Enhancement of Mechanical Properties of Geopolymer-TiO₂ Membrane by Carbon Fiber Reinforcement

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**Abstract.** This study was conducted with the aim of determining the effect of adding carbon fiber to the TiO₂ geopolymer membrane on increasing the compressive strength and flexural strength of the membrane. In this study, geopolymer membranes were synthesized from kaolin calcined for 2 hours at a temperature of 650 °C, sodium silicate, NaOH, TiO2, H2O2, and carbon fiber. The variations of carbon fiber added to the geopolymer were 1.5, 2.5, and 3 cm². The optimum results of adding carbon fiber will be applied to the geopolymer-TiO2 membrane, with TiO2 variations of 2.5, 5, and 10% of the weight of the geopolymer. Characterization of the geopolymer-TiO₂ membrane was carried out through XRD, FTIR, compressive strength, and flexural strength tests. From the results obtained, carbon fiber gave positive results to the geopolymer and geopolymer membranes. The optimum compressive strength of geopolymer with the addition of carbon fiber with an area of 2.25 cm² is 16.19 MPa, increasing by 25% compared to geopolymer without carbon fiber and decreasing by 31.05% after the addition of 2.25 cm² of carbon fiber. Meanwhile, the geopolymer-TiO₂ membrane with the addition of carbon fiber increases the compressive strength of the membrane up to the addition of 10% TiO₂, which is 1.39 MPa compared to without the addition of carbon fiber. The optimum yield and flexural strength obtained for the 10% TiO₂ geopolymer membrane are 3.192 MPa. Thus, the results of the study indicate that the addition of carbon fiber to the TiO₂ geopolymer membrane can increase the compressive strength and flexural strength; this is considered an important step in the development of more durable but porous geopolymer membranes.

Keywords: *membrane, geopolymer, compressive strength, flexural strength*

# INTRODUCTION

Membrane is one of the technologies applied in separation processes such as pharmaceuticals, food processing, seawater desalination, and wastewater treatment. Based on the raw material, membranes are divided into organic and inorganic membranes. Inorganic membrane have advantages over organic ones because of its long-term stability, compatibility, and resistance to harsh environments (1). Inorganic membrane is difficult to prepare due to high temperatures requirement and expensive processing costs. (2). This challenge can be addressed by the application of geopolymer.

Geopolymer is an amorphous material that is easy to synthesize, has a high availability of raw materials, is low in price, and can be applied in separation processes with high efficiency (3). In membrane applications, the innovation in geopolymer development is creating porous geopolymers (4). The porosity of geopolymer membrane can be fabricated through physical and chemical foaming that resulting in open and close pores (5). However, porous geopolymer is known to have weak mechanical strength, reducing its applicability as membrane (6).

To overcome this, various fibers are used as reinforcement to improve the compressive strength and flexural strength of porous geopolymer membranes (7) by increasing plastic behavior and shrinkage resistance. Steel fibers are widely used in the construction industry; however, due to their high strength, low density, and resistance to corrosion, carbon fibers (CF) are preferred for structural repair and upgrading, especially in the construction field, despite their high cost (8). Studies show promising prospects for the future with CF-reinforced GPMs. Carbon fiber is the most employed reinforcement in geopolymer membrane. This synthetic fiber exhibits high tensile strength and Young’s modulus (7). In the previous study, the amount of carbon fiber added to the geopolymer composite is 1–2% of the weight of the geopolymer mass, or less than 1.0% of the volume (9). This amount effectively increased the mechanical properties of the composite as well as its ductility (10). A study was conducted on metakaolin-based geopolymers with the addition of slag and carbon fiber composites (11). The flexural strength of the composites was tested with 1.0 wt% carbon fiber with a diameter of 10 μm and a length of 7 mm. The research results show that there is reinforcement in the composite. For the pure matrix, the bending strength was

6.9 MPa, and for the material with carbon fiber, it was 11.7 MPa. Carbon fiber also reportedly adds a compressive strength of 234.2 MPa (12). The addition of carbon fiber of more than 1% by volume can reduce the ratio of length to diameter, and high carbon is not capable of pressure (13). In addition, the addition of carbon fiber to 1.6% of the fraction can reduce thermal conductivity and water absorption by 39% (14).

Efforts to increase the compressive and flexural strength of geopolymer membranes are not only produced by carbon fibers. The addition of photocatalyst material as a filler provides an important contribution to the geopolymer formation process. Photocatalysts will diffuse through microcavities during the geopolymer formation process so that adhesion occurs between particles in the matrix (15). This causes the geopolymer to become denser, increasing its flexural and compressive strength. (16) reported research on geopolymers composited with TiO2 using a geopolymer mixing method that can increase compressive strength by 17-41%. The authors reported that the addition of up to 5 wt% improved mechanical properties and durability and attributed this behavior to the refinement of pores and compaction of the geopolymer microstructure (17). Similarly, Yang et al (6) studied the mechanical and physical behavior of alkali-activated slag added with TiO2 nanoparticles at 0.5 wt% and reported a decrease in porosity and shrinkage of the material; however, a percentage of more than 10% TiO2 reduced the fluidity and hardening time of the geopolymer (18). TiO₂ has advantages over other photocatalysts. All previous studies have focused only on geopolymers as building materials, but there has been no research on the addition of carbon fiber and TiO₂ to geopolymers as membranes. TiO₂ was chosen as a filler because it has self-cleaning properties that, when applied to the membrane, can reduce fouling compared to other photocatalyst materials (19).

This study focuses on the addition of carbon fiber and TiO2 to produce the optimum compressive strength and flexural strength. The synthesis was carried out by adding carbon fiber to the geopolymer with variations of 0, 1.5; 2.25; and 3 cm2. The optimum results for the compressive strength of the geopolymer were then applied to the geopolymer membrane and added with TiO2, with variations of 2.5; 5; and 10 wt%. The characteristic of geopolymer membrane was analysed by FTIR and XRD. The geopolymer membrane were tested using compressive and flexural strength.

# EXPERIMENTAL SECTION

## Materials

Kaolin as the aluminosilicate source was obtained from Bangka Belitung, Indonesia. sodium hydroxide (NaOH) flakes p.a purchased from Merck, Sodium silicate (waterglass/Na2SiO3) (PT. Brataco, 28,79% SiO2, 19,35% Na2O, dan 28, 94% H2O) H2O2 30% p.a from Merck, carbon fiber, demineralized water (aqua DM), TiO2.

## Methods

**Activator Base Manufacturing**

The procedure for making the activating base solution refers to research conducted by Faradilla (2021), namely mixing 10 M NaOH solution with sodium silicate (waterglass/Na2SiO3). The NaOH solution was made by dissolving 40 g of solid NaOH in 40 mL of aqua DM, then the solution formed was diluted with aqua DM until the volume reached 100 mL. Making the NaOH solution is exothermic, so it is left for 24 hours until the temperature returns to room temperature. Next, 25 g of NaOH solution was added to the sodium silicate solution (waterglass/Na2SiO3) and stirred until homogeneous. This mixing reaction is also exothermic, so it requires the same treatment as making a NaOH solution.

## Geopolymer synthesis with variations of carbon fiber

Geopolymer synthesis was carried out by adding the activating base solution to 40 g of metakaolin which had been calcined at 650 °C, then stirring for 4 minutes until homogeneous and a thick paste was formed. In this study, the addition of carbon fiber was varied as tabulated in Table 1. The printed geopolymer is left at room temperature until it hardens (24 hours). After hardening, the geopolymer is cured by heating at 55 °C for 24 hours. After the curing process, the geopolymer was left at room temperature for 28 days for compressive strength testing.

**Table 1.** variations in the addition of carbon fiber

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Materials | Carbon | fiber (cm2) |  | |
|  | V0 | V1 | V2 | V3 |
| Metakaolin (g) | 40.0 | 40.0 | 40.0 | 40.0 |
| NaOH 10M (g) | 25.0 | 25.0 | 25.0 | 25.0 |
| Na2SiO3 (g) | 25.0 | 25.0 | 25.0 | 25.0 |
| Carbon fiber (cm2) | 0 | 1.5 | 2.25 | 3.0 |

## Synthesis of Geopolymer-carbon fiber membranes with variations of TiO2

The geopolymer composition with the highest compressive strength was then selected for the synthesis of the geopolymer membrane. The geopolymer membrane was synthesized by adding 40 g of metakaolin

TiO2 and stirring with a mixer for 1 minute, varying the amount of TiO2 added by 0; 2.5; 5; 10% wt (20) of geopolymer mass. Then add 50 g of the activating base solution to the mixture and stir for 4 minutes using a mixer until a paste forms. A 7% H2O2 solution was added to the paste, as much as 3% of the total mass of the geopolymer, and stirred for 2 minutes until homogeneous. The paste formed is then poured into the mold until it fills half the mold. The carbon fiber is then placed on top of the paste layer before being covered again with geopolymer paste.

**Table 2.** Photocatalyst Geopolymer Membrane Composition with Carbon Fiber

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Materials | Addition of TiO2 |  | | |
|  | control | V2T1 | V2T2 | V2T2 |
| Metakaolin (g) | 40.0 | 37.75 | 35.5 | 31.0 |
| NaOH 10M (g) | 25.0 | 25.0 | 25.0 | 25.0 |
| Na2SiO3 (g) | 25.0 | 25.0 | 25.0 | 25.0 |
| Carbon fiber (cm2) | 0 | 2,25 | 2,25 | 2,25 |
| TiO2 (g) | 0 | 2.25 | 4.5 | 9.0 |
| H2O2 3% (g) | 2.7 | 2.7 | 2.7 | 2.7 |

## Characterization

The phase and crystallinity of the geopolymer membrane were analyzed using X-Ray Diffraction (XRD, Xpert Panalytical) with a Cu Kα light source (λ = 1.5406 Å). Compressive strength was tested by UTM (Universal Testing Machine) machine with a test object measuring 24.0 × 24.0 × 24.0 mm which had been aged for 28 days at room temperature. The flexural strength test was conducted based on the ASTM C328 standard with the dimension of sample as follow: 80.0 mm long, 18.91 mm wide, and 5 mm thick which has also been aged for 28 days at room temperature. Universal testing machine, TARNO GROCKI, UPH- 100 kN, was applied using three-point bending at a speed of 2 mm/s. The functional group of geopolymer was observed using Fourier transform infrared spectroscopy (FTIR, Shimadzu Instrument Spectrum One 8400S).

# RESULTS AND DISCUSSION

## Geopolymer Compressive Strength

The geopolymer compressive strength test was used to determine the influence of carbon fiber addition. The results of the compressive strength test were shown in Figure 1. The optimum compressive strength results were obtained with the addition of 2.25 cm² of carbon fiber. The addition of carbon fiber with an area of 1.5 cm² did not yield optimum results but did show some improvement. This condition was because the interzone bonds were not strong enough due to the small amount of carbon fiber, causing the geopolymers to still retain metakaolin properties. The increased compressive strength was caused by the hydrophilic nature of carbon fiber, allowing high fiber/matrix interactions (15). With the addition of carbon fiber with an area of 3 cm², there was a sharp decrease in compressive strength. However, adding too much carbon fiber reduced the bond between the matrix and aggregate. (16) and also caused agglomeration due to van der Waals forces

(21). The phenomenon of clumping can be caused by several causes including too short mixing time, spindles that are not suitable for mixing certain types of fibers. In this case, the mixing time should be longer than for natural fibers or fibers made using other technologies(22).

18



16.19

14.9

13.32

11.35

16

14

Compressive Strength (MPa)

12

10

8

6

4

2

0

0 1 2 3

Carbon Fibre (cm2)

**Figure 1**. Compressive strength of geopolymer membrane with variations in the addition of carbon fiber

## The influence of TiO2

The geopolymer membrane, enhanced with TiO₂ and carbon fiber, was tested for compressive strength. The carbon fiber added to the geopolymer membrane was 2.25 cm², based on the strong results of the pressure tests on previous geopolymers. The compressive strength test on the geopolymer membrane, shown in Figure 2, indicated a decrease in compressive strength. This was caused by the presence of pores in the geopolymer; the higher the number and the larger the pores, the weaker the geopolymer strength became. The addition of 2.5% TiO₂ generated 1.02 MPa compared to the geopolymer without the addition of TiO₂. Overall, the compressive strength increased as the amount of TiO₂ increased. The addition of 5% TiO₂ produced 1.2 MPa, and 10% TiO₂ produced 1.39 MPa. In general, the compressive strength of geopolymer membranes with TiO₂ increased due to the presence of TiO₂ as a filler, which improved the structure and accelerated initial hydration by quickly dissolving the geopolymer (23). Furthermore, the addition of TiO₂ caused the formation of stronger C-S-H groups, increasing the density and homogeneity of the geopolymer matrix with higher distribution (24). From Figure 2, it can be seen that the compressive strength value increased with the increasing TiO₂ composition added to the membrane.

1.6



1.39

1.26

1.2

1.06

1.02

1.4

1.2

Compressive Strength (MPa)

1.0

0.8

0.6

0.4

0.2

0.0

GM GM+CF 2,5% TiO2+CF 5% TiO2+CF 10% TiO2+CF

Geopolymer Membrane

**Figure 2.** Compressive strength of geopolymer membranes with variations in the addition of TiO2

## Geopolymer Membrane Flexural Strength Test

Geopolymer membrane compressive strength testing was carried out after the membrane was 28 days old using the ASTM C328 procedure. The results obtained showed that the addition of carbon fiber to the membrane gave higher results than without carbon fiber. The flexural strength data of the geopolymer membrane showed a rise of 20% when carbon fiber was added. The increase in flexural strength resulted from the addition of carbon fiber improving membrane ductility (25)**.**

The addition of carbon fiber prevented the formation of microcracks when a load was applied since the geopolymer had a brittle structure. Figure 3 shows that the addition of TiO₂ increased the strength of the slide. However, the addition of 2.5% TiO₂ resulted in a decrease in flexural strength to 1.27 MPa due to the uneven dispersion of TiO₂, which caused aggregates to form, thereby reducing the strength of the matrix (17). It is possible that the decrease occurs because the binding phase is still insufficient to embed new particles in the matrix, resulting in lower cohesion (26). The addition of 5% and 10% TiO₂ significantly increased the flexural strength to 1.68 MPa and 3.192 MPa, respectively, owing to denser pore formation. This was because the open pores of the geopolymer membrane were closed by the presence of TiO₂, making the structure denser. This denser structure results in lower membrane permeability and porosity compared to TiO2 (24). In addition, TiO₂ accelerated the formation of gel nucleation of NASH and CSH compounds, resulting in a clearer and stronger structure (27).

5



4.992

3.984

3.192

1.68

1.272

4

Flexural Strength (MPa)

3

2

1

0

GM GM+CF 2.5% TiO2 + CF 5% TiO2 + CF 10% TiO2 + CF

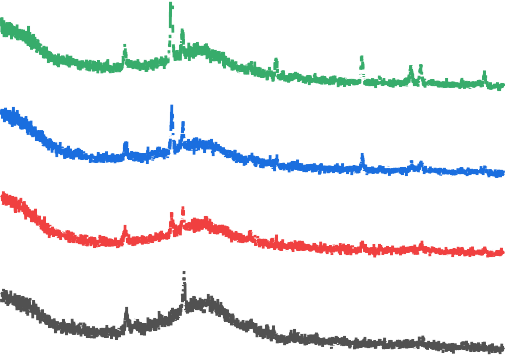
Geopolymer Membrane

**Figure 3.** Flexural strength of geopolymer membranes with variations in the addition of TiO2

## Geopolymer Membrane Phase Analysis

The diffractogram of the geopolymer membrane was depicted in Figure 4. The feature at 2θ = 20 - 40° indicated the amorphous structure of the geopolymer membrane resulting from the formation of a gel structure from aluminosilicate (28). Each geopolymer membrane had a sharp peak at around 2θ = 20° and 28°, inferring the presence of quartz crystals (SiO₂) that remained unreacted in alkaline environments during the geopolymerization process (29). Additionally, the typical peak of the geopolymer membrane near 2θ = 34° was identified as the peak of the Na₂CO₃ phase. Sodium carbonate (Na₂CO₃) was the result of the reaction of Na⁺ ions with CO₂ in the air. This reaction was based on the calculation of a high Na₂O/Al₂O₃ mole ratio (> 8). The effect of adding TiO₂ showed a typical peak at 2θ = 25°, which corresponded with the standard TiO₂ diffractogram (JCPDS number 21-1272) where the TiO₂ diffraction pattern of the anatase phase position 2θ was at 25.281° (30) (31). The intensity of the diffractogram in anatase increased with the increasing addition of TiO₂ to the membrane (32). Other observations showed that increasing the addition of TiO₂ to the membrane also increased crystallinity (33). Additionally, the effect of adding carbon fiber to the geopolymer was not very significant to the diffractogram. This indicated that the carbon fiber content in the geopolymer was not

dissolved by the alkaline activating solution. This result was in accordance with research by Novais et al. 2016 (34), which also showed that the addition of carbon fiber had no effect on the X-ray diffractogram results.



Geopolymer Membrane + 10% TiO2

Geopolymer Membrane + 5% TiO2

Geopolymer Membrane + 2,5% TiO2

Geopolymer Membrane

0 10 20 30 40 50 60 70

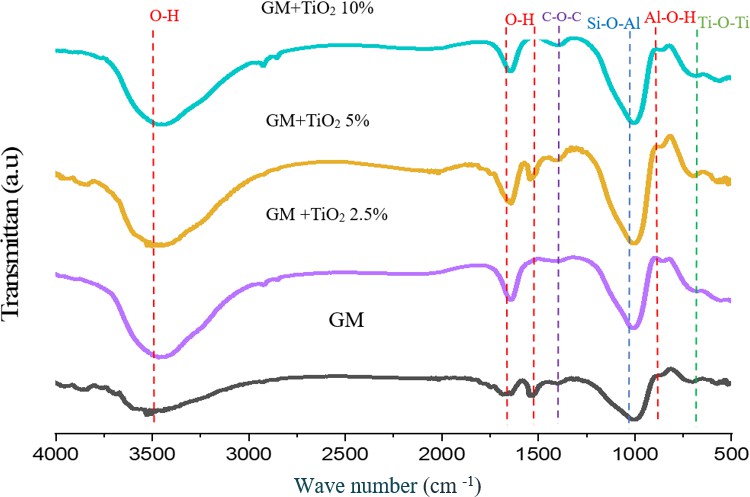
2θ(°)

Intensity (a.u)

**Figure 4.** Geopolymer Membrane Difactogram

***Bond and Functional Group Analysis***

The FTIR spectra of the geopolymer membrane are illustrated in Figure 5. In the geopolymer membrane with TiO2, there was no significant difference between the geopolymer membrane without addition and the addition of 2.5%, 5%, or 10% TiO2. The characteristic band located at 1089 cm−1 was due to the asymmetric stretching of the Si-O-T band (T is an Al or Si tetrahedron), which was associated with the lower intensity of the Si-O-Si band. This indicated significant changes in the chemical environment of Si and Al that occurred during geopolymerization to produce new aluminosilicate gel products (35).



**Figure 5.** FTIR Spectra of Geopolymer Membranes

The band located around 703 cm−1 indicated the presence of Ti-O-Ti photoactive species and also resulted in an overlap with the amorphous Al-O band, while also showing a decrease in intensity due to gel formation. The higher frequency shifts in the 3436 and 1650 cm−1 bands, which corresponded to the vibrations of the O- H and H-O-H groups, represented an increase in the hydrophobicity of the material as a result of the increase in TiO2 additive content. In this study, there was a small absorption peak at a wavenumber of around 1350– 1400 cm−1, indicating the presence of efflorescence, namely the formation of O-C-O bonds in the geopolymer membrane due to the porous structure of the geopolymer membrane (36), his certainly affects the stability of the membrane in the separation process. Where the porosity is high, then the flux will be high; the relevant parameters that need to be taken into account for efflorescence control are by taking into account the molar ratio of Na2O /Al2O3 (37). This process was not caused by the H2O2 reaction but by the presence of water vapor from solution migration. The many cavities in the pores migrated alkaline solutions (Na+ ions) to the surface and could react with air to form carbonate (Na2CO3).

# CONCLUSION

The Geopolymer-carbon fiber-TiO2 membrane was successfully synthesized. the addition of carbon fiber as reinforcement agent improved the flexural strength and compressive strength of the geopolymer membrane. the results obtained show that the optimum addition of geopolymer is carbon fiber with a size of 2.25 cm2. the addition of TiO2 10 wt% generated the highest compressive strength and flexural strength increase of 13 and 20%, respectively. In the future, this geopolymer membrane can be applied to membrane-based liquid waste processing, and it is necessary to consider filler materials other than TiO₂, namely perhaps the Si/Al ratio.

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