinforced with 5 mm SiC fibers had flexural strength and fracture work which was 5.6 and 63 times higher than pure geopolymers [2]. Also, According to Mukhallad et al., 2018 The addition of fiber increases the strength characteristics of geopolymer composites, for example, the presence of steel fibers and polyvinyl alcohol fibers increases the flexural strength of geopolymer composites of 31.45% (11.116 MPa) and 39.84% (11.825 MPa, respectively) [3].



So far, geopolymer composites are usually used only for building materials, instead of Ordinary Portland Cement (OPC). This research was conducted to produce geopolymer composite material with good mechanical properties and can be applied as a material in making environmentally friendly pipes, by replacing sand/coral with reinforcing fibers. Identifying the influence of the composition of the geopolymer base material using the Taguchi method.

2. Materials and Experiment

2.1. Materials

Materials used in the manufacture of geopolymers are kaolin, silica powder, CaO, carbon fiber, and activators. The chemical composition of K is investigated by XRF (X-Ray Florence) testing, and the respective content is shown in **Table 1**. Another material is commercial silica powder, carbon fiber type medium (1.56 kg / m³), Calcium Oxide (CaO) and activator Sodium Hydroxide (NaOH).

Table 1. Chemical Composition (%) of Kaolin[4]

Sampel	Si	Al	Fe	Ti	Zr	Mn	Sb	Zn	Sn	Ni	Pb	Ga
Kaolin	57,6	37,2	3,82	1,23	0,1	-	-	0,04	-	0,04	-	0,02

2.2. Preparation of Geopolymer Composite

In geopolymer composite preparation, several steps must be prepared. The first step is to prepare a solution of Sodium Hydroxide (NaOH) 14 M, Sodium Hydroxide (NaOH) in the form of dissolved crystals by mixing it with Aquades solution. The next step is to prepare geopolymer forming materials such as Silica Powder (Si), Caolin, Calcium Oxide (CaO), and Carbon Fiber(CF) as seen in **Table 2**. In this study, we used the Taguchi experimental design as seen in **Table 3** to determine the composition of the geopolymer.

Table 2. Matrix compositions used in present research

Level	Si(g) (A)	Caolin (g) (B)	CaO(g) (D)	CF(g) (E)
1	23	60	4	13
2	25	55	6	15
3	27	50	9	18

The nine samples prepared in this study were made with a variety of different compositions referring to Table 2. The orthogonal matrix for the nine samples is shown in Table 3. The samples are symbolized by T1, T2, etc. shown in Table 3.

Table 3. Experimental design taguchi Orthogonal matrix L9(3⁴)

Mix	A	B	C	D
T1	1	1	1	1
T2	1	2	2	2
T3	1	3	3	3
T4	2	1	2	3
T5	2	2	3	1
T6	2	3	1	2
T7	3	1	3	2
T8	3	2	1	3
T9	3	3	2	1

The next step is mixing the powder materials: Silica Powder, kaolin, CaO, and CF until evenly distributed. Then add activator solution, with the ratio of powder to the activator solution is 30%. After stirring evenly, put the mixture into an 80x6x8 mm mold and press at 1200 Psi for about 30 minutes. Geopolymer material is then cured with temperature 30-35 °C for ≥ 28 days.

2.3. Experiment Testing

Rectangular bars with size (80x6x8) mm subjected to the three-point bending test (based on ASTM C1161) to determine the strength and flexural modulus [5], as shown in Fig. 1. The test machine used is MTS Landmark (capacity 100kN) with loading speed of 1.0 mm / min. Flexural strength can be determined by Eq. (1).

$$\sigma_f = \frac{3.P_m.S}{2.W.D^2} \quad (1)$$

Where σ_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, $\left(\frac{\Delta P}{\Delta x}\right)$, using the Eq. (2)

$$E_f = \frac{S^3}{4.W.D^3} \left(\frac{\Delta P}{\Delta x}\right) \quad (2)$$

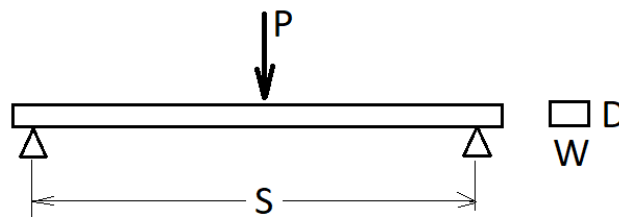


Figure 1. The threepoint bend test

3. Results and discussion

There were nine samples tested, and each sample consisted of three specimens, as seen in **Table 4**. **Figure 2** and **Figure 3** show the best composition (Flexural Strength) is on the composition of T9 which is 41.48 MPa with an average modulus of elasticity of 6 GPa. Composition with the second best value (Flexural Strength) is at T4 which is 32.11 MPa with a modulus of elasticity of 3 GPa. Furthermore, the third best composition with Flexural Strength is at T6 which is 29.67 MPa with a modulus of elasticity of 4 GPa.

Table 4. Flexural Strength Test Results

Eks	Silica	Kaolin	CaO	Carbon Fiber	Average Flexural Strength (MPa)
T1	23	60	4	13	18,54
T2	23	50	6	15	18,26
T3	23	55	9	18	15,37
T4	25	60	6	18	32,11
T5	25	50	9	13	28,26
T6	25	55	4	15	29,66
T7	27	60	9	15	24,75
T8	27	50	4	18	19,53
T9	27	55	6	13	41,47

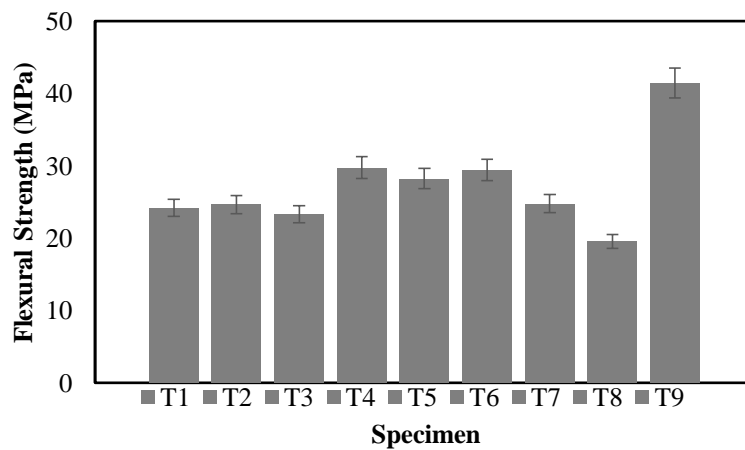


Figure 2. Graph of the results of flexural strength testing

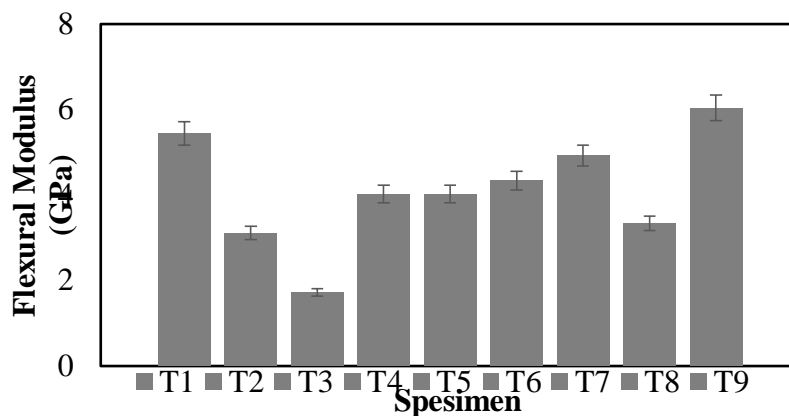


Figure 3. The flexural modulus of the geopolymers composite

Figure 4 is a graph of the effect of each composition on the mechanical properties of geopolymers composites, which in this graph can see the effect of composition based on the average results of geopolymers flexural strength and standard deviation.

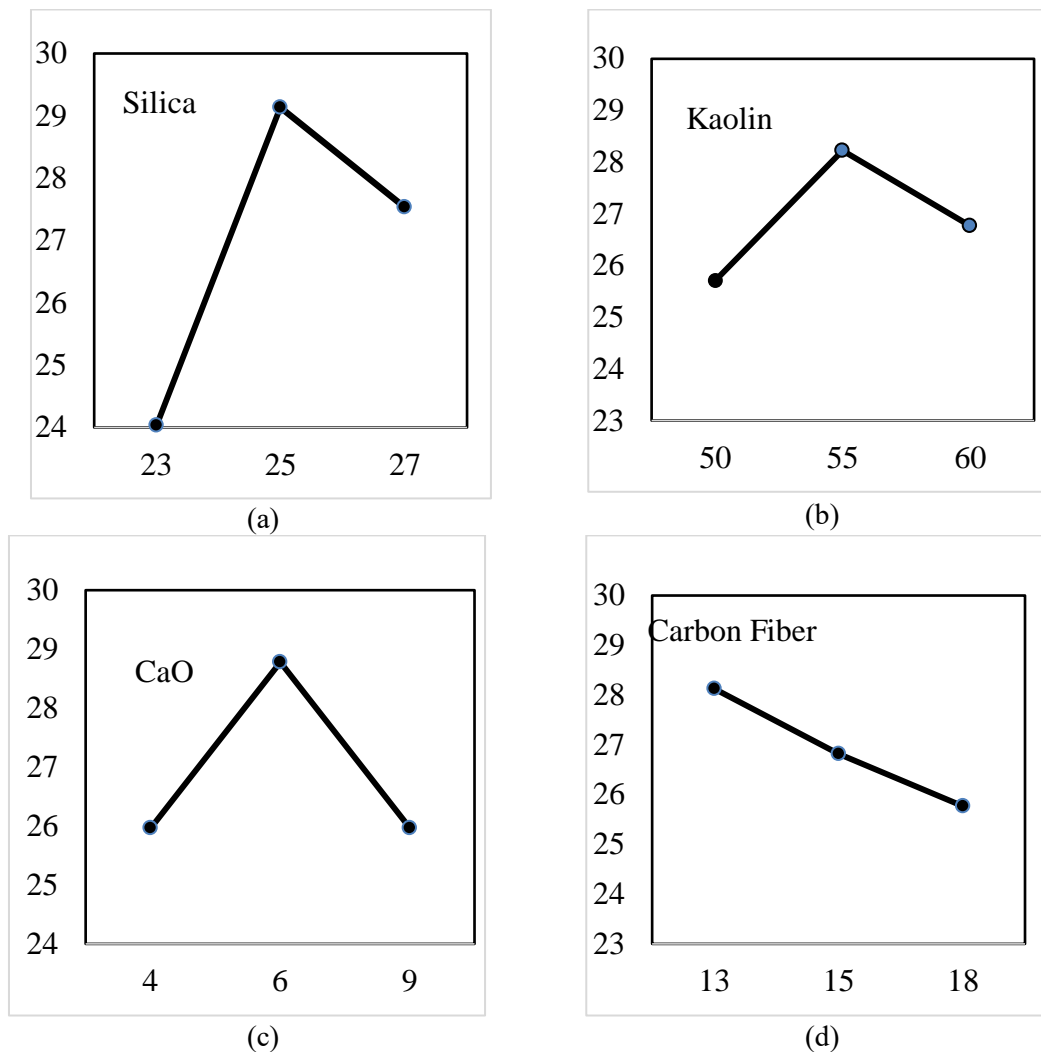


Figure 4. Graph of the effect of each composition on the flexural strength of geopolymer composites (a) Silica Powder, (b) Kaolin, (c) CaO, (d) Carbon Fiber

Table 5. Value of the response S / N ratio Larger the better

Level	Silica	Kaolin	CaO	Carbon Fiber
1	24,03	25,71	25,96	28,12
2	29,13	28,22	28,78	26,81
3	27,53	26,77	25,96	25,76
Delta	5,11	2,52	2,82	2,35
Rank	1	3	2	4

To see the Rank value of each composition of the geopolymer flexural strength can be seen in **TABLE 5**. Silica has the highest influence on the flexural strength of geopolymer composites at level 2, namely on silica with a weight of 25 g, then CaO (Calcium oxide) at level 2 with a weight of CaO

of 6gr, then Kaolin at level 2 with a weight of 55 gr, and the last one is carbon fiber at level 1 with a weight of 13 gr.

The SEM-EDX analysis was used to evaluate the surface morphology of synthesized geopolymer, focusing on particle shape. **FIGURE 5** and **TABLE 6** respectively show the surface morphology of the geo-polymer with a carbon fiber content of 0 and 15%. EDS analysis in the white particle (shown in “B” letter) shows that an element of O, Na, and Si are a large number of compositions.

Secondary electron image (SEI) on surface geopolymer composite from sample T9 is shown in Fig. Xa. Cavities are seen clearly in the image of SEI of Fig. Xa. The carbon fiber shows the low bonding with the matrix. The matrix compromises of Al, Si, Ca oxides is evident with EDX results in the Table. Xa. Also, small particles are attributed to the sodium and calcium oxides, which do not interact with the matrix. These particles are cause debonding carbon fiber in geopolymer composite. To investigation of surface fracture of composite geopolymer, a similar sample T9 is appropriately considered to elucidate the morphology surface fracture of material. The SEI of surface fracture of T9 sample is displayed in Fig. Xb. It can be observed in Fig. Xb that debonding of fiber and cavities are responsible for the weakness of strength composite.

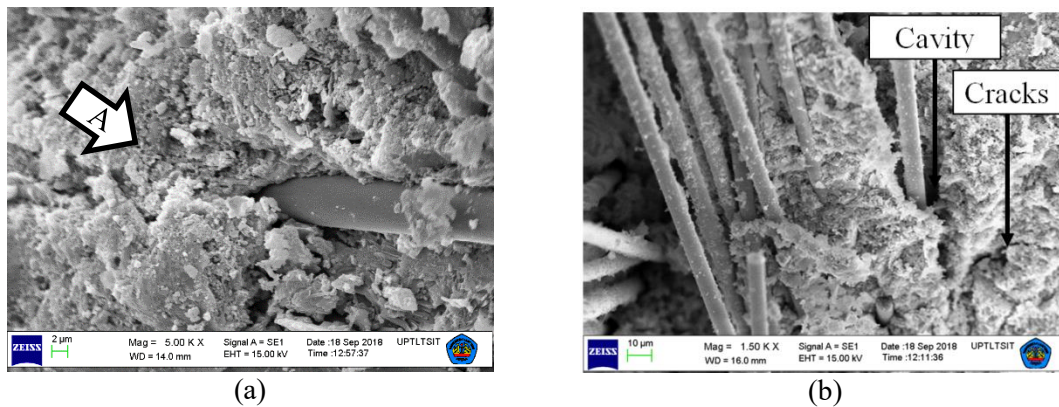


Figure 5. SEI images of (a) surface morphology of the geopolymer composite from sample T9 with EDX results and (b) fractography of sample T9 after the flexural test.

Table 6. Geo-polymer with carbon fiber content

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	25.89	29.87	47.42	4.07
Au	79	M-series	20.20	23.31	3.01	0.86
Si	14	K-series	12.00	13.85	12.53	0.56
Al	13	K-series	9.51	10.98	10.33	0.50
Ca	20	K-series	6.92	7.99	5.06	0.30
Na	11	K-series	6.81	7.86	8.68	0.49
C	6	K-series	5.32	6.14	12.98	1.47
Total:			86.65	100.00	100.00	

4. Conclusion

Geopolymer composites with the addition of Silica Powder and random short carbon fiber have good mechanical properties, from the results of the experiment produced the best flexural strength in T9 specimens with details of the composition of Silica Powder (27 g), Kaolin (55 g), CaO (6 g), and Carbon Fiber (13 gr) with a flexural strength of 41.47 MPa.

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