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## Beneficial use of geopolymers in geotechnical engineering applications

### Utilisation bénéfique des géopolymères dans les applications d'ingénierie géotechnique

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**ABSTRACT:** Geopolymers are inorganic aluminosilicate materials produced by mixing a source of Al and Si with an alkaline activating solution at or near ambient temperature. Pozzolanic material such as thermally activated natural minerals (e.g. metakaolin) or industrial by-products such as fly ash and ground granulated blast furnace slag (GGBFS) can be used as the source of Al and Si in the synthesis of geopolymers. Lower energy demand and absence of harmful CO<sub>2</sub> emissions during production as well as excellent mechanical properties of the synthesized product make geopolymers a more sustainable alternative to traditional cementing agents such as Portland Cement and soil stabilizers such as hydrated lime. This paper presents findings from a detailed research study that investigated beneficial use of geopolymers synthesized using a metakaolin and fly ash in two different geotechnical applications. The first application of the geopolymer was as a stabilizing agent for sulfate-rich soils with high shrink-swell potential. In the second application, geopolymer was used as an admixture in flowable, controlled strength materials to enhance rate of strength gain and reduce setting time.

**RÉSUMÉ:** Les géopolymères sont des matériaux d'aluminosilicate inorganique produits en mélangeant une source d'Al et de Si avec une solution d'activation alcaline à ou près de la température ambiante. Les matériaux pouzzolaniques tels que les minéraux naturels activés thermiquement (par exemple le métakaolin) ou les sous-produits industriels tels que les cendres volantes et les scories de haut fourneau granulées broyées (GGBFS) peuvent être utilisés comme source d'Al et de Si dans la synthèse de géopolymères. Une demande énergétique plus faible et l'absence d'émissions nocives de CO<sub>2</sub> pendant la production ainsi que d'excellentes propriétés mécaniques du produit synthétisé font des géopolymères une alternative plus durable aux agents de cimentation traditionnels tels que le ciment Portland et aux stabilisateurs de sol tels que la chaux hydratée. Cet article présente les résultats d'une étude de recherche détaillée qui a examiné l'utilisation bénéfique des géopolymères synthétisés à l'aide d'un métakaolin et de cendres volantes dans deux applications géotechniques différentes. La première application du géopolymère était comme agent stabilisant pour les sols riches en sulfates avec un potentiel de retrait et de gonflement élevé. Dans la deuxième application, le géopolymère a été utilisé comme adjuvant dans des matériaux fluides à résistance contrôlée pour améliorer le taux de gain de résistance et réduire le temps de prise.

**KEYWORDS:** geopolymer, stabilization, sulfate-bearing, flowable

## 1 INTRODUCTION

Geopolymers are produced by mixing a source of Al and Si with an alkaline activating solution. Pozzolanic material such as thermally activated natural mineral (e.g. metakaolin) or industrial by-products such as fly ash and ground granulated blast furnace slag (GGBFS) can be used as the source of Al and Si in the synthesis of geopolymers. Geopolymers represent inorganic aluminosilicate materials formed from the polycondensation of tetrahedral silica (SiO<sub>4</sub>) and alumina (AlO<sub>4</sub>). These molecules are linked alternately by sharing oxygen atoms. Geopolymers have been explored previously as a lightweight inorganic polymeric building material and a substitute for Portland Cement (Wu & Sun 2007, Bakri *et al.* 2012). Superior mechanical properties (Bakri *et al.* 2012, Fan *et al.* 2018), high resistance to thermal variations (Zhang *et al.* 2016 & 2018) and excellent chemical resistance (Albitar *et al.* 2017, Mehta & Siddique 2017) make geopolymer an attractive construction material.

Geopolymers are synthesized in the range of temperatures from 25 to 80°C. Accordingly, geopolymer production requires much less energy when compared with traditional binders such as Portland Cement and conventional soil stabilizing agents such as lime. Moreover, geopolymer production does not lead to CO<sub>2</sub> emissions (Davidovits, J. 2015). For these reasons, geopolymers have drawn much attention in recent years as a sustainable construction material. This paper describes a research study that investigated potential use of geopolymer in geotechnical applications.

## 2 GEOPOLYMERS FOR SOIL STABILIZATION

Clay soils with high plasticity are sensitive to moisture variations and tend to shrink and swell with the fluctuations in the water table or changes in moisture level. Traditionally, lime and Portland cement have been used to stabilize such clay soils. These traditional stabilizers are effective in controlling volume changes in soil as calcium saturated clay minerals have reduced

affinity for water. Further, these stabilizers help formation of cementation products that resist volumetric changes in soil (Diamond & Kinter 1965). However, lime and Portland cement are not recommended for use when the treated soil contains significant levels of soluble sulfates. The treatment of sulfate rich expansive soils with these stabilizers results in the formation of an expansive mineral called ettringite which aggravates the expansive behavior of the soil. In this research, two geopolymers synthesized from aluminosilicate sources, fly ash and metakaolin, were investigated to determine their effectiveness in stabilizing shrink-swell behavior of high sulfate expansive soil. The geopolymers were then modified with lime and gypsum to improve their efficacy as stabilization agents for sulfate bearing soil. Modifier contents were optimized to achieve maximum reduction in swell potential and at the same time prevent ettringite formation. This was accomplished by conducting a series of swell tests on high sulfate soil that has been treated with different concentrations of modified geopolymers.

### 2.1 Synthesis of the modified geopolymer stabilizer

Two forms of geopolymer were used in this research. The first used metakaolin as the aluminosilicate source and the second used Class C fly ash. The chemical compositions of metakaolin and Class C fly ash are presented in Table 1 below.

Table 1. Chemical Compositions of metakaolin and Class C fly ash

Chemical Components	Metakaolin	Fly ash
SiO <sub>2</sub>	52%	41.96%
Al <sub>2</sub> O <sub>3</sub>	43%	20.04%
Fe <sub>2</sub> O <sub>3</sub>	< 2.20%	5.76%
CaO	< 0.20 %	21.09%
MgO	< 0.10 %	4.30%
Na <sub>2</sub> O	< 0.05 %	1.44%
K <sub>2</sub> O	< 0.40 %	0.73%
SO <sub>4</sub>	< 0.05%	0.99%
Water Content	0.33%	0.33%

The alkaline activator solution was prepared by mixing 50% NaOH solution with technical grade sodium silicate to obtain a 12M solution. The aluminosilicate and the alkaline activator solution were mixed together in a rotary mixer for 5-10min until a homogenous geopolymer slurry was obtained. The 5-10min mixing time was deemed to be sufficient for geopolymerization reactions to occur. Prior research had demonstrated that a Al/Si/Na molar ratio of 1:1.66:1.06 provided optimum properties for the geopolymer and therefore, the same composition was used for the geopolymers synthesized in this research (Khadka *et al.* 2018).

Several researchers have previously reported that when additional calcium is present, it can serve as a catalyst for the formation of additional hydration products, namely C-S-H phases in the geopolymer matrix (Ma & Brown, 1997, Yip & Deventer 2003, Yip *et al.* 2005, Dombrowski *et al.* 2007). Therefore, in this study the decision was made to use additional sources of calcium as modifiers to enhance the formation of C-S-H in the geopolymer matrix so that reduced swelling in high sulfate expansive soil could be achieved. The amount of the modifier incorporated into the geopolymer, however was controlled so that it would not adversely affect the swell behavior by ettringite formation. A separate series of tests showed that the optimum modifier content in the geopolymer was in the range of 6.0-9.5% by mass. Therefore, two modifiers, lime and gypsum were added

to the geopolymer slurry at the above concentration to obtain the modified geopolymers which were then used as the soil stabilizers. The modified geopolymer contents in the soil were varied from 6% to 18% in increments of 3%.

### 2.2 High PI, sulfate-bearing soils

Clay soils used in this experimental study were obtained from project sites located in Atlanta and Dallas in Texas. Therefore, they are referred to as *Atlanta Clay* and *Dallas Clay*, respectively in this paper. The basic soil characterization data for the two soils are presented in Table 2 below. The soils were initially dried in an oven at 110 °C for 24 hours. The dried soil was then pulverized in a grinder and then sieved through No. 40 sieve. Since neither soil has sufficient soluble sulfate content, reagent grade calcium sulfate dihydrate was added to the minus 425 µm fraction of the soil to produce a soil with a sulfate concentration of 15,000 ppm.

Table 2. Geotechnical characteristics of Atlanta and Dallas clay soils

Soil	USCS	LL	PI	OMC	$\gamma_{d-max}$ (kN/m <sup>3</sup> )	% Swell
Atlanta Clay	CH	82	53	31	17.4	16.2
Dallas Clay	CH	63	40	18	19.2	6.8

USCS=Unified Soil Classification System LL=Liquid Limit, PI = Plasticity Index, OMC =Optimum Moisture Content,  $\gamma_{d-max}$ =Maximum dry unit weight

### 2.3 Measurement of percent swell

One-dimensional swell tests were performed in accordance with ASTM D4546. First, water was added to the dry soil to bring its water content to OMC, mixed thoroughly and allowed to cure a minimum of 16 hours in a sealed container. Then, the specified amount of the geopolymer stabilizer was added to the moist soil and blended to achieve a uniform mixture. The soil-geopolymer mixture was then compacted into a compaction mold with dimensions 70mm diameter and 100mm height. Subsequently, a consolidation ring was pressed into the compacted material and a disc shaped specimen with diameter 64mm diameter and height 15mm was obtained for swell testing. Three replicate test specimens were prepared for each soil-stabilizer combination and each stabilizer content so that repeatability of test results could be verified. Prepared samples were sealed in an airtight bag for 7 days under ambient conditions (Relative Humidity ~ 40%, temperature ~ 23°C) prior to testing.

Height of each specimen was measured at the beginning and end of 7-days curing period to account for volumetric expansion while curing. At the end of the curing period, the specimen was placed inside a consolidation cell where it was laterally confined by the consolidation ring. Water was added to the annular space between the inner wall of the consolidation cell and the circular ring and maintained at constant level throughout each test. Moisture absorption occurred through the porous disk at the bottom of the specimen. Metallic cap with a mass of 113±5g was placed at the top of the specimen to obtain swell occurring under a minimal vertical pressure of 0.3kPa. Vertical swell was measured by Linear Variable Differential Transformer (LVDT) placed on top of the metallic cap. The complete setup used in swell measurement is presented in **Error! Reference source not found.** Swell measurements were recorded every 20 minutes until there was no increase in vertical swell over 10 consecutive readings. Total volumetric increase was determined as a

summation of volumetric expansion during curing period and expansion during the period of moisture absorption.

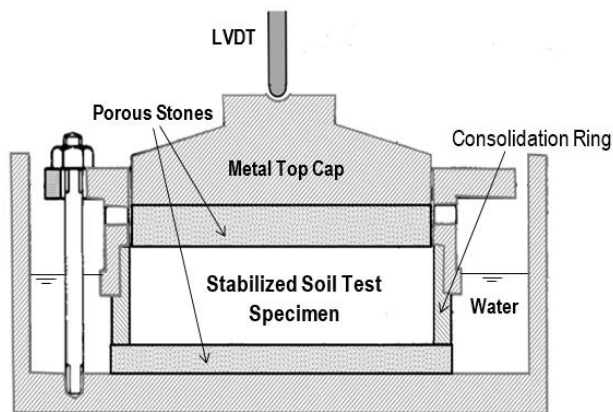


Figure 1. Test setups used for measurement of swell

#### 2.4 Results from swell measurements

The results obtained from measurement of percent swell on Dallas clay stabilized with lime and gypsum modified geopolymer are shown in Figures 2 and 3. Figure 2 presents data obtained for metakaolin based geopolymer while Figure 3 presents similar data for fly ash based geopolymer.

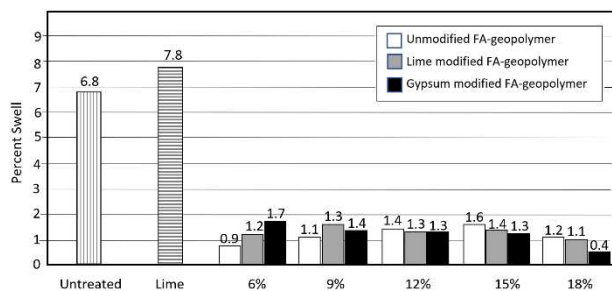


Figure 2. Percent swell data for Dallas clay treated with metakaolin-based geopolymer

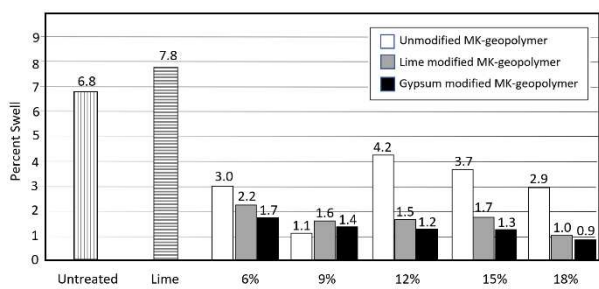


Figure 3. Percent swell data for Dallas clay treated with fly ash-based geopolymer

Review of data shown in Figs. 2 and 3 shows that treatment with modified geopolymer resulted in significant reduction in swell potential in sulfate added Dallas clay. Data obtained for Atlanta clay were very similar to that shown above for Dallas clay (Khadka *et al.* 2020) and are summarized in Table 3 below. Between the two types of geopolymers, the fly ash based geopolymer proved to be more effective than the metakaolin based geopolymer. Fly ash based geopolymer was able to control swell percentage to 2.0% or less at all concentrations regardless of whether modification with lime or gypsum was used or not. The beneficial effects of lime and gypsum modification was more evident in Metakaolin based geopolymer. Once again, the

gypsum modified FA-based geopolymer proved to be the most effective. At stabilizer contents of 9% and above the percent swell for Atlanta clay was reduced from 16.2 for untreated clay to less than 2.0 when treated with gypsum modified FA-based geopolymer.

Table 3. Percent swell data for Atlanta clay treated with metakaolin and fly ash-based geopolymers

	Metakaolin based geopolymer				
	6%	9%	12%	15%	18%
Unmodified	22.7	15.1	23.6	18.1	11.1
Lime Modified	3.2	3.6	6.8	3.4	3.4
Fly Ash Modified	1.5	2.5	4.1	1.8	1.9
	Fly ash based geopolymer				
	Unmodified	2.9	2.8	0.9	1.2
Lime Modified	3.6	2.0	2.3	2.2	1.9
Fly Ash Modified	3.1	1.5	1.6	1.9	1.7

#### 2.4 XRD analysis of stabilized clay

Data obtained from X-ray diffraction studies conducted in previous phases of this study revealed formation of ettringite in sulfate-rich soils stabilized with lime within 7-days after treatment (Khadka *et al.* 2018, Mahrous *et al.* 2018). Similar XRD analysis was performed on clay soil treated with modified geopolymers to determine if there was any evidence of ettringite formation. Even though equivalent quantities of lime had been used to modify the geopolymer that was used to treat high sulfate soil, ettringite was not observed within 7-days. Other research studies have reported that ettringite can develop in treated soil after extended periods (Puppala *et al.* 2005, Cerato *et al.* 2011). To verify that the ettringite did not develop in the modified geopolymer treated clay soils after extended periods, X-ray diffraction tests were performed on treated specimens up to 120 days after treatment. X-ray diffraction patterns of these specimens did not reveal presence of any ettringite indicating that no further changes occurred beyond the initial observation period of 7-days.

### 3 GEOPOLYMERS IN FLOWABLE FILL

Flowable fill is commonly used in restricted spaces where it is difficult to achieve proper placement and compaction of soil backfill. Self-leveling and self-hardening properties of flowable materials allow backfill to be completed more efficiently and with less labor. Flowable fill mixes primarily consist of Portland cement, fly ash, fine aggregate and water. Flowable fills use fly ash in their mix designs to achieve improved flowability and reduce water demand. There is special incentive to use larger amounts of fly ash in the mix instead of cement because such mixes are more economical and sustainable. However, reduction in cement reduces formation of CSH and lime, subsequently limiting CSH produced by the reaction between lime and fly ash. This severely affects the initial setting time and development of early strength in the flowable material. Increase in setting time and slower rate of strength gain become serious limitations when the flowable material must meet a certain minimum strength requirement before subsequent construction can be undertaken. This research examined possible use of geopolymer in flowable mixes to decrease their setting times and increase rates of strength gain while maintaining flowability.

Different techniques have been adopted to reduce the initial setting time of flowable mixes. Accelerating admixtures reduce the amount of water required during mix design without

changing the flowability and provide early stiffening and setting, improve workability of the mix and reduce segregation and bleeding (Najafi & Tia, 2004). In a study performed by Ports et al., use of non-chloride accelerating admixture, *Pozzutec 20*, reduced the setting time of flowable mix from 3 weeks to less than 6 hours (Ports et al. 1998). Similarly, Crouch et al., 2004 developed a rapid set, high-early strength flowable admixture ZOOM®. However, in all of the above studies, admixtures were applied on a mix which primarily consisted of cement and did not contain fly ash in the mix design. In another study, only slight reduction in setting time of high volume fly ash flowable mix was observed when cement was partially replaced by red mud (Do & Kim, 2016). Initial setting time was reduced by 3-4 hours when Chloride and Non-Chloride Accelguard was employed on a high fly ash flowable mix (Pierce& Ihekweazu 2003).

### 3.1 Flowable fill Mix designs

The test program conducted to evaluate the influence of geopolymers on the flowable mix setting times and strength gain rates consisted of a total of 12 mix designs that did not contain any geopolymer and 6 mix designs with geopolymer. Four different water-to-cementitious material ratios (W:C ratios), 0.7, 0.8, 0.9 and 1.0 were considered. Ordinary Portland Cement Type I conforming to ASTM C150 and Class C fly ash, obtained from a local coal power plant were used in the preparation of flowable mixes. Manufactured limestone sand with  $C_u = 6.2$  and  $C_c = 1$  was used as the fine aggregate. Table 4 summarizes design parameters for these mixes. The mixes were evaluated for flowability, and initial and final setting times at 10°, 21° and 32° C. In this paper, only the data obtained for 4 mix designs that were used as controls and the corresponding 4 modified mix designs with geopolymer are presented.

Table 4. Mix design parameters and flowability

Mix	W:C ratio	c:g:f ratio	Spread diam.(mm)
C1	0.9	25:0:75	241.0
C2	0.8	25:0:75	216.0
C3	0.9	15:0:85	254.0
C4	0.8	15:0:85	223.5
M1	0.9	15:10:75	317.5
M2	0.8	15:10:75	279.5
M3	0.9	10:5:85	266.5
M4	0.8	10:5:85	228.5

W:C = water: cementitious material  
c:g:f = cement: geopolymer: fly ash

As shown in Table 4, mix designs C1 through C4 were conventional flowable mixes with high fly ash content. The fly ash consisted of 75% of cementitious material in C1 and C2 and 85% in C3 and C4 with the remainder being Portland cement. When preparing the modified mixes, part of the cement was replaced with an equivalent quantity of geopolymer as shown in Column 3, Table 3.

### 3.2 Flowable fill Testing

After ingredients were properly mixed, flowability of the fresh mix was measured according to ASTM D6103. A plastic flow cylinder open at both ends with a 150mm height and 75mm diameter, smooth interior surface, flat ends and rigid shape was used for the test. The flow cylinder was properly secured on a flat metal pan and then the mix was poured into the cylinder until it was completely filled. The flow cylinder was then raised in a vertical direction avoiding any lateral or torsional movement.

The spread diameter was measured immediately in two perpendicular directions and the average reported as the spread diameter. Table 3 presents flow diameters corresponding to all eight flowable mixes. All of these mixes met the minimum 200mm spread diameter specified in ACI229R. ASTM D 1558 was used to determine setting times for the flowable mixes. The data obtained for the setting times measured at 21°C for all eight flowable mixes are presented in Figures 4 and 5. Figure 4 presents the results from initial setting time measurements while Figure 5 presents the final setting times. In these plots, the gray shaded column represents the control mix and the black shaded column represents the corresponding modified mix with geopolymer.

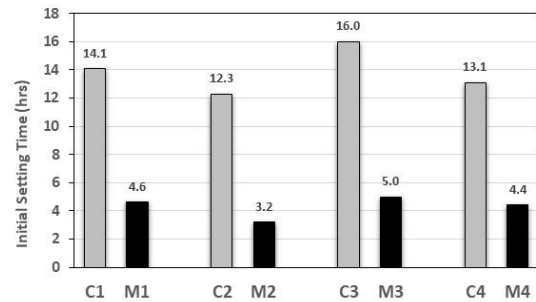


Figure 4. Comparison of initial setting times for control and modified flowable mixes

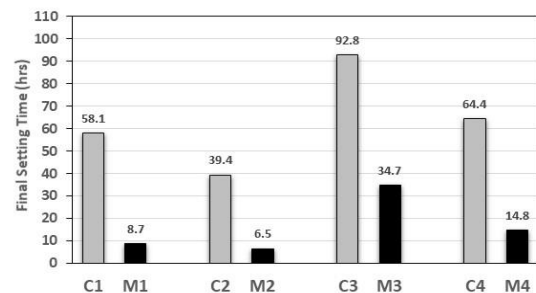


Figure 5. Comparison of final setting times for control and modified flowable mixes

From Figures 4 and 5 it can be readily seen that incorporation of geopolymer in the flowable fill mixes made a significant improvement in the initial and final setting times in the mix. It is equally important to note that this improvement did not come at the expense of flowability. To the contrary, it can be seen that the addition of geopolymer to the mix helped to improve the flowability of the modified mix (i.e. increased the spread diameter) in comparison to the spread diameter of the corresponding control mix.

### 3.3 Microstructural Characterization

Finally, microstructural characterization of the control and modified mixes was undertaken to observe the differences in morphology between them. This was performed using a Hitachi S-4300 E/N field emission variable pressure Scanning Electron Microscope (SEM) with high-magnification back-scattered electron (BSE) and secondary electron (SE) imaging. A 1.5 nm resolution at 15 kV accelerating voltage was employed to assure best imaging conditions. Ancillary analytical system EDAX Pegasus 4040 was used to obtain atomic percentages of a spot or area and the results were normalized to 100%. Specimens were sputter coated with gold and a sticky double-sided conductive

tape was used to attach each specimen to aluminum stub prior to imaging. SEM analysis was performed after curing the specimens for 72 hours in an ambient condition (Relative Humidity ~ 40%, temperature ~ 21 °C). Constant curing period of 72 hours was adopted for all test specimens. Morphological features in both control and modified mixes were analyzed to determine possible explanation for the change in setting time observed when small quantities of geopolymer was added.

Figure 6 compares the SEM images obtained at  $\times 2.5k$  magnification for the control mix C4 and the corresponding modified mix M4. Differences in morphology in the crystalline structures of the two mixes can be easily seen in this side-by-side comparison. Both images show unreacted particles of fly ash that are spherical in shape. But in addition to these, the SEM image obtained for the modified mix shows tubular crystals (see areas that have been encircled). These tubular crystal structures closely resemble the zeolites identified by LaRosa et al. 1992, Katz et al. 1995, and Brough et al. 1995 during their study of cement-fly ash blends activated by alkaline activator.

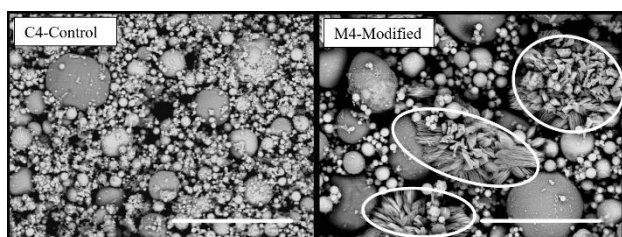


Figure 6. SEM images of the control mix, C4 and modified mix, M4 (magnification = 2.5k)

This observation was further confirmed by observing zeolitic crystal structure at a higher magnification of  $\times 9.0k$  (See Fig. 7). The crystal structures were further verified by using EDS that provided a  $\text{SiO}_2/\text{Al}_2\text{O}_3$  molar ratio of 2.6. Presence of zeolitic crystals was determined to be the main contributing factor that leads to rapid hardening of modified flowable fill.



Figure 7. Zeolite crystalline structure in the modified mix, M4 (magnification = 9k)

#### 4 CONCLUSIONS

This paper presented findings from a research study that explored potential use of geopolymers in applications in geotechnical engineering. Geopolymers are inorganic aluminosilicate materials produced by mixing a source of Al and Si with an alkaline activating solution at or near ambient temperature. It has previously been used as a lightweight inorganic polymeric building material and a substitute for Portland cement. Geopolymers offer many advantages. First, they have excellent mechanical properties, high temperature and chemical resistance. Most importantly, their production does not involve high energy consumption or emission of large quantities of  $\text{CO}_2$ . Moreover, geopolymers may be synthesized from low-cost industrial wastes, such as fly ash. Since, the raw materials readily available for cement production are being over-consumed, geopolymers are considered as a more sustainable alternative.

In the first application described in this paper, two different aluminosilicates, metakaolin and fly ash, were used to synthesize two separate forms of geopolymer, metakaolin and fly ash based geopolymers. Lime and gypsum were used as modifiers in the two geopolymers to achieve enhanced geopolymerization in increased C-S-H formation. The modified geopolymers were then used to stabilize Montmorillonitic and Vermiculitic clays from Atlanta and Dallas respectively. Both lime and gypsum modified geopolymers were found to be effective in controlling the swell behavior of expansive clay. In all cases, gypsum modified geopolymer was more effective in reducing the swell compared to lime modified geopolymer for both metakaolin and fly ash based geopolymers. Formation of ettringite did not occur in any of the specimens treated with modified geopolymer. Absence of ettringite can be attributed to the optimum quantity of additives used during the synthesis of the geopolymer which minimized the amount of free  $\text{Ca}^{2+}$  ions released into the treated soil.

The second geotechnical application described in this paper investigated possible use of geopolymer to improve the initial and final setting times and rate of strength gain in flowable mixtures with high fly ash content. The selected mix designs use c:f ratios (c=cement, f=fly ash) of 25/75 and 15/85. These mixes were prepared at W:C ratios (W=water and C=cementitious material) of 0.9 and 0.8. Their setting times and flowability characteristics were then evaluated at 21 °C. The control mixes with c:f ratios of 25/75 and 15/85 were then modified by reducing the cement content in the mix and substituting it with equivalent combined quantity of geopolymer (fly ash and alkaline solution). Modified mix was then evaluated for setting time and flowability at different W/C ratios. The findings show that incorporation of geopolymers in the flowable mixes were very effective in improving rates of strength gain and setting times of the flowable mixes. In each mix, there was at least a threefold reduction in the setting times. It was further observed that this improvement did not come at the expense of the flowability. To the contrary, the geopolymers contributed to improvement of flowability behavior. The scanning electron microscopic studies undertaken demonstrated that use of geopolymer contributed to formation of zeolitic crystals within the mix. It is believed this change in morphology is associated with changes observed in the setting times.

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