Corrigendum: Effect of adding nano-calcined clay and nano-lime on the geotechnical properties of expansive clayey soil (*IOP Conf. Ser.: Mater. Sci. Eng.* **615** 012058)

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This corrigendum corrects the following error. There was a missing reference in the reference list. This has been included as reference 18.

[18] Al-Swaidani A M, Hammoud I., al-Ghuraibi I and Mezyab A 2019 Nanocalcined clay and nanolime as stabilizing agents for expansive clayey soil: Some geotechnical properties Advances in Civil Engineering Materials 8(3) 327-345

[19] Gueddouda M K, Goual I, Lamara M and Mekarta B 2011 Chemical stabilization of expansive clays from Algeria Global J. of Researches in Engineering: J. General Engineering 11(5) 1-8

Reference 18 should have been referred to at the following points.

[20] Kariuki PC, Shephered K D and Van Der Meer F D 2006 Spectroscopy as a tool for studying swelling soils. In: alRawas AA, Goosen, MFA editors Expansive soils-recent advances in characterization and treatment 15-24 (London, UK: Taylor and Francis Group)

Table 1. The reference [18] should have been indicated in the caption

Figure 2(a), Figure 2(b) and Figure 2(c). The reference [18] should have been indicated.

Figure 7 the reference [18] should have been indicated.

The reference (Gueddouda et al.) numbered as 18 in the older copy was corrected to 19. See the selection of the reference list above.

Figure 8 should include a reference to [18].

Figure 9. The reference [18] should be indicated.

The reference (Kariuki et al.) numbered 19 in the older copy was corrected in this version to 20. See attached reference list above.

Table 4. The reference [18] should have been indicated

References list Reference [18] was added in this version. See above list.
Effect of adding nano-calcined clay and nano-lime on the geotechnical properties of expansive clayey soil

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Abstract. The objective of this paper is to investigate the effect of nano-calcined clay (NCC) and nano-lime (NL) on some engineering properties of the expansive clayey soil. Three soil samples quarried from three different sites in the south of Syria have been investigated. They were thermally treated up to three different levels (450, 650 and 850°C) for 3 hours. Then, they were ground to have a particle size of less than 100 nano-meter. Three replacement levels of NCC were used, i.e. 0%, 1% and 2%. XRD technique has been employed to detect the phases occurring in the clayey samples before and after the thermal treatment. Pozzolanic activity of the calcined clayey soil has been studied using the modified Chapelle test. Atterberg limits, compaction, free swell, swelling pressure, linear shrinkage, shear strength and CBR have particularly been investigated. Test results revealed the positive effect of NCC. Plasticity index (PI) was reduced by more than 50% when 2% NCC was added to the natural soil. In addition, 0.6% NL was added to further investigate the combined effect of NL and NCC on the properties of the clayey soil. All investigated properties were significantly improved when NCC and NL were added together, i.e. swelling pressure and linear shrinkage values were reduced to less than 15% when compared with those of the natural soil.

1. Introduction
Expansive clayey soil has problematic properties. Many additives have been used to stabilize the clayey soil. Lime is considered the oldest stabilizer for clayey soil [1]. Many researchers reported the positive combined effect of adding lime and mineral admixtures on the stabilization of expansive clayey soil [2-6]. In Syria, a wide area is covered with clayey soils. They occupy about 12% of the country’s area [5]. They caused serious damage to the infrastructure. Therefore, there will be a growing need for stabilized clayey soil in the future, particularly in the rebuilding stage in Syria. Calcined clay has widely been used as cement replacement [7-11]. However, the authors believe that very little work or even no detailed work on using calcined clay as a soil stabilizing agent were found in the literature.

Nano-additives react very actively with other particles in the soil matrix. Adding nano-additives to the natural soil not only increases strength and compressibility but also decreases swelling and plasticity index [12]. Furthermore, nano-additives reduce the porosity by filling the space between soil particles and thus bond the particles together [13]. In the study of Luo et al. [14], nano-alumina contents of 0, 1, 2 and 3% were added to the clay soil stabilized with sewage sludge ash and cement. In their study, it
was found that the UCS of the soil treated with 1% nano-alumina was about 4 higher than that of the nano-free soil after 7 day curing. In addition, The PI value and the swell potential for 1% nano-alumina were the smallest. Taha and Taha [15], in their detailed experimental study, investigated the effect of adding each of nano-clay, nano-alumina and nano-copper on the expansion and shrinkage properties of the clayey soil. They found that the addition of nano-alumina reduces both the expansive and shrinkage strains. However, they also found that the nano-clay particles had very little effect on the properties of the soil, and sometimes their effect was negative rather than positive. The increase in nano-clay causes an increase in plasticity index and linear shrinkage. These results were attributed to the nature of nano-clay, which is originally an expansive clay. Moreover, adding nano-copper to soil gave more positive effect than nano-alumina in terms of expansive and shrinkage strain. The particle density of nano-copper increases the specific gravity of the stabilized soil leading to an increase in the maximum dry density of the soil mixture. The increase in the dry density subsequently leads to decrease in the soil shrinkage and expansive strains. In a study conducted by Pham and Nguyen [16], adding nano-silica reduced significantly the swelling index of clay. Mohammadin and Niazzian [17] found that the addition of nano-clay increases the shear strength of treated clayey soil. Thus, adding nano materials to clayey soil, even at low dosages, can enhance its properties.

The objective of the current study is to investigate the effect of adding NCC and NL on the geotechnical properties of expansive clayey soil. The examined properties of the modified clayey soil are; Atterberg limits, compaction, free swell, pressure swell, linear shrinkage, shear strength and CBR. The study can be useful for other areas of similar geology, such as Jordan and KSA and could be of a great importance in the rebuilding stage in Syria.

2. Materials and methods

The investigated clayey soils have been quarried from three different sites in the south of Syria. Table 1 shows some important properties of the investigated clay samples. Quick lime obtained from Hama city has been used in the experimental part. It has been calcined at 950 °C. Its CaO content is about 94%. The obtained quick lime was ground in a laboratory ball mill for six hours. SEM, EDX, AFM and grading of the investigated NL are illustrated in figure 1. The quarried clayey soils have been thermally treated up to three different temperatures; 450, 650 and 850 °C, respectively, for three hours. The clayey samples thermally treated at 850 °C were ground for six hours. SEM, EDX, AFM and grading of the studied NCC are illustrated in figure 2. Six soil mixtures for each clay soil have been prepared. Table 2 shows the mixture ingredient contents as a percent. The mixtures have been designated according to the replacement level. For instance, NCC2NL0.6 refers to the clay mixture, which contains 2% nano-calcined clay and 0.6% nano-lime. All replacement levels were made by mass on a dry basis.

Table 1. Some important properties of the studied clayey soils.

| Soil | Depth (m) | Specific gravity | Passing (%) sieve nr. 200 | Plasticity Index (%) | Soil classification (USCS) | Optimum Moisture Content (OMC)(%) | Maximum Dry Density(MDD)(kN/m³) |
|------|-----------|-----------------|---------------------------|----------------------|---------------------------|----------------------------------|---------------------------------|
| S1   | 1         | 2.69            | 93                        | 29                   | CH                        | 25.5                             | 14.2                            |
| S2   | 0.75      | 2.67            | 91.5                      | 35                   | CH                        | 23                               | 13.8                            |
| S3   | 1.25      | 2.72            | 95                        | 42                   | CH                        | 26                               | 13.0                            |

Table 2. Clay mixtures used in the study.

| Soil mixture     | Percentage (%) |
|------------------|----------------|
| Natural clayey soil | NCC | NL |
| NCC0NL0 (control) | 100 | 0  | 0  |
| NCC0NL0.6        | 99.4| 0  | 0.6|
| NCC1NL0          | 99  | 1  | 0  |
| NCC2NL0          | 98  | 2  | 0  |
The laboratory tests conducted for this work can be summarized as follows:

1) XRD analysis; SATOE STADI X-ray diffractometer (XRD), operated at 40 kV and 30 mA with a scan mode ranging from 5 to 70° and a scan speed of 2°/min has been employed.

2) Modified Chapelle test. This test was employed to measure the pozzolanic activity of the calcined clayey samples. It was conducted in accordance with NF P 18-513. Another test has been carried out using cubic mortar samples. These samples have been prepared by mixing the thermally treated clay with lime and standard sand, and cured for 2, 7 and 28 days, respectively.

3) Atterberg limits test. The test has been conducted in accordance with ASTM D4318 (2000), at room temperature.

4) Standard Proctor compaction test. The test was done to determine the maximum dry density (MDD) and optimum moisture content (OMC) of the investigated soils. This test has been conducted in accordance with ASTM D698 (2000). The soil mixtures, with or without additives, were thoroughly mixed for 1 h prior to compaction.

5) Free swelling and swelling pressure of the soil mixtures were measured in the oedometer device in accordance with ASTM D 4645 & ASTM D 2435. Deformation readings were recorded at 1, 2, 4, 8, 15 and 30 minutes and at 1, 2, 4, 8, 24, 48, 72 and 96 hours. Vertical pressures of 5, 10, 20, 40 and 80 kPa were applied to each sample.

6) Linear shrinkage test. This test has been conducted in accordance with BS 1377 (1990). The linear shrinkage index ($L_{SI}$) of the soil was calculated using the following formulas (al-Swaidani et al., 2016):

$$L_{SI} = 1 - \frac{L_{avg}}{L_{0}}$$

Where $L_{avg}$ is the average length of soil (mm), and $L_{0}$ is the original length of brass mold (mm).

7) Direct shear strength test. This test has been carried out in accordance with ASTM D6528. The samples were prepared in the shear ring (6cm × 6cm × 2.2cm) based on the proctor results. The samples have been cured for 24 h in plastic bags at the ambient temperature. The rate of horizontal displacement was maintained at 5 mm/min. Samples were sheared under undrained condition. For each soil mixture, three samples were prepared and the average was reported.

8) CBR test. This test was carried out in accordance with ASTM D1883. The soil samples were tested after being soaked in water for 4 days.
Figure 1. SEM micrograph (a), EDX analysis (b), AFM (c) and grading (d) of the investigated NL.

Figure 2. SEM micrograph (a), EDX analysis (b), AFM (c) and grading (d) of the investigated NCC.
3. Results and discussion

3.1. XRD analysis

Figure 3 shows the results of XRD analysis for the investigated three soils before and after the thermal treatment. The main clay minerals; kaolinite, illite and montmorillonite are clearly seen in figure 3. Quartz, feldspar and calcite as non-clay minerals were detected, as well. The thermal treatment up to 450 °C did not show any appreciable change in the mineralogy phases where the minerals retain their crystalline phases. At temperature 650°C, peaks representing kaolinite and illite started to decay, and so did the peak of montmorillonite but at a lower rate. All peaks of clay minerals approximately disappeared when the investigated soils were thermally treated at 850°C. Only peaks of non-clay minerals still exist. This gradual change in the peaks representing clay minerals is an evidence of the transition to the amorphous phase that took place due to the thermal treatment.

3.2. Pozzolanic activity

The principle of the modified Chapelle test depends on determination of the amount of calcium hydroxide Ca(OH)$_2$ that can be fixed by the thermally treated clay. The amount of CH fixed by the thermally treated clay can be calculated using the following formulas:

$$Ca(OH)_2=2\times \frac{V_1-V_2}{V_1} \times \frac{74}{56} \times 1000$$

where $V_1$ is the volume of HCl needed to react with CH (without thermally treated clay) and $V_2$ is the volume of HCl needed to react with CH (with thermally treated clay).

French Norm (NF P 18-513) requires the amount of 700mg as a minimum level to consider the pozzolanic activity of the tested material acceptable.

![Figure 3. XRD analysis of the investigated soils before and after the thermal treatment (I: illite; Q: quartz; M: montmorillonite; K: kaolin; F: feldspar; C: calcite).](image)

The results of the modified Chapelle test for all the investigated clayey soil before and after the thermal treatment are presented in figure 4. As shown in figure 4 the Soil (S1), which demonstrated the highest amorphous phase in the XRD analysis, gave the highest number of pozzolanic activity (745 mg) when heated up to 850 °C. This value exceeded the acceptable limit (700 mg) specified by NF P 18-513. The clayey soils S2 and S3 even when heated up to 850 °C had values of relatively lower than 700 mg.
As the strength required for soil application is relatively lower than that for structural concrete, the obtained pozzolanic activity numbers can be considered satisfactory.

![Figure 4. Results of modified Chapelle test for the investigated soils.](image)

**Figure 4.** Results of modified Chapelle test for the investigated soils.

| Table 3. Compressive strength of clay mortar cubes cured for 2, 7 and 28 days. |
|---------------------------------------------------------------|
| **Compressive strength (MPa)**                                    |
| **Clay soils** | **2 days** | **7 days** | **28 days** |
| Soil 1 | 2.64 | 6.73 | 11.93 |
| Soil 2 | 2.80 | 5.23 | 8.45 |
| Soil 3 | 2.35 | 5.36 | 9.86 |

### 3.3. Atterberg limits

Figure 5 shows the results of plasticity index of the clay soil mixtures. The decrease in PI values indicates the improvement of soil workability. As illustrated in figure 5, the three soils showed a significant decrease in PI when NL is added by 0.6%. Similar behaviour was reported in the literature for soils with a similar classification [3, 5, 6]. This improvement could be attributed to the flocculation and aggregation of colloidal clay particle when NL is added. The addition of NCC enhances the workability as a result of a reduction in the plasticity of the soils. The addition of NCC to the three natural soils had reduced the PI by about 40% and 60% when added at percentages of 1% and 2%, respectively. However, the combination of 2% NCC and 0.6% NL exhibited the highest influence when the PI is concerned.

![Figure 5. Plasticity index of the investigated clay mixtures.](image)
3.4. Compaction test results
Figures 6 & 7 show the effect of NCC, NL and their combinations on the compaction properties of the investigated soils. It can be clearly seen that adding 0.6 NL alone to the three soils decreases MDD and increases OMC. This can be attributed to the lower specific gravity of lime and the higher water retention property of lime, when compared with those of soils [5, 6]. Adding NCC alone caused a decrease in OMC and an increase in MDD with the increase of NCC content. This can be due to the lower affinity to water and the higher specific gravity of NCC, when compared to the natural soil. The combination of NL and NCC kept the values of the maximum dry density (MDD) and the optimum moisture content (OMC) around their values in the natural soils as shown in figures 6 & 7, respectively.

![Figure 6. Maximum dry density of the investigated clay mixtures.](image1)

![Figure 7. Optimum moisture content of the investigated clay soil mixtures.](image2)

3.5. Free swell and swelling pressure
Generally, swelling potential has been used to describe the ability of a soil to swell, in terms of volume change or the pressure required to prevent swelling [18]. The results of free swelling and swelling pressure are displayed in figures 8 & 9, respectively. The natural soils showed the highest values of free swelling and swelling pressure. They ranged from (8.4%) to (10.6%) after 96 hours. This free swelling was restricted and the swelling pressure was reduced when 1% and 2% NCC were added to the soil mixture. Further restriction and pressure reduction were observed when 0.6% NL was added to the
natural soils. The best result was obtained when NCC and NL were added together at the following percentages: 2% and 0.6%, respectively.

3.6. Linear shrinkage index
The linear shrinkage test was conducted on elongated clayey soil samples to measure shrinkage deformation when the samples are placed in a brass mold and dried in an oven for twenty-four hours. The test results are illustrated in figure 10. As shown in figure 10, a significant reduction in linear shrinkage was observed when NL was added by 0.6%. This reduction can be attributed to the chemical reactions between NL and clay [19]. Adding NCC alone had also caused a positive effect, particularly when added at 2% NCC replacement. Its linear shrinkage index was reduced by half when compared with the untreated soil. The lowest strain was attained in the mixture NCC2NL0.6, which can be considered non-problematic [19].
3.7. Shear strength and CBR characteristics

Table 4 presents the shear strength characteristics and CBR values of the studied expansive clayey soils. It is clearly seen in table 4 that the shear strength characteristics of expansive clayey soils increased with the increase in NCC content. Further increase is observed when NL is added. For instance, with addition of 2% NCC, the angle of internal friction increased by about 20% when compared with the natural clayey soil. This value increased up to 50% in the mixture prepared with both 2% NCC and 0.6 NL. In addition, the cohesion values increased with adding NCC, NL or both. A similar trend was also noted in the CBR test results. This improvement in the shear characteristics and CBR values can be attributed to the formation of cementing materials such as C-S-H and C-S-A-H, as confirmed by SEM and EDX analysis.

3.8. Discussion of results

Addition of nano-calcined clay or nano-lime or both has led to noticeable improvements in the investigated geotechnical properties, such as (i) a significant reduction in PI, (ii) important reductions in free swell and swelling pressure, (iii) a significant reduction in linear shrinkage, (iv) a significant increase in both shear strength and California bearing ratio. The obtained results are well correlated with the calcination temperature of clay, and can be attributed to the following:

1) Calcination of clay up to 850 °C has caused a change from crystalline to amorphous phase, as confirmed by XRD analysis. The amorphous phases in the calcined clay react with the compounds in either soil or lime or both to form cementing compounds such as C-S-H and C-S-A-H. These reactions which are referred to as “pozzolanic reactions” are mainly responsible for the noticeable reductions in the free swelling, swelling pressure and linear shrinkage and the significant increase in shear strength and CBR.

2) The absence of expandable minerals like montmorillonite & illite when clay was calcined up to 850 °C, as detected by XRD analysis, is also responsible for these remarkable improvements.

3) The prolonged grinding of calcined clay and quick lime may form a highly reactive material on the surface of the mineral particles. Therefore, nano-particles react very actively with other compounds in the treated soil.

3.9. Microstructural analysis

Three soil-2 mixtures, i.e. NCC0NL0, NCC2NL0 and NCC2NL0.6, prepared and cured for 7 days were analysed using SEM and EDX techniques. Figures 11 & 12 show the results of the microstructural investigation of NCC0NL0, NCC2NL0 and NCC2NL0.6, respectively. The natural soil (NCC0NL0)
has a discontinuous structure with no detected hydration compounds. However, it is clearly shown from figure 12 (a & b) that the microstructure of the natural soil was significantly modified when either NCC or NCC & NL were added. It can be easily observed in figure 12 (b) that cementing compounds such as C-A-S-H and C-S-H were formed. This observation is in line with that reported by Al-Swaidani et al. [5] and Al-Swaidani et al. [6]. These cementing compounds have been formed as a result of pozzolanic reactions occurred between CaO present in NL and SiO₂ & Al₂O₃ in the soil and NCC.

### Table 4. Angle of internal friction, cohesion and CBR values of clayey soil mixtures.

| Clay mixture | Soil 1 | Soil 2 | Soil 3 |
|--------------|--------|--------|--------|
|              | Angle of internal friction, Φ (°) C (kPa) CBR (%) | Angle of internal friction, Φ (°) C (kPa) CBR (%) | Angle of internal friction, Φ (°) C (kPa) CBR (%) |
| NCC0NL0      | 23 60 2.9 21 | 83 2.5 19 | 90 2.6 |
| NCC0NL0.6    | 29 68 56.2 28 | 90 45.8 24 | 97 41.3 |
| NCC1NL0      | 25 63 32.5 24 | 86 28.4 22 | 94 22.2 |
| NCC2NL0      | 27 66 46.8 25 | 88 34.7 24 | 95 30.8 |
| NCC1NL0.6    | 31 79 73.7 29 | 100 62.8 26 | 114 58.1 |
| NCC2NL0.6    | 33 86 83.5 31 | 113 71.2 28 | 125 67.4 |

### 4. Conclusion

The Effect of adding NCC and NL to the natural expansive clay soil has been studied. Consistency, compaction, swelling, linear shrinkage, shear strength and CBR have particularly been investigated. The following conclusions can be drawn:

1) Calcination of clayey soil up to 850 °C showed a significant transformation in the studied clay from the crystalline to the amorphous phase. This was confirmed by XRD analysis and the modified Chapelle test, where the highest pozzolanic activity were noted in the samples treated at this temperature level.

2) Plasticity index decreased with an increase in NCC content.

3) Use of NCC alone led to a reduction in swelling and compressibility of the natural clay soil. Further reduction was also observed when NL was added.
Figure 12. SEM micrograph of 7 days-cured NCC2NL0 mixture (a) and 7 days-cured NCC2NL0.6 mixture (b) (soil 2).

4) The shear strength characteristics (angle of internal friction and cohesion) have significantly been improved by adding NCC, NL or both. Significant improvements in CBR values were also observed when such nano-additives were used.

5) The results have been confirmed using SEM and EDX analysis. Formation of cementing compounds such as C-S-H and C-A-S-H induced significant improvements in the geotechnical properties of the examined clayey soils.

6) Calcination of the natural expansive clay soil could be considered a promising approach. Adding NCC not only improves the geotechnical properties of the natural clay soil but also saves natural sources.

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