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Self-healing Performance of Geopolymer Mortar with Polypropylene Fiber and Bacteria

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ABSTRACT

Fibre-Reinforced Cementitious Composites (FRCCs) have been extensively used as repair materials in the construction industry. The presence of the fibers can enhance not only the physical and mechanical properties of the material, but it also has the capacity to minimize and control the microcracks in the cementitious composite. Geopolymer is a sustainable material that can be used as a replacement for concrete in a structure. Similar to concrete, geopolymer is a brittle material, which makes it vulnerable to microcracks. The combination of fiber and bacteria would be a promising solution to address this problem. However, there are a limited number of studies that explore the potential application of bacteria in geopolymer mortar as a repair material. Hence, this study was conducted in order to determine the mechanical performance of bio-geopolymer with polypropylene fiber in terms of the following parameters: strength regain, damage degree, and self-healing percentages. A polypropylene fiber with varying content was used in the fly ash-based geopolymer mortar mixtures. The result of the experiment revealed that polypropylene fibers do not have a significant effect on the compressive strength of the geopolymer mix since the percentage increase is only 3.59%. The inclusion of bacteria and polypropylene fiber in geopolymer has the potential to be used as a self-healing material since the strength-regain ratio is 199.97%. Hence, geopolymer mortar with polypropylene fiber and bacteria can be used as a repair material.

INTRODUCTION

The word geopolymer was introduced by Davidovits in 1978. He is considered the inventor and father of geopolymers. It is formed by mixing aluminosilicate source materials (precursors), e.g., fly ash (FA), ground-granulated blast-furnace slag (GGBS),

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and metakaolin, with alkaline activators, e.g., sodium or potassium-soluble silicates, together with water and possibly aggregates to form hardened concrete-like materials with similar mechanical properties (Duxson et al., 2007). In comparison with concrete, geopolymers emit less carbon dioxide into the environment and possess lower embodied energy. Geopolymer concrete can be formed by replacing the precursor with cement. The Geopolymer Concrete (GPC) sample prepared with sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) showed better mechanical, durable, and microstructural properties (Pimraksa, 2011). Similar to the traditional concrete, geopolymer concrete is a brittle material with a low tensile strength, which makes it vulnerable to microcracks. To overcome these drawbacks caused by brittleness, fibers have been incorporated into the cementitious matrix as reinforcement, creating fiber-reinforced cementitious composites (FRCCs) (Yao et al., 2020). The role of the fiber is to serve as a bridge over the microcracks caused by the stress and prevent these cracks from propagating within the composite material, like concrete and geopolymer. Fibers enhanced the brittle behavior of the geopolymer matrix into a ductile one with improved mechanical strength and residual impact strength (Samal et al., 2015). The fiber reinforcement can be characterized by differences in types (steel, synthetic, inorganic, and natural fibers), content, aspect ratio (fibre length divided by fiber diameter), geometry, and physical and mechanical properties (Larsen and Thorstensen, 2020). One of the fibers that can be used to reinforce the geopolymer material is polypropylene. Polypropylene fiber can offer a bridging effect over the pores and defects and change the expanding ways of cracks, resulting in a great improvement in strength and toughness (Zhang et al., 2009).

There are few studies that explore the healing potential of bacteria in geopolymers. Most of the researchers used fly ash as the precursor, applied the direct addition of bacilli as a microbial agent, and almost all of them conducted destructive tests. De Koster et al. (2015) encapsulate the bacteria and nutrients with a geopolymer coating. They found out that the metakaolin coating material can interact sufficiently inside the concrete mixture with a compressive strength of 29.85 MPa. It is interesting to note that adding the bacteria directly to the geopolymer matrix without encapsulation is also possible. This statement was proven by the study by Jadhav et al. (2018), in which the cracks were sealed with calcium carbonate using Sporosarcina pasteurii in a metakaolin-based geopolymer. Aside from metakaolin, fly ash can also be used as a precursor. Dwi Wulandari et al. (2018) found out that microbial agents can be used in fly-ash-based geopolymers as well as Portland cement paste. Doctolero et al. (2020) discovered that co-culturing Bacillus sphaericus and Bacillus thuringiensis bacteria can significantly improve the self-healing efficiency of geopolymer material, and the maximum crack width was 0.65 mm. Granulated blast furnace slag (GBFS) was utilized by Ekinci et al. (2022) as a precursor with Bacillus subtilis, and it was discovered that a 1% bacteria/binder ratio should be employed in order to increase the compressive strength by 118% in comparison to the control samples.

For this study, the researchers investigated the effects of the incorporation of polypropylene fibers and bacteria into geopolymer mortar by evaluating the compressive strength and ultrasonic pulse velocity (UPV). The two types of bacteria employed in the study were Bacillus megaterium and Bacillus subtilis. The self-healing ability of the bacteria was tested by inducing artificial cracks and observing the changes in compressive strength, damage degree, and self-healing percentages. To the knowledge of the authors, there were no studies undertaken on the use of fly-ash-based geopolymer mortar with varying amount of polypropylene fiber and bacteria.

2. MATERIALS AND METHODS

2.1 Raw Materials

Fly ash was acquired from Pozzolanic Philippines Inc. (PPI), and it was categorized as Class F (low in calcium) in line with the test specification of ASTM C618-19.

Alkaline Solution. The alkaline activator (AA) solutions utilized in the study were sodium hydroxide flakes (NaOH), potassium hydroxide flakes (KOH), and sodium silicate solution (Na_2SiO_3). All of the batch mixtures contained NaOH and KOH at a molar concentration of 12 M.

Fine aggregates (sand) and water. The graded standard sand was acquired from a nearby hardware store and met the requirements and standards listed in ASTM C778 for graded standard sand. Ordinary tap water was used to mix the mortar.

Bacteria. Bacillus Subtillis and Bacillus Megaterium were acquired from the Philippine National Collection of Microorganisms in Laguna, Philippines.

Polypropylene fiber. A 12-mm-long polypropylene fiber (Fibrin 23) was bought from Tertex International Philippines Inc. The polypropylene fiber percentage range of 0.25% to 0.75% was utilized in accordance with the prior study of Nematollahi et al. (2018).

2.2. Mix Design Formulation and Geopolymer Preparation

Various geopolymer combinations were prepared in batches in sextuplicate with variable amounts of polypropylene and bacteria as shown in Table 1. Three specimens were used to determine the ultimate compressive strength and the remaining three were used to pre-crack the specimens at 60% of the ultimate compressive strength capacity. The results of a prior work by Quiatchon et al. (2021) were used as a reference for batch formulation.

Table 1. Batch formulation of the different geopolymer mixtures with varying amounts of polypropylene (PP) and bacteria

Batch code	Content	Fly-ash (%)	Sand (%)	PP (%)	NaOH (%)	KOH (%)	Na_2SiO_3 (%)	Water (%)
PP0	Geopolymer with 0%PP, bacteria free	43.3	30.7	0.0	4.9	1.6	13.0	6.5
PP1	Geopolymer with 0.25%PP, bacteria free	43.3	30.7	0.25	4.9	1.6	13.0	6.5
PP2	Geopolymer with 0.5%PP, bacteria free	43.3	30.7	0.50	4.9	1.6	13.0	6.5
PP3	Geopolymer with 0.75%PP, bacteria free	43.3	30.7	0.75	4.9	1.6	13.0	6.5

BP0	Geopolymer with 0%PP and bacteria	43.3	30.7	0.00	4.9	1.6	13.0	6.5
BP1	Geopolymer with 0.25%PP and bacteria	43.3	30.7	0.25	4.9	1.6	13.0	6.5
BP2	Geopolymer with 0.5%PP and bacteria	43.3	30.7	0.50	4.9	1.6	13.0	6.5
BP3	Geopolymer with 0.75%PP and bacteria	43.3	30.7	0.75	4.9	1.6	13.0	6.5

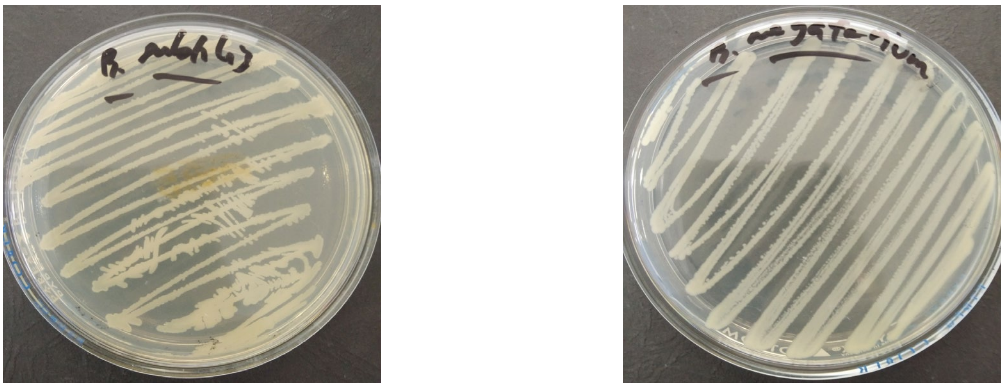
The following raw materials, such as fly ash, sand, polypropylene fiber, potassium hydroxide (KOH), and sodium hydroxide (NaOH) flakes, were weighed beforehand with an automatic balance for preparation in the production of geopolymer mortar. Both NaOH and KOH flakes were dissolved in water separately in a plastic basin and placed over an ice bath while stirring for ten (10) minutes. The dissolved NaOH and KOH flakes were added to the sodium silicate solution to prepare the alkaline solution, which was then stirred for fifteen (15) minutes.

Fly ash, sand, and polypropylene fiber were mixed for two (2) minutes manually using a spatula. After that, using a JJ-5 cement mortar mixer, dry materials such as fly ash, sand, and fiber were mixed with water and an alkaline solution (NaOH, KOH, and sodium silicate) for ten (10) minutes. Lastly, two different bacterial solutions (B. subtilis and B. megaterium) in the microcentrifuge tube were added directly to the geopolymer mortar mixtures.

After mixing all the raw materials, the geopolymer mortar was then poured into two (2) sets of plastic square molds with three compartments measuring 50 mm by 50 mm by 50 mm. The demolded samples were kept in an undisturbed with a relative humidity of 50±10% and an ambient temperature of 30 to 35 °C.

2.3 Culture Maintenance and Induction of Sporulation

Bacterial cultures were streaked on Nutrient Agar (TM Media) plates, incubated at 37° C for 18 to 24 hours, and then stored at 4 to 8° C. The bacterial cultures were bought from the National Institute of Molecular Biology and Biotechnology (BIOTECH-UPLB). Bacillus subtilis and Megaterium's streaked agar plates are shown in Figure 1.



(a) (b)

Fig. 1 Streaked agar plates of (a) Bacillus subtilis and (b) Bacillus megaterium

A sterile saline solution standard at 0.5 McFarland Standard was used to inoculate cultures that were seven days old, and 1 mL was then aliquoted into microcentrifuge tubes. To promote sporulation, the samples were first immersed in an ice bath for 5 minutes after being in a dry bath at 80° C for 10 minutes. Samples were utilized right away to stop germination after the confirmatory test of Schaeffer-Fulton staining (Doctolero et al., 2020).

2.4 Test procedure

2.4.1 Compressive Strength

The capacity of a material to endure the maximum applied load before failing is referred to as its compressive strength. The unconfined compressive strength of each individual specimen may be estimated using Eq. (1) by dividing the maximum compressive force recorded by the testing instrument by the specimen's perpendicular surface area.

$$Unconfined\ Compressive\ strength = \frac{P}{A} \quad (1)$$

P is the maximum applied load and A is the perpendicular surface area of the specimen.

A MATEST S.p.A. Treviolo (Model: E161PN279) with a capacity of 250 kN and a loading rate of 1.2 KN/s was used to carry out the unconfined compressive strength (UCS) test. ASTM C109/C109M was used as a reference for the specification and procedure for the compressive strength test.

2.4.2 Ultrasonic pulse velocity (UPV)

The UPV test was conducted on cubical specimens after 28 days of ambient curing using ultrasonic pulse velocity equipment with 54 kHz transducers (UPV, Pundit Lab+ CT-133, Proceq, Schwerzenbach, Switzerland). This was done in line with ASTM C597-16. Three readings were typically recorded for each batch of geopolymer mortar using the direct UPV method. Both before and after the cracks were generated, the UPV values of cubical specimens were obtained. The UPV values were likewise determined after 14 days of healing period.

In this study, the damage degrees of fly-ash-based geopolymer mortars with polypropylene and bacteria were calculated using Eq. (2). Equations 2 and 3 were takenfrom the study conducted by Tanyildizi et al. (2022).

$$DD = 1 - \left(\frac{V_1}{V_2}\right) \tag{2}$$

DD is damage degrees of geopolymer mortars in Eq. (1). V1 is the UPV after the cracking, and V2 is the UPV before the cracking. Also, the self-healing percentages of geopolymer mortars were calculated using Eq.3.

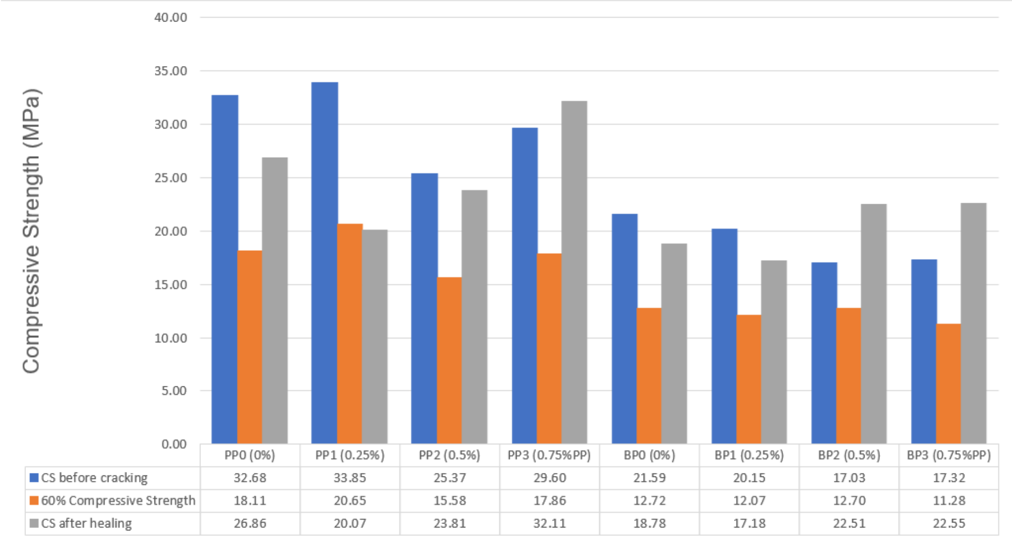
$$H = \frac{(V_3 - V_2)}{V_2} \text{x} 100 \tag{3}$$

The V3 in Eq. 2 is the UPV of geopolymer mortar after the healing period.

3. RESULTS AND DISCUSSION

3.1 Compressive Strength

A total of 48 cubical geopolymer mortar samples were tested using a UTM machine, in which half of them were subjected to ultimate compressive strength for 28 days and the other half to 60% of the ultimate compressive strength. After a healing period of 14 days immersed in water, all the pre-cracked batches were subjected again to the ultimate compressive strength test.



	PP0 (0%)	PP1 (0.25%)	PP2 (0.5%)	PP3 (0.75%PP)	BP0 (0%)	BP1 (0.25%)	BP2 (0.5%)	BP3 (0.75%PP)
CS before cracking	32.68	33.85	25.37	29.60	21.59	20.15	17.03	17.32
60% Compressive Strength	18.11	20.65	15.58	17.86	12.72	12.07	12.70	11.28
CS after healing	26.86	20.07	23.81	32.11	18.78	17.18	22.51	22.55

Fig. 2 Compressive strength of Geopolymer Mortar with varying amount of Polypropylene and Bacteria at 28 days

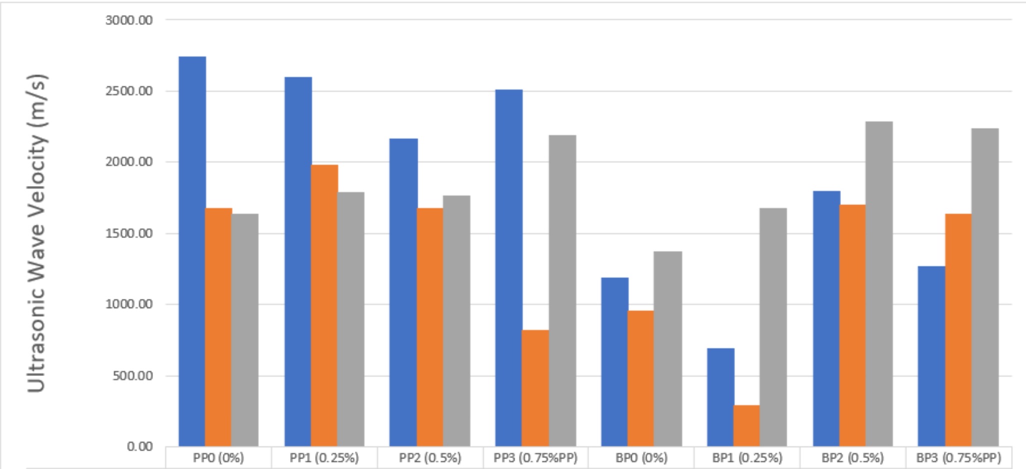
The variation in compressive strength of geopolymer mortar mixtures with polypropylene and bacteria at 28 days is shown in Figure 2. The highest average compressive strength was attained by geopolymer batch PP1 (without bacteria) with 0.25% polypropylene, while geopolymer batch BP2 (with bacteria) with 0.5% polypropylene had the lowest average compressive strength of 17.03 MPa. The results of the compressive test are similar to those of Nematollahi et al. (2018), who reported that geopolymer mortar with 0.25% polypropylene fibers had the highest compressive strength, reaching more than 30 MPa. This suggests that, following the action of the compressive force in the perpendicular direction, a substantial amount of the fibers can assist in holding the geopolymer matrix intact within the microcracks. However, subsequent increases in fiber content led to a reduction in compressive strength of 25% for PP2 (0.5%) and 12% for PP3 (0.75%) in comparison to PP1. The hydrophobic properties of the polypropylene fiber, which led to weak attachment of the fiber to the geopolymer matrix, are one potential cause of the decline in compressive strength. Furthermore, this might be the result of a fiber-induced increase in the amount of trapped air, which would enhance the mixture's porosity (Nematollahi et al., 2016).

Comparing the two batches (PP0 and BP0) without polypropylene fibers, it can be observed that the addition of the bacteria in the geopolymer mortar decreased the compressive strength by 33.94%. However, the strength regain ratio is more prevalent in geopolymer batch with bacteria relative to the geopolymer batch without bacteria. The average strength regain of BP3 was the highest with a value of 199.97%, whereas PP1 had the lowest average strength regain with a value of 97.21%. The strength-regain ratio of the geopolymer material with bacteria is significantly affected by the addition of polypropylene fiber at 0.75% in geopolymer mortar since the difference compared to the control is 52.35%. Nevertheless, geopolymer mortar with bacteria has the potential to be used as a repair mortar since its compressive strength value is at least 15 MPa with an R2 classification as defined by Ducman et al. (2018).

It is also interesting to note that the addition of polypropylene fibers has no significant effect on the compressive strength of geopolymer mortar since the percentage increase is only 3.58%. However, the addition of polypropylene fiber at 0.75% in geopolymer mortar has a substantial impact on the material's strength-regain ratio because the difference between it and the control is 31.51%.

3.2 Ultrasonic Pulse Velocity (UPV)

The homogeneity of the geopolymer mortar samples was evaluated using Pundit Lab equipment in accordance with the ASTM C 597 standards. Twenty-four (24) samples of geopolymer mortar were tested for compressive strength both before and after cracking, as well as after a 14-day healing period.



	PP0 (0%)	PP1 (0.25%)	PP2 (0.5%)	PP3 (0.75%PP)	BP0 (0%)	BP1 (0.25%)	BP2 (0.5%)	BP3 (0.75%PP)
UPV before cracking	2736.33	2591.33	2161.00	2503.00	1187.67	686.33	1794.67	1261.67
UPV after cracking	1672.00	1980.00	1671.00	817.33	951.33	288.33	1695.33	1634.67
UPV after healing	1637.33	1789.33	1761.33	2186.00	1365.33	1677.33	2281.67	2234.00

Fig. 3 UPV values (before cracking, after cracking and healing) of Geopolymer Mortar with Polypropylene and Bacteria

The variation of ultrasonic pulse velocity (UPV) values of geopolymer mortar mixtures with varied quantities of polypropylene at 28 days is shown in Figure 3. PP0 (0% PP fiber) achieved the highest average UPV value of 2,736.33 m/s, whereas, BP1 (0.25% PP fiber) recorded the lowest average UPV value of 686.33 m/s. According to Borg et al. (2016), higher ultrasonic pulse velocity values indicate that the material has good density, homogeneity, and uniformity. Because of the discontinuity, a lower value indicates that the pulse power is weaker and that it propagates through the material for a longer period of time. The discontinuity could be in the form of defects, voids, or fissures that could prevent the pulse from being transmitted.

Comparing the two batches (PP0 and BP0) without polypropylene fibers, it can be observed that the addition of the bacteria in the geopolymer mortar decreased the ultrasonic pulse velocity by 56.60%. However, the UPV values after healing are substantially higher in geopolymer batch with bacteria than the geopolymer batch without bacteria. It only demonstrates that the presence of bacteria significantly improves the overall quality of the geopolymer mortar with polypropylene.

Table 2. Average UPV values (before cracking, after cracking & after healing), damage degree and self-healing percentages of geopolymer mortar mixtures

Batch code	Ultrasonic Pulse Velocity (UPV)			Damage Degree, DD (%)	Self-healing, H (%)
	Before cracking V1 (m/s)	After cracking V2 (m/s)	After healing V3 (m/s)		
PP0	1672	2736.33	1637.33	38.61	-38.66
PP1	1980	2591.33	1789.33	19.78	-28.43
PP2	1671	2161	1761.33	22.63	-18.50
PP3	817.33	2503	2186	67.20	-12.05
BP0	951.33	1187.67	1365.33	20.47	16.77
BP1	288.33	686.33	1677.33	58.36	147.18
BP2	1695.33	1794.67	2281.67	4.21	28.77
BP3	1634.67	1261.67	2234	-39.94	90

The highest average damage degree was obtained from PP3 (0.75% PP) with a value of 67.20%, while the lowest average damage degree was from the BP3 (0.75% PP) batch with a value of -39.94%. In terms of healing percentages, BP1 (0.25% PP) had the highest average self-healing percentage with 147.18% and the lowest average self-healing percentage of -38.66% from PP0(0% PP) as shown in Table 2. Clearly, polypropylene with bacteria has a considerable impact on reducing the severity of damage and raising the healing rates of fly-ash based geopolymer mortar.

4. CONCLUSION

An alternative technique for repairing and improving the quality of fly-ash-based geopolymer mortar with polypropylene fibers is co-culturing of bacteria using Bacillus Subtilis and Megaterium. The maximum average compressive strength of geopolymer mortar with 0.25% polypropylene fibers (PP1) was 33.85 MPa, whereas geopolymer mortar with 0.25% polypropylene fibers and bacteria (BP1) had the lowest average compressive strength of 17.03 MPa. Polypropylene fibers do not have a significant effect on the compressive strength of the geopolymer mix since the percentage increase is only 3.59%. Nevertheless, geopolymer mortar with bacteria and polypropylene fiber has the potential to be used as a repair mortar since its compressive strength value is at least 15 MPa with an R2 classification.

Meanwhile, the average strength regain for the geopolymer mortar with 0.75% polypropylene fibers and bacteria (BP3) is 199.97%, whereas the lowest average strength regain for the geopolymer mortar with 0.25% polypropylene fibers (PP1) is 97.21%. The inclusion of bacteria and polypropylene fibers in geopolymer mortar significantly influences strength-regain ratios because they can improve the ratio by 51.69% after inducing cracks.

Based on the ultrasonic pulse velocity (UPV) test result, geopolymer mortar with polypropylene and bacteria had a significant effect on minimizing the damage degree by 58.36% and increasing the healing percentages by 147.18%. The quality of the geopolymer mortar with polypropylene fiber is thereby significantly improved by the addition of bacteria, as observed from the result of the strength regain ratio and UPV values after the healing period.

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