Strength Improvement Using Polypropylene Fiber as Reinforcement in Natural Pozzolana-lime-stabilized Expansive Clayey Soil Artificially Contaminated by Sulfates

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Abstract
This paper aims to assess the behavior of the unconfined compressive strength (UCS) of polypropylene fiber (PF) reinforced natural pozzolana-lime-stabilized expansive grey clayey soil (GS) contaminated by sulfates. Lime (0–8%), natural pozzolana (NP) (0–20%), and PF (0–3%) by dry weight of soil were used. GS was contaminated by different sulfate contents (0-6%) and UCS was assessed at various curing periods (7–120 days). Results showed that adding 8% lime alone or with 20% NP considerably improved UCS. Including 1% or 2% PF as reinforcement without sulfates in lime-stabilized GS transferred stress from GS to PF due to bonding. However, increasing PF beyond 2% formed lumps, reducing contact and friction coefficient, thus decreasing UCS. This means that the optimum PF dosage was found to be 2%. The UCS of GS increased with PF content up to 2% and then decreased. The UCS significantly increased with higher calcium sulfate content and curing period due to observed cementing agents in X-ray diffraction (XRD) and scanning electron microscopy (SEM). Adverse effect of ettringite mineral on UCS caused by sodium sulfate addition was suppressed with the combination of 20% NP and 2% PF in lime-stabilized GS. Binding forces from lime and NP resisted to ettringite expansion. Sensitivity of UCS to sulfate effect was more pronounced with sodium sulfate than calcium sulfate. In addition, NP–PF mixture in lime-stabilized GS was more effective, improving the strength of expansive soil, especially with sodium sulfate. In conclusion, reinforcement of NP-lime-stabilized expansive soils with PF is an effective method.

Keywords
expansive grey clayey soil, additives, unconfined compressive strength (UCS), microstructure and mineralogy, reinforcement

1 Introduction
For any project, the UCS is one of the fundamental parameters, which has the greatest influence on the appropriate choice of construction materials. By knowing the UCS, engineers can accurately assess the suitability of materials for various applications, such as foundations, retaining walls, and structural elements. A higher UCS indicates a stronger and more durable material, ensuring stability and safety in construction projects. Conversely, a lower UCS may lead to structural failures and compromised performance. Therefore, understanding and evaluating the UCS is vital for ensuring the quality and reliability of construction materials and the overall success of a project. However, expansive soils presented a high tendency to swelling, low bearing capacity, and high plasticity index are commonly encountered all over the world. Clay swelling is generally caused by the increase in the water content, which induces fractures and cracks in soil foundations and structure walls where their amplitude varies according to the extent of the phenomenon. The swell phenomenon observed in these soils has been known to damage building structures, runways, and pavements [1]. For road construction, inadequate treatment of expansive soils before constructing a subgrade layer may lead to natural hazards resulting in loss of millions of dollars in repairs and rehabilitations [2]. In Algeria, most of the soils used in highway projects as a subgrade material present insufficient geotechnical property, which can be chemically stabilized for making them able to bear the intensive traffic loads [3]. Chemical stabilization technique using mineral additives was widely used with the main aim is
to make the problematic soils capable of supporting different natures of loads [4]. Indeed, many studies have been conducted for investigating the effect of cement, lime, and volcanic materials on the physico-mechanical properties of clay soils [5–12].

Unfortunately, a large volume of CO₂ was emitted into the atmosphere through different phases of a building life cycle where the incidence of CO₂ emissions from the construction industry is very noticeable [13]. Indeed, it has been reported that a third of the energetic expense, and therefore of the production of this gas, comes directly or indirectly from construction activities [14]. For example, it has been reported that the production of one metric-ton of cement leads to the emission of about one metric-ton of CO₂ and requires a large amount of energy [15]. According to the literature, CO₂ emissions are known as the main chemical compound responsible for global warming, floods, significant increase in temperatures, and drying up of rivers and sea levels [16]. For these reasons, cement replacement by using other mineral additives becomes a key to reducing CO₂ emissions. Accordingly, several researchers have recommended the use of volcanic materials instead of cement in some earth structures in order to reduce both CO₂ emissions and energy consumption [7, 11–12, 15, 17]. In addition, the combination of lime and volcanic ash as an alternative solution can be used because of their environmental advantages and technical performances [7]. For example, it has been reported that lime used alone or in combination with natural pozzolana (NP) considerably improved the mechanical performances of the cohesive soil as compared to the control soil [9].

However, the presence of sulfate in the stabilized soil caused the formation of the expansive ettringite mineral, which is responsible for damages observed on the infrastructures such as cracks and expansion [1, 18–25]. These damages have been found in dependent on the type of the additive used and its amount, the type of sulfate and its amount and the soil nature [15, 17, 26–31].

Recently, Ebaillila et al. [32] studied the effective role of different gypsum contents on the long-term performance of lime-stabilized kaolin soil. They found that the addition of lime into the kaolin soil sample in the absence of gypsum increased the strength and reduced the expansion through both changing of fabric soil, and the development of pozzolanic reactions with the curing time. However, the presence of gypsum was found to have a considerable influence by increasing both strength and expansion of the lime-stabilized soil as the gypsum content increased, especially with a more pronounced effect at higher gypsum and lime contents. This behavior was explained by the formation of a high amount of ettringite mineral, which is depending on both lime and gypsum contents (e.g., the swelling of the kaolin soil stabilized with 6% lime was found to increase from 5% up to 30% in the presence of 9% gypsum). This new mineral caused a high expansion and strength gain under the water-soaking condition due to its higher water absorption capability. A similar study has been conducted by Aldaood et al. [33] where they assessed the influence of different amounts of gypsum and lime percentages on the strength of the fine-grained soil. Results indicated that the effect of gypsum addition on the strength of soil samples depended not only on the curing period but also on the content of lime used. However, the optimum gypsum and lime percent beyond which the improvement in the strength was reduced was found to be 5%. Abdi et al. [34] investigated the effects of sodium and calcium sulfate on the swelling of lime-stabilized kaolinite soil. The obtained results showed that soaking lime-stabilized kaolinite soil samples in sodium and calcium sulfate solutions reduced the strength and promoted swelling as the result of the formation of ettringite mineral, which is significantly influenced after a longer curing period. However, swelling is the result of the formation of the ettringite mineral with a high affinity for water absorption, which results in a reduction in strength. Boz and Sezer [35] studied the influence of sulfate attack on the strength of cement-stabilized kaolin clay containing various concentrations of sodium and magnesium sulfates at different curing periods. According to the results, the increase in cement content and curing time increased the strength of kaolin clay; however, the rate of strength gain decreased in specimens exposed to sulfate attack. In addition, an adverse effect on the hydration bonding between soil and cement was recorded when the magnesium sulfate salt is present in stabilized specimens. Furthermore, magnesium salt not only is responsible for ettringite formation but also causes a reduction in the binding properties of cement. On the other hand, ettringite formation in samples due to the presence of sodium sulfate causes high expansion, accompanied by strength reduction and formation of micro-cracks. Boz et al. [36] assessed the effect of sodium and magnesium sulfates on the engineering properties of highly plastic clay (montmorillonite) by considering several factors such as cement type, sulfate type and its concentration, cement content, and curing period. The obtained results showed that strength decreased as
the salt concentration of the solution increased, despite the sulfate and cement type. It was also observed that the reduction in strength of specimens cured in magnesium sulfate solutions was higher than those cured in sodium sulfate solution, regardless of the cement type. This confirms that magnesium sulfate salt is more aggressive than sodium sulfate, which caused more damage in specimens, especially after a longer curing period.

As for highway engineering, pavement design has a significant process, and the used materials should be economic because of the importance of the projects. Hence, the improvement of soil behavior by using waste materials such as fibers is one of the critical subjects [37]. Indeed, synthetic fibers have been widely used in different civil engineering projects such as polypropylene fiber (PF), which were generally used as a material for concrete reinforcement and soil stabilization [1, 38-41]. For example, in 2013, Olgun [42] investigated the effect of PF on soil volume change characteristics, the tensile and UCS of clayey soils stabilized with cement and fly ash. They have found that the compressive and tensile strength values increased to a great extent by adding more fiber content into the stabilized soil, volume changes decreased by increasing fiber content and the shrinkage values increased with an increase in both the fiber content and fiber length. In addition, in 2010, Ayyappan et al. [43] studied the influence of PF on the engineering behavior of soil-fly ash mixtures used as materials for road construction where they observed that the addition of PF considerably improved the UCS of soil-fly ash mixtures and the optimum dosage rate of the PF was identified as 1% by dry weight of soil-fly ash mixture. The same findings have been found by several researchers when using synthetic fibers in combination with lime for stabilizing fine-grained soils [20, 44–45]. For example, in 2012, Pradhan et al. [46] investigated the effect of random addition of PF on strength of cohesive soils where they observed that 0.8%PF content (20 mm length) provided the highest soaked CBR value, increasing the CBR value by three times compared to the control soil. In addition, in 2010, Tang et al. [47] studied the micro-mechanical interaction between soil and fiber where they concluded that the interfacial shear resistance of soil or fiber depends primarily on the arrangement of soil particles, contact area, fiber roughness, and soil composition. Kalipciar et al. [48] investigated the effect of different basalt and PF contents as reinforcement on the strength of lime-stabilized high-plastic clay by subjecting different specimens to several cycles of freeze-thaw tests. The obtained results showed that the strength of all specimens decreased by increasing the application of freeze-thaw cycles; however, the PF was found to be more effective in comparison with basalt fiber against freeze-thaw action. Kalipciar et al. [49] studied the effect of different fiber types on strength of lime-stabilized low-plastic clay at different curing periods. It was found that for specimens stabilized with lime, the strength of PF-reinforced specimens was strongly higher than that reinforced with basalt fiber for lime-stabilized kaolin clay. However, the greatest strength improvement was obtained when using 0.75% basalt fiber of 19 mm length with 9% lime content after 90 days curing period. However, large volumes of soils with insufficient geotechnical properties were widely encountered in the north of Algeria, which can be improved to be used in several construction projects such as road pavements and earth dams [3]. The NP is found in abundance in areas of Beni-Saf located in the west of Algeria [50]. The PF is commercially available, has a high tensile strength and is cheap in cost [41]. The effect of lime, NP, and their combinations on the engineering properties of cohesive soils has been studied without sulfates by Harichane et al. [51] and Harichane et al. [11]. However, according to the literature, it can be seen that the effect of PF as reinforcement in NP-lime-stabilized expansive clayey soils with and without sulfates has not yet been studied. Hence, the present work is focused on studying the UCS behavior of use of PF as reinforcement in NP-lime-stabilized expansive grey clayey soil (GS) artificially contaminated by sodium (Na₂SO₄) and calcium sulfates (CaSO₄·2H₂O).

2 Materials used and identification

2.1 Soil and natural pozzolana (NP) extraction

In this study, the expansive grey clayey soil (GS) was obtained from an embankment project site located in Chlef town (west of Algeria), which was used as material for road construction (Fig. 1). However, the NP used as an additive for improving the GS was obtained from Beni-Saf deposit located in Oran town (west of Algeria). These two materials were extracted and transported to the laboratory for preparation and testing.

2.2 Expansive grey clayey soil (GS)

A series of laboratory tests were carried out for the characterization and classification of the GS (Fig. 2(a)). Physicomechanical and chemico-mineralogical properties of the GS are depicted in Table 1 [52–59] and Table 2, respectively.
The particle size distribution curve and mineralogical composition of the GS based on X-ray diffraction analysis are illustrated in Fig. 3 and Fig. 4(a), respectively.

2.3 Lime and natural pozzolana (NP)
In this study, the NP rock was ground to the specific surface area of 420 m$^2$/kg (Fig. 2(b)), which was verified according to [60] and added with 20% by dry weight of soil. It was found to have a high amount of amorphous silica (SiO$_2$) [61]. However, the lime used (8% lime by dry weight of soil) was a hydrated lime (Ca(OH)$_2$), which was commercially available lime typically used for construction purposes (Fig. 2(c)). The mineralogical composition of both NP and lime (based on X-ray diffraction analysis) are illustrated in Figs. 4(b) and 4(c), respectively. In addition, the physico-chemical properties of these additives are presented in Table 3.

| Table 1 Physico-mechanical properties of the gray clayey soil [9] |
|---------------------------------|-----------------|-----------------|
| Physico-mechanical properties  | Standards  | GS |
| Depth (m)                      | -            | 4.0            |
| Natural water content (%)      | [52]        | 32.90          |
| Specific Gravity (-)           | [53]        | 2.71           |
| Passing 80 µm sieve (%)        | [54]        | 85.0           |
| Classification System (USCS), (-) | [55]  | CH              |
| Unconfined Compressive Strength UCS (kPa) | [56] | 100            |
| Liquid Limit (LL, %)           | [58]        | 82.8           |
| Plastic Limit (PL, %)          |             | 32.2           |
| Plasticity Index (Pl, %)       |             | 50.6           |
| OMC ($W_{opt}$, %)              | [59]        | 28.30          |
| Compaction                     |             |                |
| MDD ($\gamma_{d,max}$, kN/m$^3$) |             | 13.80          |
2.4 Sodium and calcium sulfates

Two types of sulfates were used in this study (0, 2, 4, and 6% by dry weight of soil). The first is a sodium sulfate (Na$_2$SO$_4$) (Fig. 2(d)). The second is a calcium sulfate dihydrate (CaSO$_4$·2H$_2$O) (Fig. 2(e)). Both sulfates were produced by Biochem Chemopharma as a leading international manufacturer and supplier of laboratory reagents. The physico-chemical properties of these elements are shown in Table 4.

2.5 Polypropylene fiber (PF)

In this study, the fiber used is a polyethylene (added with 0, 1, 2, and 3% by dry weight of soil), which is commercially available synthetic plastic fiber. Polyethylene fibers used as a stabilizer can reduce the space required for landfilling of such large-scale wastes. The photographic view of the fiber used in this study is shown in Fig. 2(f). However, the physico-mechanical properties of the PF used as reinforcement are shown in Table 5.

3 Tests and methods

In this study, based on the ASTM standards, several UCS [56], microstructure [62], and mineralogy [64] tests were made on the lime-stabilized GS samples, reinforced by different PF amounts with and without sulfates at different curing periods.

3.1 UCS test

The Wykeham Farrance Eng-Ltd-(5-ton) compression machine apparatus (WFEng-Ltd-5Ton-CM) with a manual data recording system was used for the UCS test according to [56] in order to assess the strength improvement of NP–PF–reinforced lime-stabilized GS artificially contaminated by sodium and calcium sulfates. For this purpose, a total of 112 combinations based on the GS samples were tested after different curing periods (Tables 6 and 7). The air-dried soils were initially mixed with the predetermined quantity of NP, lime (L), or L–NP in a dry state, and then the calculated water was added to the soil mixture.

Indeed, the obtained specimens were prepared by compaction at maximum dry density (MDD) and optimum

![Fig. 3 Particle size distribution curve of the GS](image-url)
Fig. 4 X-ray diffraction analysis showing the mineralogical composition of the materials used; (a) GS, (b) NP, (c) Lime

3.2 Microstructure and mineralogy tests
Scanning electron microscopy (SEM) [64] and X-ray diffraction (XRD) [65] tests were invited in order to examine respectively the mineralogical and microstructure changes in the GS samples stabilized with lime, NP, and their combination (curing with and without sulfates). For XRD
an analysis, a PHILIPS-PW-3020 diffractometer was used to observe possible changes in mineralogy before and after stabilization. The obtained samples from UCS tests were dried for 1 day at 40°C. Before testing, the collected soil samples were crushed into a fine powder and passed through a 400 μm sieve to use as samples for XRD tests (Fig. 5(a)). Diffraction patterns were conducted using Cu–Kα radiation with a Bragg angle (2θ) range of 4°–60° running at a speed of 0.83.10–2/2 sec. In this study, the assessment of the eventual formation of cementing agents and ettringite was conducted on the unstabilized and stabilized GS samples after 60 days of curing. In addition, the investigation of the formation of these new compounds was also conducted on the GS samples containing 4% of sodium and calcium sulfates. However, it is known that the SEM gives magnified images of the size, composition, crystallography, shape, and other physico-chemical characteristics of a specimen.

In fact, physico-chemical analyses (in addition to physico-mechanical experiments) are very important to observe the reactions occurred between soil and additives used as a result of soil enhancement. For this purpose, the PHILIPS-S50 SEM model was used in this study to observe the modification in the microstructure of the stabilized GS samples (Fig. 5(b)). Indeed, after performing UCS tests at 7-, 30-, and 120 days curing period, SEM observations were done on the same unstabilized and lime-stabilized GS samples with and without sulfates. SEM images were taken at 1000× magnification. For this, all the GS samples were mounted on metal stubs and coated with gold-palladium alloy at 200 nm thickness in a low-pressure argon atmosphere, which dried them. In addition, the GS samples (volume 1cm³) were injected by epoxy fix resin, polished, gold-coated, and then scanned by a high-resolution SEM (PHILIPS-S50 SEM model). Several digital images at different magnifications were made in order to observe the formation of cementitious materials and ettringite. It should be noted that all the GS samples were prepared in the same manner.
4 Samples preparation

4.1 GS Samples prepared without sulfates

The GS samples prepared for UCS tests were stabilized using 8%L, 20%NP, 8%L + 20%NP, and reinforced by different PF contents to obtain several mixtures without sulfates (GS‒lime, GS‒lime‒PF, GS‒NP, GS‒NP‒PF, GS‒lime‒NP, and GS‒lime‒NP‒PF mixtures).

Fig. 5 Assessment of the mineralogy and microstructure modifications of unstabilized and lime-stabilized GS samples; (a) Samples for SEM and XRD analysis obtained from specimens used for UCS tests, (b) PHILIPS-S50 SEM model apparatus used in this study for assessing the microstructure modification

The predetermined quantity of NP and lime were initially mixed with the GS in a dry state to obtain different mixtures. These mixtures were sieved through 1 mm sieve mesh openings and then mixed with different PF contents. The content of each mixture was calculated and weighted according to the MDD and then placed into a glass container. In addition, the calculated water according to the OMC was also added to each mixture and let stand for 1 hour prior to specimen preparation. However, several specimens were performed at MDD and OMC and then submitted to UCS test at different curing periods. The UCS tests of all samples were repeated on two identical specimens. In fact, the accepted peak stress was
an average of two tests carried out on each sample type. In term of UCS results compatibility, a maximum difference of 3% between the UCS results for the two tests carried out on two identical samples is accepted; otherwise, a third test must be carried out.

4.2 GS samples artificially contaminated by sulfates
All the mixtures were prepared following the same method as above where different sodium and calcium sulfates contents were added to each mixture in a dry state (GS‒lime–sulfate, GS‒lime–PF–sulfate, GS‒NP–sulfate, GS‒NP–PF–sulfate, GS‒lime–NP–sulfate, and GS‒lime–NP–PF–sulfate mixtures). The content of each mixture was calculated and weighted according to the MDD and then placed into a glass container. In addition, the calculated water according to the OMC was also added to each mixture and let stand for 1 hour prior to specimen preparation. A static press was used for performing all the specimens after the same time (1 hour) and in the same way as presented above.

5 Results and discussion
5.1 Effect of PF and NP on the UCS of lime-stabilized GS samples
Fig. 6 shows the modifications in the UCS caused by adding 20%NP and different amounts of PF into the GS stabilized samples.

<table>
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<th>Designation</th>
<th>Mixture (%)</th>
<th>Designation</th>
<th>Mixture (%)</th>
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<td>GS-N4-PF2</td>
<td>94 0 0 4 2</td>
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<td>86 0 8 4 2</td>
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Fig. 5 Assessment of the mineralogy and microstructure modifications of unstabilised and lime-stabilised GS samples (a) Samples for SEM and XRD analysis obtained from specimens used for UCS tests; (b) PHILIPS-S50 SEM model apparatus used in this study for assessing the microstructure modification
with 8%L. As shown in Fig. 6(a), the unstabilized GS was found to have UCS values of 0.1, 0.11, and 0.12 MPa after 7-, 30-, and 120 days curing period, respectively. A slight change was observed in UCS values when using NP alone as a stabilizer for the GS. However, it is quite clear to observe that adding 8% lime into the GS results in an increase in the UCS, especially when combining 8% lime and 20%NP. A similar trend was observed by Anggraini et al. [66]. Indeed, there is a considerable increase in UCS values with lime added alone as compared with the unstabilized GS. In addition, the increase in UCS values was found to be more important when using the combination of 8%L and 20%NP, especially after a longer curing period.

For example, after 7 days curing period, the UCS of the GS samples increased from 0.1 MPa up to 1.1 and 1.79 MPa by adding 8%L and 8%L + 20%NP, respectively. However, after 120 days curing period, a further increase in the UCS value was recorded from 0.12 MPa up to 2.74 and 4.34 MPa by adding 8%L and 8%L + 20%NP, respectively. This can be explained by the formation of new cementing agents (calcium silicates hydrates: C–S–H and calcium aluminates hydrates: C–A–H) during pozzolanic reactions caused by the addition of lime alone or mixed with NP as shown in Fig. 7.

Fig. 7 presents the XRD patterns of the GS samples stabilized with 8%L, 20%NP, and 8%L + 20%NP after curing for 60 days in the presence of 4% of both sodium and calcium sulfates. Indeed, it is obvious to observe that before stabilization, the XRD analysis showed that the main components of the GS are the quartz, montmorillonite, illite, kaolinite, and calcite clay minerals, while the calcite is the predominant clay mineral (Fig. 7(j)). However, the SEM image of the initial microstructure of the unstabilized GS showed the presence of natural CaO and CaCO$_3$, as shown in Fig. 8(a). When adding 8%L alone or in combination with 20%NP to the GS samples, the XRD analysis showed the formation of cementing compounds (C–S–H and C–A–H), which are responsible for the changes observed in its mineralogical composition, and consequently, the increase in its UCS was observed (Fig. 7(g), (h)). However, with 20%NP as a stabilizer added alone into the GS, the formation of these compounds (C–S–H and C–A–H) was not revealed by the XRD patterns (Fig. 7(i)) and SEM images (Fig. 8(b)). This can be explained by the low pH value developed by NP addition, which cannot react with the clay minerals in the absence of lime or other pozzolanic reaction activators [3]. In the case of lime used alone or

![Fig. 6](image_url)

**Fig. 6** Effect of 8%L, 20%NP and 8%L+20%NP on the UCS of the stabilized GS samples reinforced by 1, 2, and 3% PF at different curing periods; (a) GS samples stabilized without PF, (b) GS samples reinforced by 1%PF, (c) GS samples reinforced by 2%PF, (d) GS samples reinforced by 3%PF.
in combination with NP and based on the results of SEM images, it is clear to observe the formation of cementitious compounds (C–S–H and/or C–A–H) in the GS samples, which are responsible on the densification of their microstructure as compared to that of the unstabilized GS, and consequently, the strength improvement was reported with the presence of some cracks and voids (Fig. 8(c), (d)). In general, the above results indicated that adding lime alone or in combination with NP act as a very good stabilizing agent, which is valuable for strength gain.

On the other hand, introducing PF into the lime-stabilized GS samples caused a further increase in UCS values up to 2% of PF, especially with an increasing curing period, and then decreased beyond this value (2%PF) (Fig. 6(b)–(d)). The increase in the UCS value becomes more and more important when introducing 20%NP in combination with 1 or 2%PF, particularly with the curing period, but adding 3%PF was found to decrease the UCS of the same samples. However, stabilization with 20%NP alone or adding any content of PF alone into the...
unstabilized GS samples were found to have a slight effect on the UCS. It is obvious to observe that the increase in PF content plays an important role in increasing the UCS of NP–PF–reinforced or PF–reinforced lime-stabilized GS samples. Inclusion of PF increased the UCS up to 2%PF and beyond that, it increased. For example, with this optimum of PF content (2%), NP–PF–reinforced lime-stabilized GS samples increased the UCS values of the unstabilized GS from 0.31, 0.37, and 0.44 MPa up to 2.4, 3.85, and 5.29 MPa after 7-, 30-, and 120 days curing periods, respectively. A similar behavior was reported by Anggraini et al. [66]. It is suggested that the higher inter-particle bond developed between NP, lime, expansive GS, and fiber leads to obtain a great improvement in strength. In addition, as pozzolanic reactions depend on time, the strength of the soil increases more and more with the curing period which leads to an additional increase in strength value. Moreover, Dash and Hussain [67] and Olgun [42] have reported in their studies that the enhancement of the UCS for lime-stabilized PF-reinforced clay soil might be attributed to the fact that cementing agents formed after lime treatment have irregular surfaces and high rigidity, which binds the PF–clay soil particles together and provides a dense matrix structure. This leads to an increase in the effective contact area and interlocking between PF and modified clay particles, and thus enables greater mobilization of friction between them with an increase in PF content [66]. It can be seen that the bonding between PF and modified GS particles helps to transfer the stress from GS to PF. However, the increase in PF content (beyond 2%) forms lumps and adheres to each other; thus, there may be a deficiency in the contact between GS and PF which is responsible for the reduction of friction coefficient and hence reduce the UCS.

Fig. 8 SEM images show the modification in the microstructure of the GS samples stabilized with 8%L, 20%NP and their combination after 30 days curing period; (a) Unstabilized GS sample, (b) GS sample stabilized with 20%L, (c) GS sample stabilized with 8%L, (d) GS sample stabilized with 8%L+20%NP.
5.2 Effect of sulfates and PF on the UCS of unstabilized GS samples

The results of the UCS variation of the unstabilized GS samples assessed under different sulfate contents and PF are depicted in Tables 8 and 9, respectively.

Table 8 Changes in UCS values of the unstabilized GS samples performed with and without sulfates at different curing periods

<table>
<thead>
<tr>
<th>Mixture</th>
<th>UCS (MPa)</th>
<th>7 days</th>
<th>30 days</th>
<th>120 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>GS without sulfates</td>
<td>GS</td>
<td>0.10</td>
<td>0.11</td>
<td>0.12</td>
</tr>
<tr>
<td>Na$_2$SO$_4$-contaminated</td>
<td>GS-N2</td>
<td>0.10</td>
<td>0.10</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td>GS-N4</td>
<td>0.10</td>
<td>0.10</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>GS-N6</td>
<td>0.10</td>
<td>0.10</td>
<td>0.12</td>
</tr>
<tr>
<td>CaSO$_4$·2H$_2$O-contaminated</td>
<td>GS-C2</td>
<td>0.14</td>
<td>0.20</td>
<td>0.68</td>
</tr>
<tr>
<td></td>
<td>GS-C4</td>
<td>0.34</td>
<td>0.48</td>
<td>0.87</td>
</tr>
<tr>
<td></td>
<td>GS-C6</td>
<td>0.53</td>
<td>0.64</td>
<td>1.17</td>
</tr>
</tbody>
</table>

As shown in Table 8, it is quite clear to see that the unstabilized GS samples showed a negligible improvement in UCS values upon the addition of any sodium sulfate contents. However, the UCS was found to decrease considerably with increasing calcium sulfate content, especially with the curing period. It can be seen that after 30 days curing period with the presence of 4% calcium sulfate, SEM images showed the development of new cementing agents (C–S–H and C–A–H), which significantly improved the compactness of the unstabilized GS samples as compared with the natural GS samples without calcium sulfate (Fig. 9(a)). In contrast, for the same soil cured for 30 days with the presence of 4% sodium sulfate, several cracks and voids were revealed but no cementing agents were observed (Fig. 9(b)). On the other hand, for any curing

Table 9 Changes in UCS values of the PF-reinforced unstabilized GS samples performed with and without sulfates at different curing periods

<table>
<thead>
<tr>
<th>Mixture</th>
<th>UCS (MPa)</th>
<th>7 days</th>
<th>30 days</th>
<th>120 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>GS without sulfates and without PF</td>
<td>GS</td>
<td>0.10</td>
<td>0.11</td>
<td>0.12</td>
</tr>
<tr>
<td>Na$_2$SO$_4$-reinforced</td>
<td>GS-N2-PF1</td>
<td>0.12</td>
<td>0.14</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>GS-N2-PF2</td>
<td>0.15</td>
<td>0.22</td>
<td>0.26</td>
</tr>
<tr>
<td></td>
<td>GS-N2-PF3</td>
<td>0.11</td>
<td>0.12</td>
<td>0.20</td>
</tr>
<tr>
<td>CaSO$_4$·2H$_2$O-reinforced</td>
<td>GS-C2-PF1</td>
<td>0.18</td>
<td>0.27</td>
<td>0.88</td>
</tr>
<tr>
<td></td>
<td>GS-C2-PF2</td>
<td>0.26</td>
<td>0.35</td>
<td>0.96</td>
</tr>
<tr>
<td></td>
<td>GS-C2-PF3</td>
<td>0.15</td>
<td>0.22</td>
<td>0.62</td>
</tr>
</tbody>
</table>

Fig. 9 SEM images show the modification in the microstructure of the unstabilized GS samples artificially contaminated by 4% sodium and calcium sulfates after 30 days curing period; (a) Unstabilized GS sample containing 4% calcium sulfate, (b) Unstabilized GS sample containing 4% sodium sulfate
period, introducing PF into the unstabilized GS samples with and without sodium sulfate caused a slight increase in UCS values up to 2%PF content, and then decreased when inclusion a more than this content (Table 9).

However, the same behavior was observed in the case of PF–reinforced GS samples artificially contaminated with different calcium sulfate contents where the increase in the UCS was found to be more important as compared to the sodium sulfate addition. Puppala and Musenda [1] reported that introducing PF into expansive clays increased their strength.

5.3 Effect of sulfates and PF on the UCS of stabilized GS samples

The results of the UCS variation of the stabilized GS samples assessed under different contents of both sulfates and PF at different curing periods are shown in Figs. 10 and 11 and Figs. 12 and 13, respectively. In the absence of PF and for any curing period, tests results indicated that when calcium sulfate is incorporated into the GS samples stabilized with 8%L, 20%NP, or their combination (8%L + 20%NP), the UCS starts to increase significantly with increasing calcium sulfate content and curing period (Fig. 10(a), (c), (e)).
This increase was found to be more pronounced with the addition of 8%L + 20%NP than with the addition of 8%L or 20%NP alone.

The significant increase in the UCS value of the GS samples can be explained by the formation of primary ettringite mineral and cementing agents (C–S–H and C–A–H), which strongly bend the clay particles together and consequently increased its UCS, as shown in Fig. 7(d), (e). In addition, SEM images revealed the formation of cementing agents in NP-stabilized GS samples (Fig. 14(b)) and both cementing agents and the primary ettringite mineral in lime-NP-stabilized GS samples (Fig. 15(b)). According to Gadouri et al. [25], the primary ettringite mineral is a product resulting from chemical reactions between cementing agents (C–A–H and C–S–H) and calcium sulfate due to the pozzolanic reactions between calcium ions (Ca^{2+} from lime hydration) and silica/alumina from soil and/or NP addition. This type of ettringite mineral does not cause swelling because it is crystallized in the free spaces of the material before hardening. In fact, these crystals have several beneficial effects on the stabilized soil by decreasing its porosity and permeability and increasing its strength.
When sodium sulfate is present, it can be seen that for any curing period, the sodium sulfate added with any content presents a marginal effect on the UCS of NP-stabilized GS samples. However, for a shorter curing period (1 day) (Fig. 10(a)), the presence of sodium sulfate in the GS samples stabilized with 8%L, 20%NP or their combination (8%L + 20%NP) has a similar effect as that of the calcium sulfate where the UCS starts to increase considerably with increasing sodium sulfate content. In addition, the sensitivity of the UCS to the sulfate effect was found to be more pronounced with the presence of sodium sulfate than with the presence of calcium sulfate. On the other hand, for a longer curing period (30 or 120 days) (as shown above in Fig. 10(c), (d)), the UCS of lime-stabilized GS samples considerably increased up to 2% sodium sulfate but significantly decreased beyond this value. The same variation was observed when mixing 8%L with 20%NP where the UCS was found to increase significantly up to 2% sodium sulfate but a slight decrease was recorded beyond this value. It should be noted that when 20% NP was added to the lime-stabilized GS samples with different sodium sulfate contents, a decrease in the UCS was obtained as compared to the same samples without sodium sulfate.
This behavior indicates that, in the presence of NP, the adverse effect of sodium sulfate on lime-stabilized GS samples was significantly reduced and the strength was also improved. For example, in the presence of 20%NP, the UCS of lime-stabilized GS samples containing 6% sodium sulfate increased from 0.1 MPa up to 2.4 MPa and from 0.12 MPa up to 1.79 MPa after curing for 30 and 120 days, respectively.

As shown in Fig. 7(a), (b), the early increase in the UCS of GS samples containing 2% sodium sulfate can be explained by the formation of high amounts of cementing agents (C–S–H and C–A–H) due to the high pH value from NaOH as compared to the pH value from Ca(OH)$_2$ (the detailed role of the pH was given in the section below). However, the significant decrease in the UCS of lime-stabilized GS samples containing 4 and 6% sodium sulfate contents can be attributed to the formation of secondary ettringite mineral, which is highly expansive. In addition, SEM images revealed the formation of both cementing agents and secondary ettringite mineral in lime-NP-stabilized GS samples (Fig. 15(a)); whereas, no cementing agents were formed in NP-stabilized GS samples (Fig. 14(a)). In fact, the secondary ettringite mineral is a product resulting from chemical reactions between cementing agents (C–A–H and C–S–H)
Fig. 14 SEM images show the modification in the microstructure of NP-stabilized GS samples artificially contaminated by 4% sodium and calcium sulfates after 30 days curing period; (a) NP-stabilized GS sample containing 4% calcium sulfate, (b) NP-stabilized GS sample containing 4% sodium sulfate.

Fig. 15 SEM images show the modification in the microstructure of the unreinforced and PF-reinforced NP-lime-stabilized GS samples artificially contaminated by 4% sodium and calcium sulfates after 120 days curing period; (a) Unreinforced GS sample containing 6% sodium sulfate, (b) Unreinforced GS sample containing 6% calcium sulfate, (c) Reinforced GS sample containing 6% sodium sulfate and 2%PF, (d) Reinforced GS sample containing 6% calcium sulfate and 2%PF.
and sodium sulfate. According to Gadouri et al. [25], the secondary ettringite mineral generates a high internal swelling which is mainly attributed to the crystallization pressures, increases the porosity of the soil (consequently the permeability), decreases the free water content during ettringite nucleation, absorbs a lot of water molecules (consequently producing a high expansion), improves the compressive strength at an early stage (densifies the structure), reduces the compressive strength at an early stage and deteriorates the specimens at a later stage.

In the presence of any PF content and for any curing period, tests results indicated that when PF is included in the GS samples stabilized with 8%L, 20%NP, or their combination (8%L + 20%NP), the UCS was found to increase significantly with increasing calcium sulfate content and curing period (Fig. 10(b), (d), (f)). This increase was also found to be more pronounced with the addition of 8%L + 20%NP than with the addition of 8% lime or 20%NP alone. However, it can be seen that for any curing period, the sodium sulfate added with any content presents a marginal effect on the UCS of NP-stabilized GS samples. In addition, after 7 days curing period and for any content of PF (Fig. 10(b) and Fig. 11(a), (b)), the presence of sodium sulfate in PF-reinforced GS samples stabilized with 8%L or in combination with 20%NP presents a similar effect as that of the calcium sulfate where the UCS starts to increase significantly with increasing sodium sulfate content. Indeed, the sensitivity of UCS of PF-reinforced stabilized-GS samples to the sulfate effect was found to be more pronounced with the presence of sodium sulfate than with the presence of calcium sulfate. In addition, after 30 and 120 days of curing periods and for any PF content (Fig. 10(d), (f) and Fig. 11(c)–(f)), the UCS of lime-stabilized GS samples considerably increased up to 2% sodium sulfate but significantly decreased beyond this value. The same behavior was observed when mixing 8%L with 20%NP where the UCS was found to increase significantly up to 2% sodium sulfate but a considerable decrease was recorded beyond this value. It can be seen that, after 120 days curing period, SEM images revealed the formation of both cementing agents and ettringite mineral in NP–PF–reinforced lime-stabilized GS samples artificially contaminated by 6% sodium and calcium sulfates (Fig. 15(c), (d)). In addition, as shown in (Figs. 12 and 13), introducing PF into the lime-stabilized GS samples with and without sulfates caused a greater increase in the UCS up to 2% of PF, especially with increasing curing period and then decreased beyond this value (2%PF). The increase in the UCS value becomes more and more important when introducing 20%NP in combination with 1 or 2% PF, particularly with the curing period but adding 3%PF was found to decrease the UCS of the same samples. It should be noted that when 2%PF + 20%NP was added into the lime-stabilized GS samples with different sodium sulfate contents, instead of a decrease in the UCS, an improvement in strength was obtained. This behavior indicates that, the use of 2%PF + 20%NP as an optimum treatment, the adverse effect of sodium sulfate on the strength of lime-stabilized GS was entirely suppressed and the strength gain was observed.

5.4 Mechanism of strength improvement linked in the GS–NP–PF–lime–sulfate system

In the absence of sulfates, the addition of lime alone or mixed with NP to the clayey soils immediately modified their physical properties due to the cation exchange capacity. Electrically, clay particles surface is negatively charged, which develops high repulsive forces between them. The ionization of calcium hydroxide in the presence of water produces calcium and hydroxyl ions, which are attracted to the surface of clay particles, shown as Eq. (1). This reduces the repulsive forces and consequently increases the adhesion between clay particles leading to the formation of flocks due to the particles flocculation and flocks agglomeration [68]. In fact, particles flocculation is a result of the chemical reactions between clay particles and calcium cations, which leads to an immediate change in soil consistence [6, 31, 61, 69]. On the other hand, the dissolution of both sodium and calcium sulfate in the presence of water produces calcium and sodium cations, which, as shown in Eqs. (2) and (3). However, according to Roy [70], the reaction between sodium sulfate and lime in the presence of water leads to the formation of NaOH, which develops a higher alkaline solution as compared to that developed by the Ca(OH)$_2$, shown as Eq. (4).

$$\text{Ca(OH)}_2 \rightarrow \text{Ca}^{2+} + 2\text{OH}^{-} \quad \text{(Ionization of calcium hydroxide in the presence of water)}$$

$$\text{Na}_2\text{SO}_4 \rightarrow 2\text{Na}^+ + \text{SO}_4^{2-} \quad \text{(Dissolution of sodium sulfate)}$$

$$\text{CaSO}_4 \cdot 2\text{H}_2\text{O} \rightarrow \text{Ca}^{2+} + \text{SO}_4^{2-} + 2\text{H}_2\text{O} \quad \text{(Dissolution of calcium sulfate)}$$

$$\text{Ca(OH)}_2 + \text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2\text{NaOH} \quad \text{(Formation of sodium hydroxide and pH value rises up to 13)}$$
The addition of calcium sulfate into the stabilized GS samples decreased the particle-particle separation, which was caused by the calcium cations exchange between clay particles and calcium sulfate. In contrast, the presence of sodium sulfate with any contents has the tendency to increase the particle-particle separation, which is caused by the sodium cations exchange between clay particles and sodium sulfate. The hydrated lime in contact with water increased the pH value of the solution (Eq. (1)). The acceleration of the pozzolanic reactions rate leads to the acceleration of NP dissolution, which forms monosilicates \([\text{SiO(OH)}_3]^-\) and aluminates \([\text{Al(OH)}_4]^-\) compounds, as shown in Fig. 16. According to Shi and Day [71], when calcium cations \((\text{Ca}^{2+})\) contact these compounds, the adhesion materials such as \(\text{C}--\text{S}--\text{H}\) and \(\text{C}--\text{A}--\text{H}\) can be formed as follows (Eqs. (5) and (6)):

\[
\text{Y} \left[ \text{SiO(OH)}_3 \right]^- + X\text{Ca}^{2+} + (Z - X - Y)\text{H}_2\text{O} + (2X - Y)\text{OH}^- \rightarrow C_X - S_Y - H_Z
\]

\((\text{Formation of calcium silicates hydrates})\)

\[
2\left[ \text{Al(OH)}_4 \right]^- + 4\text{Ca}^{2+} + 6\text{H}_2\text{O} + 6\text{OH}^- \rightarrow C_4\text{AH}_{13}
\]

\((\text{Formation of calcium aluminates hydrates})\)

The role of these compounds (\(\text{C}--\text{S}--\text{H}\) and \(\text{C}--\text{A}--\text{H}\)) in the GS–lime–NP mixture is to produce a layer of stable protective film that envelops and bends the soil particles together, seals the voids between them (by decreasing the void index), and consequently reduces the water permeability and improves the compactness of the soil [31].

Moreover, in the presence of water, the reaction between sodium sulfate and GS–lime or GS–lime–NP mixtures leads to the formation of sodium hydroxide \((\text{NaOH})\), which develops a higher alkaline solution as compared with that of the calcium hydroxide \([\text{Ca(OH)}_2]\). In fact, a high pH developed by NaOH produced a large amount of silica \((\text{SiO}_2)\) and alumina \((\text{Al}_2\text{O}_3)\), which react with the remaining lime to form a high amount of cementitious products, which are responsible for the increase of the UCS at an early stage. However, the dissolution of sodium and calcium sulfate salts (as shown in Eqs. (2) and (3)), increased the concentration of sulfate ions \((\text{SO}_4^{2-})\) and then leads to the formation of the ettringite \([\text{Ca}_6\text{Al}_{2}((\text{SO}_4)_3(\text{OH})_{12}, 26\text{H}_2\text{O})\), shown as Eq. (7) and Fig. 16:

\[
6\text{Ca}^{2+} + 2\text{Al}((\text{OH})(4) + 6\text{H}_2\text{O} + 4\text{OH}^- + 3(\text{SO}_4)^{2-} \rightarrow \text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}, 26\text{H}_2\text{O}
\]

\((\text{Formation of ettringite mineral})\)

At an early stage, the effect of the primary ettringite mineral on the GS–lime–NP–\(\text{CaSO}_4\cdot2\text{H}_2\text{O}\) mixture is to increase its density, and consequently the strength gain. However, for a longer curing period and for the GS–lime mixture containing 4 and 6% sodium sulfate, the UCS was...
significantly decreased due to the formation of an expansive ettringite mineral, which is known as a secondary ettringite mineral [25]. In contrast, with a low sodium sulfate content (2%) and at any curing period, the secondary ettringite mineral was found to have a better effect by increasing the UCS of the stabilized GS.

However, the mechanism involved in PF–reinforced lime-stabilized GS is the physical interaction between the PF and soil particles, especially with the presence of cementing agents developed during pozzolanic reactions. As the PF content and cementing agents increase, the physical interaction between PF and soil particles increases and leads to an increase in the surface friction between them, and consequently the increase in the UCS was done.

On the other hand, the decrease in the UCS beyond 2%PF content can be explained by the abundant amount of PF adhered to each other to form lumps, causing a degree of interlocking and friction between PF and soil particles reduced, and hence decreases the UCS. However, the adverse effect of sodium sulfate on the lime-stabilized GS can be entirely suppressed when using the combination of 2%PF + 20%NP as an optimal treatment (Fig. 16). In general, the UCS variation was found to depend not only on the type of sulfate used but depend also on its amount, PF content, and curing period.

6 Conclusions
The effect of different contents of NP–PF mixture on the UCS behavior of the GS samples with and without sulfates was studied. Based on the results of the tests, the following conclusions can be drawn:

- Test results showed that, in the absence of sulfate, lime can be considered as a very efficient stabilizer in increasing the UCS of the clayey soils by adding only 8%L. In addition, the GS can be used as a subgrade material for most engineering projects such as road pavement, or even under brutal environmental conditions due to the improvement of its UCS due to the addition of 8%L in combination with 20%NP.
- The calcium sulfate added with any content into the GS samples stabilized with 8%L, 20%NP, or their combination (8%L + 20%NP) was found to be very effective in increasing the UCS, especially with the curing period. Also, the sodium sulfate with a low content (2%) showed the same effect as that of the calcium sulfate.
- The presence of sodium sulfate in lime-stabilized GS samples resulted in an abnormal decrease in the UCS of the GS. However, with 20%NP addition, the adverse effect of sodium sulfate on the lime-stabilized GS was considerably reduced and the strength was also improved.
- The XRD patterns of the GS stabilized with 8%L alone or in combination with 20%NP confirmed that the abnormal decrease in the UCS is due to the formation of an expansive ettringite mineral (Known as secondary ettringite mineral) due to the sodium sulfate addition.
- The presence of sodium sulfate with a high content in natural soils or in the groundwater should be considered and classified as a deleterious element for soil stabilization because of its harmful effect, which greatly affects the stabilization process. In addition, soil stabilization success depends strongly on the type of additive used and its amount, the type of sulfate and its content, PF content and the curing period.
- In general, the obtained results point out that treatment of the GS containing a high amount of sodium sulfate with lime may not always be a perfect solution to the high swelling and low strength problems. Soil stabilization using lime alone can cause the formation of an expansive ettringite mineral in the soil containing a high amount of sodium sulfate and consequently decrease in its UCS. Therefore, the use of lime as a soil stabilizer in sulfate-bearing soils should be approached with great care. The determination of the sulfate level in soils will be very important before any field application. However, the use of NP–PF mixture in lime-stabilized GS is highly recommended because of its beneficial effect in improving the strength of the expansive soil with and without sulfates.

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