

Article

Modifying geopolymer wettability by plasma treatment and fly ash

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Abstract: This paper deals with investigation of changes in geopolymer wettability with increasing mass fraction of high-carbon fly ash and surface treatment by cold atmospheric plasma (CAP). In this study, multiple samples of geopolymers were prepared, including those with 5% and 10% of high-carbon fly ash from coal-fired power station. Wettability of samples was then measured before and after plasma treatment, both on surface and cut surface. While addition of fly ash only had low effect on the wettability, as in most cases, it only lowered the initial contact angle without speeding up the speed of soaking for compact geopolymer and actually slowed the soaking for foamed geopolymer, plasma treatment had significant impact and made the geopolymer hydrophobic.

Keywords: geopolymer; plasma; surface treatment; fly ash; wettability

1. Introduction

Geopolymers are materials formed by polycondensation of aluminosilicate materials, such as metakaolin or fly ash, in strongly basic environment, usually in hydroxide or silicate solution. During the reaction, aluminosilicates are transformed to polysialates and form zeolitic (microporous) structure. In comparison with Ordinary Portland cement (OPC), most widely used construction material, geopolymers exhibit higher compressive strength, resistance against high temperatures and chemicals and lower thermal conductivity. Their manufacturing also requires less energy than OPC. The disadvantages include lower tensile strength and higher price. They may also be used for deposition of waste, such as high-carbon fly ash from coal power stations or glass. This kind of waste may also serve as coloring, unless they have a detrimental effect on properties of geopolymer. Using such commodities as a geopolymer filler may also lower the price or geopolymers and lower the amount of landfilled waste. Geopolymers are a potential alternative to OPC as a building material, as well as useful material for other special application where their properties may be used, such as passive fire protection, where foamed geopolymer is used, as foaming decreases its thermal conductivity and weight, or as insulation for buildings. Geopolymers are also a viable material for 3D printing,[1-2][27-37]

Geopolymers, as well as OPC, are also commonly reinforced by various types of fibers, to compensate for their low tensile and flexural strength, although some materials, such as common glass or light metals cannot be used, due to high geopolymer basicity. Fibers also reduce internal cracking. Materials usable to reinforce geopolymers (and other materials) include carbon, basalt, steel, microspheres, various polymers etc. Geopolymers may also be reinforced with biological materials, such as flax or hemp fibers, or waste materials (such as glass wool) [12-26]

Plasma, an ionized gas exhibiting quasineutrality and collective behavior, is used for cleaning, etching and activation of surfaces, thin layer formation and other purposes. Unlike other methods, it doesn't require high temperatures and aggressive or toxic chemicals (solvents or catalysts), and can therefore be used for less durable materials or in medicine. [3-5]

When used to activate a surface to increase its wettability, reactivity etc., plasma may change the chemical properties of the surface by depositing functional groups, such as hydroxyl, or changing composition of the surface layer. Plasma may also change the surface morphology by the process of plasma etching, which increases coarseness and surface area. If the surface is contaminated, plasma may also be used to clean it. Plasma treatments are one of the most widely used methods of surface hydrophilization, being universal and efficient, while doing no to minimal damage to the surface, especially when compared to treatments using chemicals, such as acids, and leaving no toxic residue. This makes plasma pretreatment a viable method of improving the properties of materials used for medicine, such as scaffolds or implants. [2][6-7][38-39]

As geopolymers may be used as an alternative to OPC, they may also be coated or modified with various types of coatings, including penetration coatings, hydrophobization coatings, antimicrobial coatings etc. However, these coatings, especially when used on industrial scale, may require the geopolymer surface to be hydrophilic and wettable, so that optimal spread and adhesion of the coating is ensured. Geopolymers may also form variable surfaces, depending on their composition, geopolymerization time etc. and may not always be completely wettable. This study therefore aims to investigate the wettability of various types of geopolymers, including those with high-carbon fly ash additive (as using geopolymers to encapsulate the fly ash is a potential viable method to dispose of it, but the ash might have negative impact on geopolymer properties), and the influence of CAP (cold atmospheric plasma) treatment on their wettability, to determine the possibility of using CAP to activate geopolymer surface for the purpose of further functionalization by varnishes, chemical treatments etc. A commercial waterproofing varnish was also tested the possibility of waterproofing various geopolymer materials. [12][40-41]

2. Materials and Methods

2.1. Materials used

Baucis L_{Na}, component A (cement) and B (liquid activator), České lupkové závody a.s
Baucis L_K component A (cement) and B (liquid activator), České lupkové závody a.s
Quartz sand, Sklopísek Střeleč
Silica fumes RW-Füller, AMG Silicon
Aluminium powder AIPRA, PK Chemie
Basalt fibers (length 6 mm, width 13 µm) - ORLIMEX CZ, s.r.o.
Revacryl UF 4210 varnish, Synthomer
Fly ash - Polska Grupa Energetyczna

2.2. Fly ash

High-carbon fly ash, used as a geopolymer additive, was produced by PGE Polska Grupa Energetyczna and comes from an electrostatic precipitator of a coal-fired power station. According to analysis performed at TUL, it contains 76,89 wt. % of Carbon in graphite form. It also contains oxygen, iron, calcium and other elements (presumably in oxide form). Its elemental composition is shown in Table 1.

Table 1. Fly ash composition

Element	Wt%
C	76.89
O	7.10
Fe	5.16
Ca	4.75
Si	1.38
S	1.35
Al	1.02
Mg	0.88
Cl	0.53
W	0.25
K	0.16
Na	0.11
P	0.1

2.3. Geopolymer samples

Geopolymer samples were prepared from sodium metakaolin based geopolymer Baucis L_{Na} and its water glass and sodium hydroxide based activator, both manufactured by České lupkové Závody a.s [8]. Quartz sand and silica fumes were added to all samples (in accordance with geopolymer recipe used at TUL for research), while the samples differed by other additives. From each set, both compact and foamed samples were prepared, with aluminium powder serving as foaming agent. The base composition of samples without additives is shown in Table 2. In total, 5 sets of samples were prepared.

Table 1. Geopolymer base ingredients

Material	Weight (g)
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Geopolymer base	1000
Activator	900
Sand	1000
Silica	50
Aluminium (for foamed)	15

- First set: Additional 70 g of basalt fibers.
Second set: Only the base ingredients.
Third set: 5 wt. % of high-carbon fly ash.
Fourth set: 10 wt. % of high carbon fly ash.
Fifth set: Base set of ingredients, potassium base used instead of sodium.

2.4. Sample preparation

After mixing the ingredients of each set, the paste was mixed for 5 minutes, for foamed samples, aluminium powder was also added after mixing and the paste was mixed for an additional minute, after which it was poured into wooden (for experiments) or plastic (for display) molds, wrapped with polyvinyl sheet and left for 1 day. The samples were then cured at 45°C for 5 hours, unwrapped and cured at 82°C for additional 15 hours. Samples were then cut to smaller pieces suitable for testing, cleaned with water and dried at 45°C for 5 hours. All tests were performed on both the uncut surface of geopolymer and on the cut surface. Addition of high-carbon fly ash colored geopolymers into shades of dark grey, as seen in Figure 1. Geopolymer made from potassium base foamed more significantly with the addition of aluminium powder. The difference can be seen in Fig. 2



Figure 1. Geopolymers with 0 wt. %, 5 wt.% and 10 wt.% of high-carbon fly ash



Figure 2. Foamed sodium (left) and potassium (right) geopolymer

2.5. Plasma pretreatment

For the purpose of testing plasma activation of geopolymer surface, samples were treated with Piezobrush PZ2, a device generating plasma from atmosphere using piezoelectric direct discharge device Ceraplas, which produces cold atmospheric-pressure a plasma jet from atmosphere (nitrogen-oxygen plasma) with temperature below 50°C and operating frequency of 50 kHz (RF equivalent). Its maximum output energy is 10 W. The device was fitted with near-field nozzle used for

conductive surfaces, as using the standard nozzle caused visible discharges into the surface of geopolymer. [11]

The geopolymer surfaces were first cleared with textile and treated by cold atmospheric plasma jet for 5 seconds, while the surface was treated like this for two times, to ensure consistent surface activation. The distance between Piezobrush and sample was around 1 mm or below during surface treatment, as the near-field nozzle does not produce plasma unless in close proximity of the sample.

2.6. Waterproofing varnish application

Commercially available Revacryl UF 4210, a waterproofing varnish for mineral surfaces, was used to waterproof geopolymer samples. The varnish was applied by a paintbrush and multiple layers were applied to ensure optimal surface coverage and to prevent the geopolymer from penetrating the surface. Samples from all sets and both cut and uncut surfaces were waterproofed. [41]

2.7. Wettability testing

All wettability tests were performed on Surface Energy Evaluation System, a device used to measure the contact angle of a droplet of liquid. In this study, 3,5 μ l were used for each measurement. For all samples, the contact angle was measured 3 times during each measurement and mean value was used for calculation. The interval between measurements and number of measurements taken differed for each surface modification or type of surface (uncut or cut). Contact angle was measured at $t=0$ as well. The speed of water soaking into the surface was approximated as a change in contact angle in time.

For untreated and uncut surface, the measurement took 2 minutes with 20 seconds intervals.

For surface, both cut and uncut, treated with varnish, the measurement took 7 minutes with 1 minute intervals.

For cut surface and both cut and uncut surface treated with plasma, the interval was 1 second and contact angle was measured until the droplet soaked into the geopolymer.

3. Results

3.1. Influence of fly ash and basalt fibers on wettability

As seen in Fig. 3, addition of basalt fibers in compact geopolymer only marginally quickened soaking of water into the geopolymer, with the change of initial contact angle being marginable and within the error margin. However, as seen in Fig. 4, foaming the geopolymer with basalt fibers caused it to retain the wettability of a compact sample, with even slower soaking of water into its surface and similar initial contact angle. Without fibers, however, the geopolymer became more wettable, with nearly 3 times faster soaking and lower initial contact angle. Unlike the compact samples or foamed sample with basalt fibers, the droplet had also soaked into the sample completely before reaching 100s during some measurements.

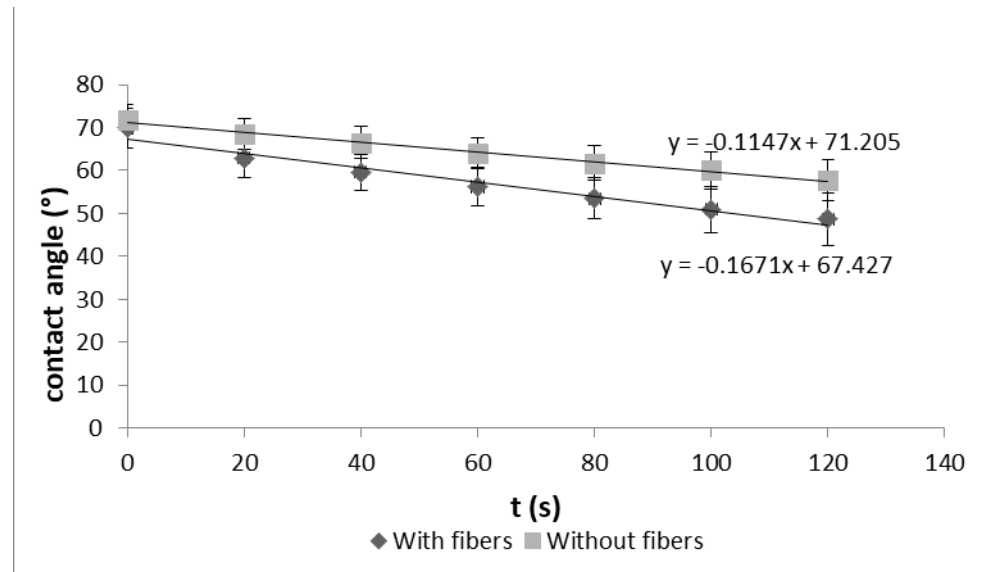


Figure 3. Wettability of compact geopolymer with and without basalt fibers

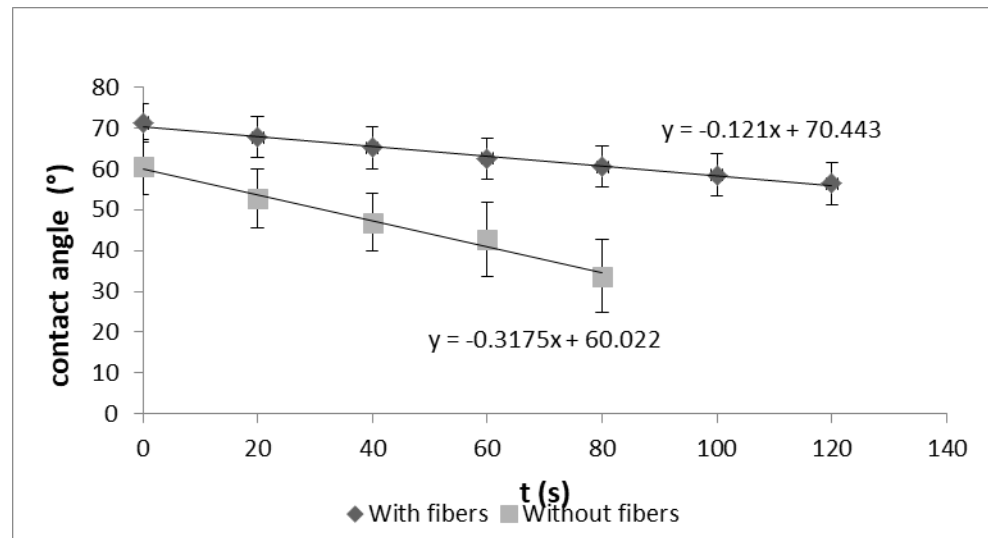


Figure 4. Wettability of foamed geopolymer with and without basalt fibers

As seen in Fig. 5, adding high-carbon fly ash into the compact geopolymer mix significantly lowered the initial contact angle, but did not speed up soaking of water into the geopolymer. At 5 wt. %, the speed of soaking is nearly identical to the sample with no fly ash. At 10 wt. %, the speed of soaking seemed to be marginally bigger. However, as seen in the graph, the difference is within the margin of error of both sets of measurements and may not exist at all. For the foamed samples, as seen in Fig. 6, adding fly ash, despite lowering the initial contact angle, slowed down soaking of water into the samples, although higher mass fraction of fly caused faster soaking than the smaller mass fraction.

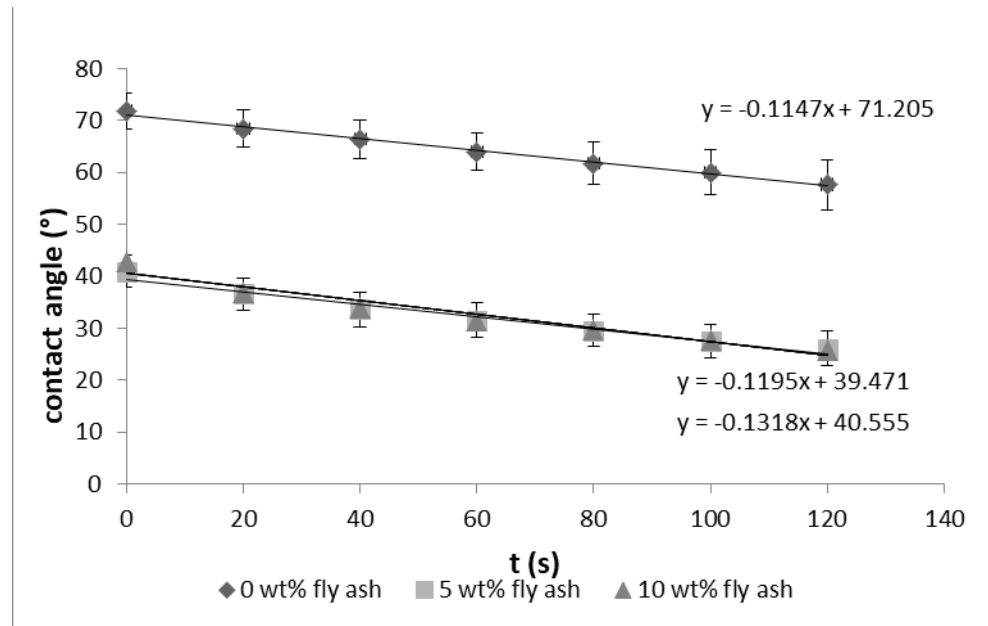


Figure 5. Wettability of compact geopolymer with and without high-carbon fly ash

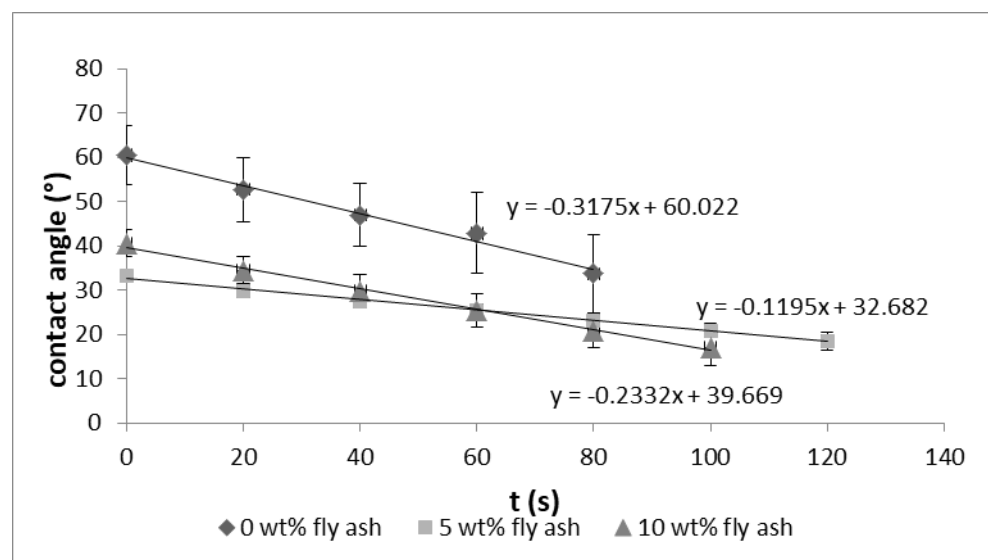


Figure 6. Wettability of foamed geopolymer with and without high-carbon fly ash

3.2. Influence of potassium base

Using potassium base significantly lowered initial contact angle and slightly increased soaking speed on compact samples, as seen in Fig. 7. Foamed potassium geopolymer, which foamed more significantly than the sodium one, could not be tested, as water completely soaked into its surface in a matter of seconds, unlike the foamed sodium geopolymer.

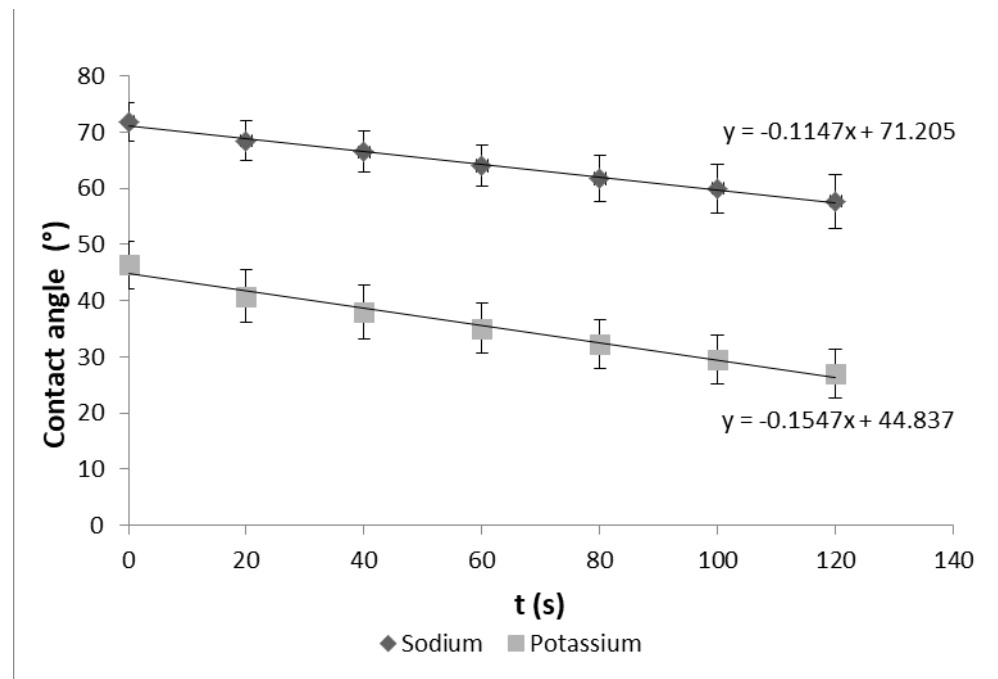


Figure 7. Wettability of compact geopolymer samples made from sodium and potassium base

3.3. Influence of plasma treatment on wettability

Treating the geopolymer surface with CAP significantly increased its wettability, with water droplet soaking into the surface in less than 3 seconds. This result was the same for all samples, with plasma treated cut surface having highest wettability, as it was impossible to even measure the droplet on the surface, as it soaked in nearly instantly.

3.4. Waterproofing

Applying waterproofing varnish on geopolymer samples did prevent water from soaking into them, with the exception of foamed potassium based geopolymer, as it was impossible to properly coat it due to presence of large cavities. While the contact angle was still getting lower, it can be attributed to water evaporating, rather than soaking into the geopolymer, as the varnish produced uniform layer with no visible pores or cracks, there were no signs of water infiltration on geopolymer itself (surface darkening) and similar results being observed on microscope slide tested by the same method. The initial contact angles were also very similar and there were no observable correlations between surface treatment or additive and the contact angle.

4. Discussion

In this paper, two main results were produced, first one determined the influence of various geopolymer ingredients on the wettability of resulting geopolymer composite, with the fly ash being the most influential ingredient, while the second one determined, and whether geopolymer surface treatment using low-energy, low-temperature atmospheric plasma makes the surface of geopolymers of various composition completely hydrophobic. As a secondary result, commercial waterproofing varnish was also tested on all geopolymer samples.

4.1. Influence of potassium base, basalt fibers and fly ash

While adding basalt fibers into the compact geopolymer increased its wettability slightly, as water soaked faster into its surface, adding it into foamed geopolymer decreased its wettability when compared to compact samples with fibers, up to the point of being nearly identical to compact samples without fibers. This can likely be attributed to basalt fibers influencing the foaming process and formation of geopolymer surface.

For compact samples, high-carbon fly ash only lowered initial contact angle without speeding up soaking into the geopolymer, which might be helpful when treating geopolymer with water based solutions, as the wettability is higher. Adding fly ash into foamed geopolymers both lowered the initial contact angle and increase the speed of soaking of water into geopolymer. Once again, this is likely caused by fly ash influencing both the foaming process and surface formation.

For all samples, however, the changes were insignificant in the long term applications, such as when using geopolymer as construction material, as water eventually soaked into all of them (except those treated by waterproofing varnish) despite some of them being seemingly hydrophobic, as the initial contact angle was nearly 90° during some measurements. All cut surfaces were highly wettable and soaked the water in almost immediately.

Geopolymer wettability was therefore influenced mostly by using fly ash and potassium base, with basalt fibers only having small impact on the wettability.

4.2. Plasma activation and waterproofing

Plasma activation was successful at making the geopolymer surface hydrophilic, regardless of additives. There are two possible explanations for this. Plasma may have changed the surface composition of the surface, as nitrogen plasma is known to nitride the surface by replacing oxygen with nitrogen [9], and deposited reactive groups, such as hydroxyl groups, on the surface, or changing the morphology of the surface by etching, as it is possible for plasma be used to etch surfaces, however, it usually requires heavier nonreactive elements, such as argon, to sputter away material from the surface without reacting with it, and CAP only contains roughly 1% of Argon [10].

It is also possible to waterproof geopolymers by commercial varnishes, such as Revacryl UF 4210, which was successfully tested on geopolymer samples, with the exception of foamed potassium based geopolymer, as it foamed much more significantly than other samples and was impossible to coat properly due to cavities.

5. Conclusion

Multiple additives were tested for their influence on geopolymer wettability, while there were differences, none of them were significant, except for foamed potassium based geopolymer, which became completely hydrophobic. Using high-carbon fly ash does increase wettability of geopolymers, but doesn't speed up soaking of water into them. Commercial waterproofing varnish was successful at making all samples waterproof.

Cold atmospheric plasma was successful at activating the surface (both cut and uncut) of geopolymer and making it hydrophilic, and is therefore a viable method for pre-treatment of geopolymer surfaces before applying water-based varnishes or functionalization solutions, although it was necessary to use special systems for treating conductive surfaces.

In future studies, mechanism of hydrophilization of geopolymers should be investigated, as the effect may have been achieved either by changing the surface

composition of geopolymer material, depositing reactive groups (such as hydroxyl groups) or changing the morphology of the surface, as well as by combination of more changes. Influence of fly ash additives on mechanical properties of geopolymer should also be investigated.

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