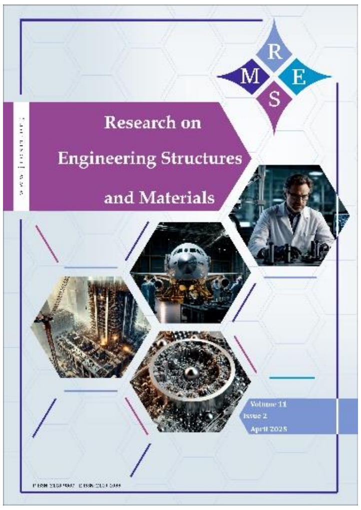
Research on Engineering Structures & Materials

www.jresm.org

Research Group



Synergistic stabilization of highly gypseous soils using nano-calcium carbonate and class C fly ash

Nabil Shihab Al-Zubaidi, Hashim Ghalib Al-Sumaiday

Online Publication Date: 20 July 2026

URL: http://www.jresm.org/archive/resm2026-1789ma0624rs.html
DOI: http://dx.doi.org/10.17515/resm2026-1789ma0624rs

Journal Abbreviation: *Res. Eng. Struct. Mater.*

To cite this article
Al-Zubaidi N S, Al-Sumaiday H G. Synergistic stabilization of highly gypseous soils using nano-calcium carbonate and class C fly ash. *Res. Eng. Struct. Mater.*, 2026; 12(4): 2995-3010

Research on Engineering Structures & Materials
www.jresm.org
Research Group

Research Article

Synergistic stabilization of highly gypseous soils using nano-calcium carbonate and class C fly ash

Nabil Shihab Al-Zubaidi [*,a], Hashim Ghalib Al-Sumaiday [b]

Department of Civil Engineering, College of Engineering, Tikrit University, Iraq

Article Info

Article History:

Received 24 June 2026

Accepted 12 July 2026

Keywords:

Fly ash;
Gypsiferous soil;
Nano calcium carbonate;
Shear strength;
Soil stabilization

Abstract

To address the stability issues associated with highly gypseous soils under water exposure, the effects of nano-calcium carbonate (NCC), Class C fly ash (FA), and their combination on the shear strength and compaction characteristics of highly gypseous soils containing 56% gypsum were systematically investigated. In laboratory tests, three FA concentrations (5%, 10%, and 15%) and three NCC concentrations (0.5%, 1%, and 1.5%) were used as well as a blend of 0.5% NCC and 10% FA. Results indicate remarkable improvements in cohesion and internal friction angle of all treated samples, with the percentage of improvement being most pronounced under soaked conditions. The best combination (0.5% NCC + 10% FA) showed the highest after-soaking cohesion (39.2 kPa) and friction angle (41.8°) with the improvement ratios of 600% and 55.97%, respectively, compared to the untreated soil. Maximum dry density (MDD) increased with NCC content, reaching 1.782 g/cm^3 at 1.5% NCC, while increasing FA content led to a decrease in MDD from 1.67 to 1.605 g/cm^3. The blend had an MDD of 1.70 g/cm^3, effectively optimizing the opposing effects of the two additives on compaction. The improvements are primarily attributed to the void-filling effect of FA, nanoscale densification and enhanced particle bonding by NCC, and the synergistic physicochemical interaction between the two additives. Early pozzolanic reactions may also have contributed. The improvements were further validated by microstructural analyses (XRD and SEM), which confirmed the formation of cementitious products. The mixture proved to be more resistant to the loss of strength due to soaking and more durable in different moisture conditions. The results of this study fill a critical gap in the literature on the combined stabilization of NCC and Class C FA and provide practical suggestions for improving the mechanical and compaction properties of gypseous soils in arid and semi-arid regions.

1. Introduction

Gypseous soils are regarded as one of the most problematic soils in geotechnical and transportation engineering, particularly in the dry and semi-arid regions. These soils are found on some 1.5 million km^2 of Earth's surface, according to the United Nations' Food and Agriculture Organization [1]. Gypseous soils cover about 32% of the total geographical area of Iraq and are widely distributed in different parts of the country as shown in Fig. 1 [2]. For gypsum to form in soils, there must be an external supply of gypsum, arid climate conditions and temperatures above 20°C. Gypsum, accumulated in soil deposits under these environmental conditions, plays an important role in their engineering properties. In civil engineering, gypseous soils are recognized as such on the basis of their gypsum content irrespective of texture or visual characteristics. Other researchers have found some limitations to this categorization, stating quantities of gypsum between 3% and 6% [3].

[*]Corresponding author: dr.hashimrasheed@tu.edu.iq
[a]orcid.org/0009-0004-2523-8645; [b]orcid.org/0000-0002-2237-6223
DOI: http://dx.doi.org/10.17515/resm2026-1789ma0624rs

Gypsum is a naturally occurring mineral, mostly calcium sulphate dihydrate ($CaSO_4 \cdot 2H_2O$). It is commonly found in sedimentary rocks of various geological ages, and it is also frequently found in crystalline deposits of various sizes in soil layers [4]. Gypsum is a temporary cement between soil particles in the absence of water, but the bond is weak and becomes greatly weakened when the soil is wet. The main engineering problem of gypseous soils is their metastable structure and very high susceptibility to collapse in the wet state. Water penetration into the soil causes the dissolution of the gypsum minerals and, as a consequence, the bonds between particles are broken down, thus leading to a fast change of the soil skeleton without any other external load [5]. This can thus result in large settlements and a considerable loss of strength of the soil in a very short time. The gypseous soils are unsuitable for many engineering applications unless proper stabilization measures are adopted for such soils. Moreover, the intensity of these problems increases with increasing gypsum content as the physical and mechanical properties of the soil become more sensitive to changes in moisture, temperature and loading conditions [6]. The collapse behavior of gypsiferous soils has led to serious engineering problems in many infrastructure projects. These challenges are manifested in excessive settlement under foundations, failure of pavement layer, cracking and collapse of residential structures and deterioration of hydraulic structures and irrigation systems [7]. Such failures may lead to significant economic losses and severe safety hazards, especially in areas constructed on highly gypsiferous deposits. This has made the enhancement of engineering properties of these soils a prime area of research in geotechnical engineering.

Many researchers have studied the application of various additives and stabilization agents for solving the problems of gypseous soils. The main aim of these studies has been to decrease soil collapsibility and to improve its mechanical properties in dry and wet conditions. Zedan et al. [8] conducted an extensive study on the gypseous soil with 34–36% of gypsum to investigate the effect of addition of concrete waste and asphalt waste on compaction characteristics, shear strength, collapsibility, and California Bearing Ratio (CBR). They observed that the dry density and the cohesion increased and the moisture content and the collapse potential decreased with the addition of 6 % concrete waste. Similarly, the addition of 2% asphalt waste increased the cohesion and helped in the reduction of soil collapse. The best overall performance was obtained when mixing both waste materials, which increased the dry density, cohesion and CBR values and decreased the tendency for collapse.

In 2020, Al-Obaidi et al. [9] studied the effect of nano-silica and silica fume on soils with 62% gypsum. The effect of each additive on different soil properties was studied. The results showed that the addition of silica fume (SF) and nano-silica fume (NSF) into the highly gypseous soils improved the engineering properties of the soils significantly in dry and wet conditions. Specifically, the addition of nano-silica enhanced the friction angle and reduced the potential for collapse of gypsum soils at nano-silica contents of 1% to 5% under all moisture conditions. Nano-silica at a concentration of 3% resulted in the highest performance. The use of alternative stabilizers for gypseous soils has also been investigated. For instance, Theyab et al. [10] studied effect of casein as a stabilizing agent in 2022. Results indicated that addition of casein decreased the maximum dry density and increased the optimum moisture content. However, this treatment reduced the collapse potential significantly by between 65 and 80 percent and improved the strength characteristics of the soil. Emad and Salman [11] studied the behavior of sandy gypseous soil containing 54% of gypsum and treated with nanomaterials at 1%, 2%, and 3%. They found that the collapse potential decreased and the shear strength increased with increasing concentration of nanomaterials. Moreover, the degree of improvement was higher with longer curing times, indicating that nanomaterials are effective in stabilizing gypseous soils. Bağrıaçık et al. [12] investigated the performance of clay stabilized with conventional $CaCO_3$ and nano-$CaCO_3$. The results demonstrated that nano-$CaCO_3$ achieved superior improvement at a dosage of only 0.75%, which was attributed to its nanoscale particle size and high specific surface area. The nanoparticles effectively penetrated the soil micro-voids, promoted interparticle bonding during curing, and contributed to the development of a denser soil structure. Consequently, the treated soil exhibited higher bearing capacity and reduced settlement compared with soil treated with conventional $CaCO_3$.

Mohammadi et al. [13] investigated the effect of nano-calcium carbonate (NCC) on the geotechnical properties of clayey sand (SC) soil by incorporating 0.3%, 0.7%, 1.1%, and 1.5% NCC. The results showed that NCC significantly increased the uniaxial compressive strength, stiffness, and particle recrystallization while reducing soil deformation. The optimum NCC content was 0.7% for soils containing 10% and 20% clay and 1.1% for soil containing 30% clay, demonstrating that the optimum dosage depends on soil composition and mineralogy. More recently, Muhauwiss et al. [14] studied the performance of gypseous soil stabilized with a biopolymer, Arabic gum, under repeated wetting and drying cycles. The soil tested had about 40 percent of the gypsum. From the results, it was observed that the shear strength increased with the addition of 2% Arabic gum after five cycles where internal friction angle and cohesion reached a maximum value. However, with further cycling, the unconfined compressive strength decreased. The results indicated that Arabic gum can improve the soil stability in the short term, but its effectiveness decreases with the increase in wetting and drying cycles. Jassam et al. [15] specifically reported that treatment of gypseous soil with 45% gypsum with the silane coupling agent KH-570 at dosages of 2%, 4% and 6% resulted in significant decrease of collapse potential and notable improvement of shear strength. Their study showed that cohesion and internal friction increased by 89% and 9.3% under semi-dry condition and 119% and 250% under soaked condition, respectively compared to untreated samples. KH-570 is more costly than traditional stabilizers like lime or cement. But KH-570 is an economical solution for stabilizing gypseous soils because of its low dosage and excellent waterproofing capability.

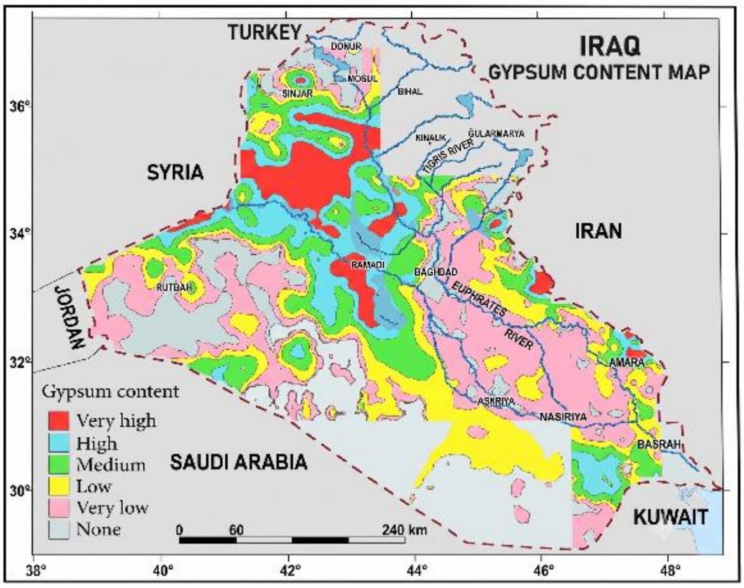


Fig. 1. Map of gypsiferous soil in Iraq [2]

On the other hand, fly ash is often used as soil improvement material in geotechnical engineering. Its application improves compressibility, shear strength and cohesion of soil, as well as reducing earth pressure. Fly ash is used in the base and sub-base layers of road construction as a binder or filler thus resulting in the increased strength of the road in the long run, reduced swelling of expansive soils and enhanced bearing capacity [16,17]. Edil et al. [18] investigated the effectiveness of self-cementing Class C fly ash for stabilizing soft fine-grained soils. Their results showed that the addition of 10% and 18% Class C fly ash significantly improved the California Bearing Ratio (CBR) and resilient modulus (Mr), with the 18% fly ash content producing the highest values among the investigated mixtures. These findings demonstrate the effectiveness of Class C fly ash as a stabilizing agent for improving soil engineering properties. Despite its well-documented benefits, the use of high-calcium Class C fly ash in sulfate- and gypsum-rich soils requires careful consideration. Previous studies have reported that calcium oxide and alumina released from Class C fly ash may react with sulfate ions in the presence of water, leading to the formation of ettringite. Although controlled ettringite formation may contribute to strength development, excessive formation can induce swelling, cracking, and volumetric instability in sulfate-bearing soils.

Therefore, understanding this mineralogical reaction is essential when evaluating the suitability of Class C fly ash for stabilizing highly gypseous soils [19]. Previous studies have demonstrated the effectiveness of various stabilizing agents for improving the engineering behavior of gypseous soils. Nevertheless, several limitations remain. Most investigations evaluated individual additives and focused on specific engineering properties, with limited attention given to the combined performance of different stabilizers. In addition, relatively few studies investigated very highly gypseous soils under both dry and soaked conditions or examined the underlying microstructural mechanisms.

Although significant advances have been made in understanding the stabilization of gypseous soils in the previous studies, there are still some critical research gaps. Most of the previous studies have focused on conventional additives, waste materials, biopolymers, silica-based stabilizers or general nanomaterials. However, the potential of nano calcium carbonate to improve particle bonding and increase soil strength has been investigated in few studies concerned with the strength behaviour of gypseous soils. Likewise, the use of Class C fly ash in stabilizing the highly gypseous soil has not been adequately explored. Also, literature relating to the combined application of nano calcium carbonate and Class C fly ash is scarce and the interaction between the two additives and their combined influence on the strength characteristics of soil is not well understood. The synergy between the high calcium content of Class C fly ash and the nucleation effect of nano-calcium carbonate is expected to enhance particle bonding and promote the formation of cementitious products, thereby improving the shear strength of very highly gypseous soil. The present study is conducted to examine the combined effects of nano calcium carbonate and Class C fly ash on shear strength parameters of gypseous soil by using direct shear tests.

2. Materials and Methodology

2.1. Gypseous Soil

The current study includes a soil sample taken from Tikrit University, in Salah ad-Din Governorate at a depth of 0.5 m. It contains 56 percent of gypsum. Fig. 2 shows the particle size distribution. The soil physical and chemical parameters are shown in Table 1. The soil is classified as poorly graded sand (SP) according to the Unified Soil Classification System (USCS) [20]. The coefficients of curvature (Cc) and uniformity (Cu) shown in Table 1 indicate that the soil is poorly graded. The chemical study indicates that the soil is highly gypseous. The pH values reported show slightly alkaline conditions. Barazanji [21] classified the soil as Very Highly Gypsiferous. These results highlight the need for geotechnical engineers to pay close attention to the hazards associated with such soils in engineering applications. The classification of gypsum soils according to Barazanji is shown in Table 2.

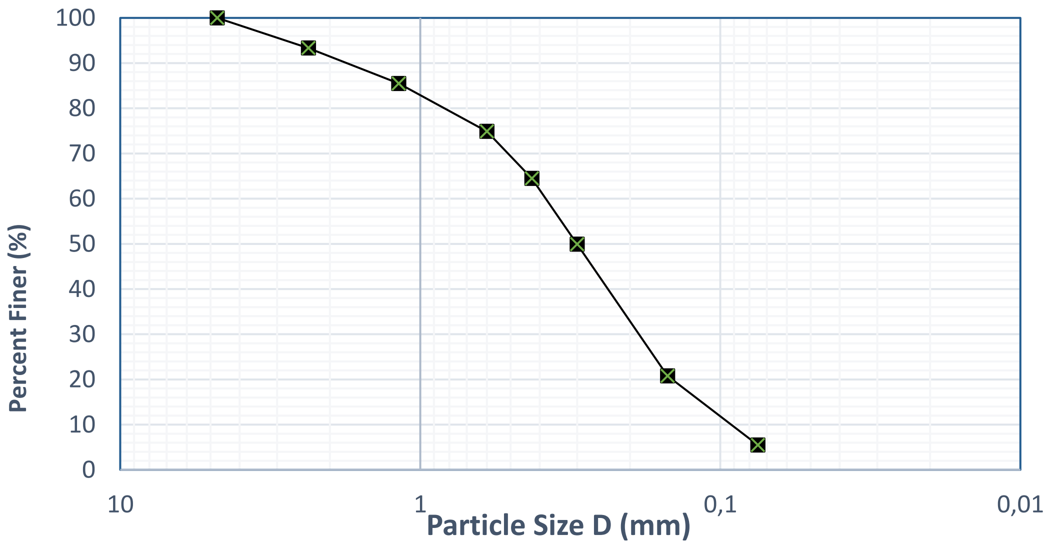


Fig. 2. Particle size distribution of the gypseous soil sample

Table 1. Physical and chemical characteristics of the gypseous soil

Properties/Description		value	Standard
Specific Gravity (Gs)		2.41	(ASTM D854)[22]
Atterberg limits	Liquid limit (L.L)%	25	(ASTM D4318) [23]
	Plastic limit (P.L)%	N.P	
D10 (mm)		0.09	
D30 (mm)		0.19	
D60 (mm)		0.39	
Coefficient of Uniformity (Cu)		4.44	
Coefficient of Curvature (Cc)		1	
USCS (Unified Soil Classification system)		SP	
Unit Weight at Field (γ_f) g/cm^3		1.54	(ASTM D1556) [24]
Field moisture content, (ω)%		6.5	
Maximum Dry Unit Weight (γ_{dmax}) g/cm3		1.71	(ASTM D1557) [25]
Optimum Moisture Content (O.M.C)%		11.5	
Gypsum Content %		56	
Total Soluble Salts (T.S.S) %		61	
pH		8.04	

Table 2. Classification of soil depends on gypsum content [21]

Gypsum Content %	Classification
0-0.3	Non Gypsiferous
0.3-3	Very Low Gypsiferous
3-10	Low Gypsiferous
10-25	Medium Gypsiferous
25-50	Highly Gypsiferous
>50	Very Highly Gypsiferous

2.2 Fly Ash (FA)

Fly ash is a fine powder, a by-product of burning pulverized coal in electrical power generating plants. The flue gases are treated with bag filters or electrostatic precipitators. Fly ash has pozzolanic and cementitious properties that make it important in civil engineering, building, and environmental applications [26]. The fly ash used in this study was manufactured by Euro build Construction Chemicals and Coatings LLC, located in the Industrial Area, Umm Al Quwain, Sharjah, United Arab Emirates, and produced in 2025. The material is shown in Fig. 3.

Fig. 3. Class C fly ash (FA)

The fly ash is of Class C and contains a high percentage of calcium (CaO > 10%) and is pozzolanic and cementitious in nature. These characteristics make the gypsum soil more stable and less liable to collapse. Fly ash was used as an additive in the ratios of 5%, 10% and 15% in this investigation. Table 3 lists the manufacturing specifications, physical characteristics, and oxide composition of the Class C fly ash (FA).

Table 3. Physical properties and oxide composition of Class C FA

Property	Description/value	Oxide Composition	Content %
Color	White to Light Grey	SiO_2	38.6
Specific Gravity	2.37 g/cm3	Al_2O_3	22.16
Blaine Fineness	6500 cm2/g	Fe_2O_3	5.9
Moisture Content	% 1.5-1	CaO	22.3
General Appearance	Fine Powder	MgO	6.86
Particle size	Predominantly < 45 µm	SO_3	0.82
ASTM Classification	ASTM C618- Class C Fly Ash	Na_2O	0.22
		K_2O	0.29
		Loss on Ignition (LOI)	≤ 2.5

2.3 Nano Calcium Carbonate (Nano-$CaCO_3$)

The word ‘nano’ comes from the Greek for one billionth of a meter. One nanometer is equal to 10^{-9} m [27]. Various chemical and physical techniques can be used for the synthesis of Nano calcium carbonate (Nano-$CaCO_3$). Common methods are sol-gel, wet carbonation and chemical precipitation.

Fig. 4. Nano-$CaCO_3$ (NCC)

Table 4. Specifications of Calcium Carbonate Nanoparticles ($CaCO_3$, 97.5%, 15–40 nm).

Property	Typical Value
Form / Appearance	White Nano Powder
Average Particle Size	15 – 40 nm
Specific Surface Area	≈ 40 m^2/g
Chemical Composition	$CaCO_3$ > 97.5%
MgO (Magnesium Oxide)	< 0.9%
Fe (Iron)	< 0.1%
Moisture Content	≈ 0.5%
Whiteness	≈ 93%
Bulk Density	≈ 0.68 g/cm^3
pH	8.0 – 9.0
Insoluble in HCl	< 0.5%
Crystal Shape	Cubic
Manufacturer	Sky Spring Nanomaterials, Inc.

These methods result in very small particles with large surface area and controlled geometries. The synthesis method directly affects the particle size and morphology. This subsequently influences the effectiveness of particle filling in soil pores, improves binding and contributes to interactions with clay minerals [28]. The present study used Nano-$CaCO_3$ as an additive with fly ash to improve the engineering properties of gypseous soils. Fig. 4 shows a sample of Nano-$CaCO_3$ (NCC), and the main specifications are given by the manufacturer in Table 4. Nano-CaCO3 mixing percentages of 0.5%, 1% and 1.5% were tested.

2.4. Sample Preparation

The maximum dry density and optimum moisture content, which were determined by the Modified Proctor Test, were used to prepare the samples after treatment with Class C fly ash, calcium carbonate nanoparticles, or a mixture of both in accordance with ASTM D1557 [25]. Specimens were prepared for testing with the parameters shown in Table 5. All additions were calculated as a percentage of the dry weight of the soil. The soil was oven-dried at 45°C (ASTM D2216-19) [29] and mixed thoroughly with the required amount of NCC and/or FA in dry condition to make sure the distribution was even. The dry mixing was performed manually for a minimum of 5 minutes. Gradually distilled water was added and continuously mixed until a homogeneous mix was obtained and the desired moisture content was achieved. The soil required for each specimen was divided into three equal portions based on mass and placed sequentially into the direct shear box. Each portion was compacted using a hand rammer with seven controlled blows. The number of blows was established through preliminary trials, which showed that seven blows per layer were sufficient to compact the entire soil mass and achieve the target maximum dry density obtained from the Modified Proctor compaction test. After compaction of the final layer, the flatness of the specimen surface was verified before conducting the direct shear test. The sample was tested directly in a semi-dry state. For soaked tests, the selected normal stress was applied before inundation, and the specimen was soaked under this load in distilled water for 24 h prior to shearing. The 24 h soaking period was selected in accordance with previous studies on gypseous soils for short-term shear strength evaluation [30]. The gypseous soil was stabilized by adding 5%, 10%, and 15% of fly ash and 0.5%, 1.0% and 1.5% of nano calcium carbonate (NCC). After establishing the optimal percentages, additional samples were prepared using the same mixing and compaction procedures as per ASTM. Subsequently, the specimens were subjected to testing.

Table 5. Compaction test results

Soil Type	NCC%	FA %	γ_{dmax} (g/cm^3)	O.M.C %
56% Gypsum	0	0	1.715	11.5
	0.5	0	1.76	11.1
	1	0	1.77	10.46
	1.5	0	1.782	10.1
	0	5	1.67	12.75
	0	10	1.62	13.6
	0	15	1.605	14.8
	10	0.5	1.7	12.2

2.5 Direct Shear Test

Direct shear test was performed using a shear testing apparatus according to ASTM D3080 (2011) [31]. A 5 kN load cell was used to measure the shearing force. The specimen was sheared at a displacement rate of 1 mm/min in a direction parallel to the moving shear box. Normal stresses of 50, 100, and 150 kPa were applied during the direct shear tests. Before shearing, each specimen was consolidated under the applied normal stress until the dial gauge reading stabilized. Drainage was maintained throughout both the consolidation and shearing stages in accordance with ASTM D3080. For soaked specimens, the water reservoir in the shear box was filled after applying the normal stress, and the specimens were soaked for 24 h before the shear test. The test was performed to determine shear strength parameters, a cohesion (c) and internal friction angle (ϕ) for untreated and treated gypseous soil stabilized with NCC and FA. The dimensions of the tested specimens were 60 x 60 × 25 mm. Table 6 presents the shear strength parameters of untreated soil (equivalent field unit weight) and blend-treated soils (maximum dry unit weight) under dry and soaked conditions.

Table 6. Shear strength parameters of untreated and blend-treated gypseous soils

Parameter	Untreated soil	Blend-treated soil
Cohesion (c) (kPa)	15.5	42
Cohesion (c) after soaking (kPa)	5.6	39.2
Friction angle (ϕ) (°)	32.12	42.5
Friction angle (ϕ) after soaking (°)	26.8	41.8

3. Results and Discussions

The results showed that the dry density of gypseous soil treated with FA decreased slightly and the Optimum Moisture Content (O.M.C) increased. The soil's dry density increased slightly with NCC treatment, while its optimal water content decreased. The reason for this behavior is that the specific gravity of the soil is higher than that of fly ash, whereas the increase in density with NCC is attributed to enhanced packing and densification due to its nano-size effect. Fig. 5 and 6 show a correlation between FA, NCC concentrations, and maximum dry density of soil, respectively.

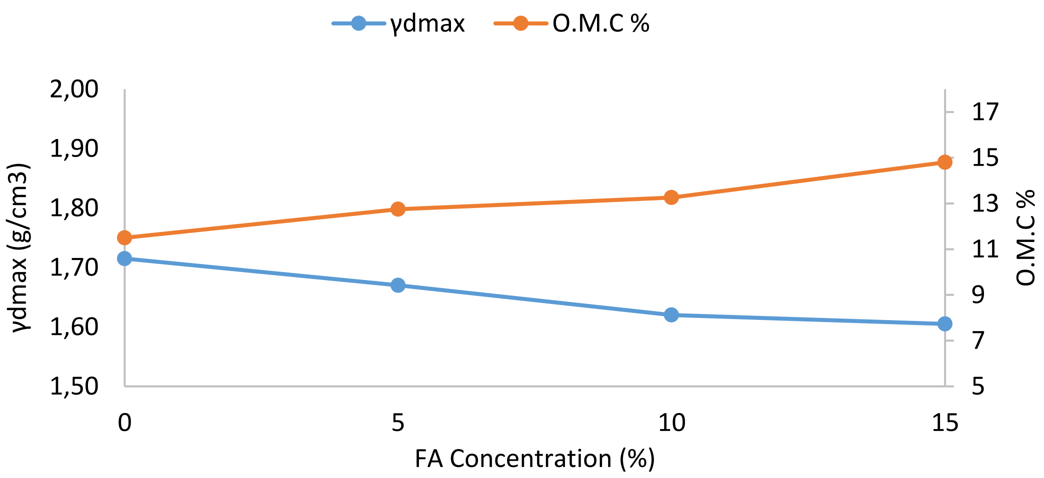


Fig. 5. Relationship between FA concentration, maximum dry density, and optimum moisture content of gypseous soil

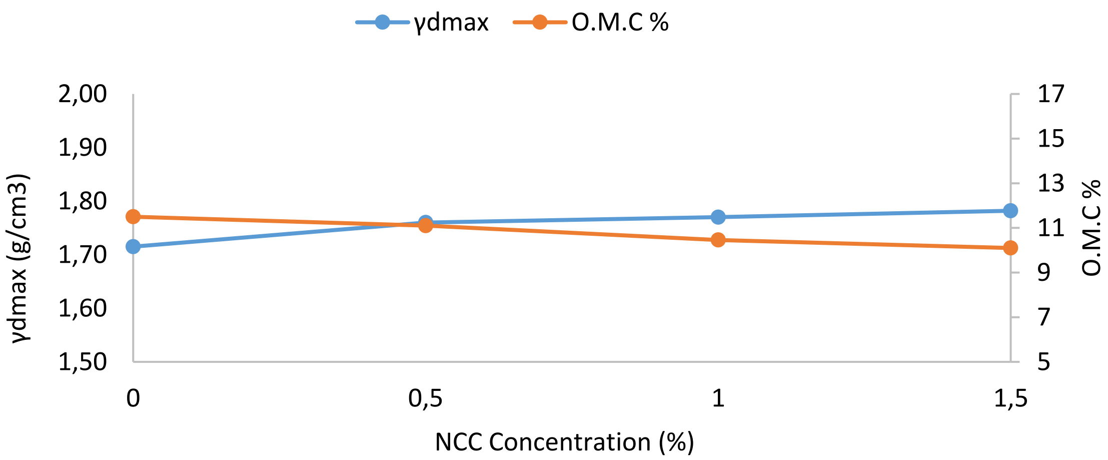


Fig. 6. Relationship between NCC concentration, maximum dry density, and optimum moisture content of gypseous soil

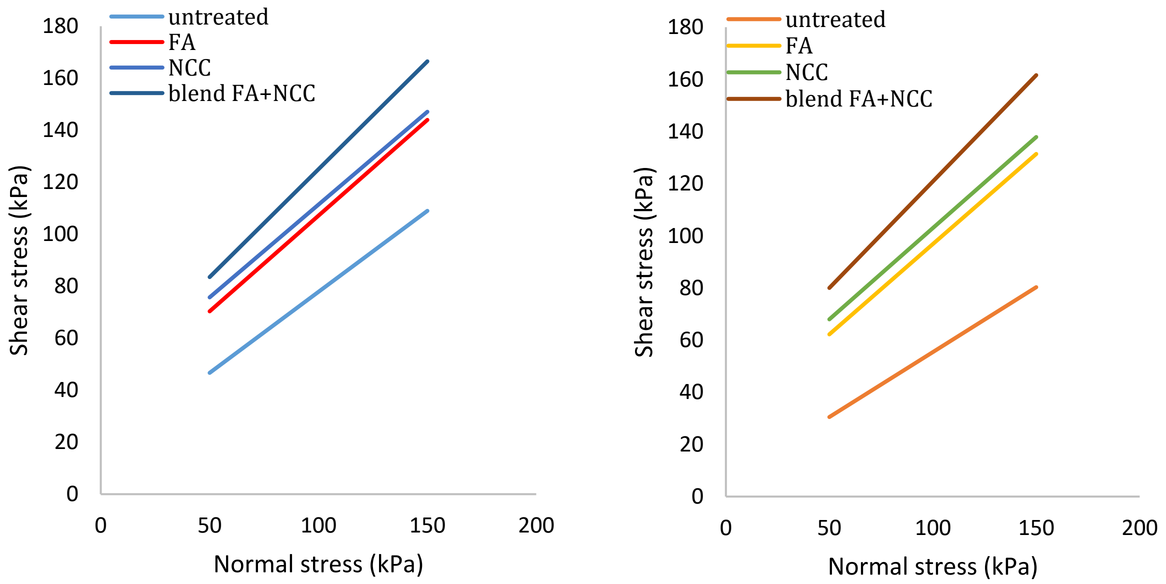
3.1. Direct Shear of Treated Soil

A series of laboratory tests were conducted to study the effect of fly ash (FA) and nano calcium carbonate (NCC) on the shear strength parameters (c, φ) of gypseous soil with various concentrations of additives. Three FA concentrations (5%, 10% and 15%) and NCC concentrations (0.5%, 1%, and 1.5%) were incorporated into the testing program. Additionally, the mixture of 0.5% NCC and 10% FA was applied to soil. As shown in Fig. 7, the overall increase in shear strength for all treated gypseous soil samples (56% gypsum content) is apparent compared with untreated soil in both dry and soaked conditions. The figure shows that the samples treated with the blend of 0.5% NCC and 10% FA have the highest shear strength envelopes for all normal stress levels. The trend directly indicates the better performance of the combined additives compared to FA or NCC alone. Representative photographs of the specimens treated with the blend of 0.5% NCC and 10% FA after direct shear testing under dry and soaked conditions are presented in Fig. 8, illustrating the physical deformation and failure patterns developed along the shear plane.

Figures 9 and 10 show the effect of FA content on the cohesion and internal friction angle of gypseous soil under semi-dry and soaked conditions. In general, both shear strength parameters increased with increasing FA content under both testing conditions. Under semi-dry conditions, the maximum cohesion and internal friction angle occurred at 15% FA. In contrast, under soaked conditions, both parameters increased up to 10% FA and then decreased slightly at 15% FA, indicating that 10% FA was the optimum content for improving the shear strength parameters after soaking. Overall, the observed improvement in shear strength is consistent with previous studies, which reported that fly ash enhances the engineering properties and strength characteristics of soils, although the optimum dosage depends on soil type, gypsum content, and testing conditions [16- 18].

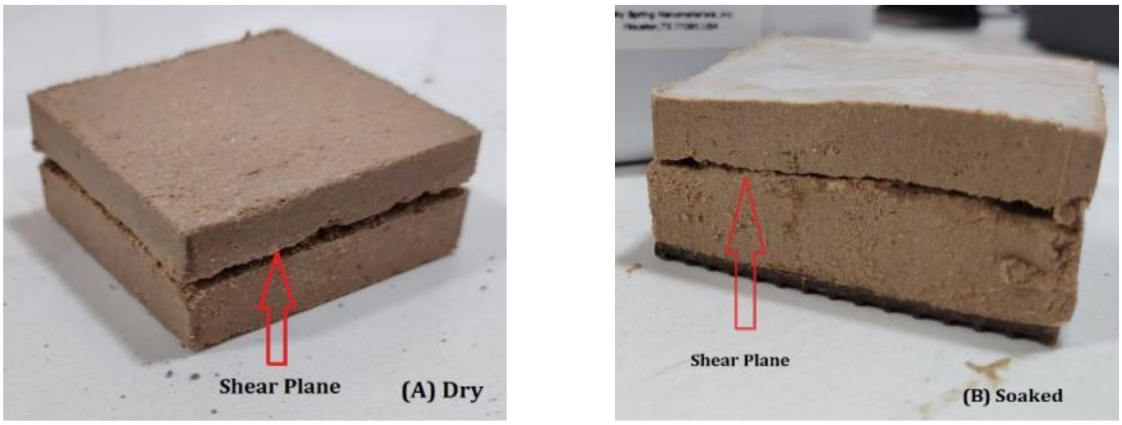
Similarly, Figs. 11 and 12 show the effect of NCC content on the cohesion and internal friction angle of gypseous soil under semi-dry and soaked conditions. In general, both shear strength parameters increased progressively with increasing NCC content under both testing conditions. The maximum cohesion and internal friction angle were obtained at 1.5% NCC under both semi-dry and soaked conditions, indicating that 1.5% was the optimum NCC content within the investigated range. Overall, this trend agrees with previous studies, which reported that nano-calcium carbonate enhances the engineering properties and strength characteristics of soils through improved particle bonding, although the optimum NCC content varies depending on soil type and testing conditions [9, 11-13]. Focusing on the quantitative results, the cohesion (c) after soaking reached 27.6 kPa with 10% FA, 33 kPa with 1.5% NCC, and 39.2 kPa for the blend (0.5% NCC + 10% FA), compared to 5.6 kPa for the untreated soil. The improvement ratios of cohesion in the dry state were 116.19% for FA, 132.26% for NCC and 170.97% for the blend. After soaking, these enhancement ratios increased dramatically to 392.86% (FA), 489.29% (NCC), and 600% (blend), as shown in Fig. 13. With respect to the internal friction angle (ϕ), before soaking, values were 32.12° for untreated soil, 36.5° for 15% FA, 36° for 1.5% NCC, and 42.5° for the blend, with corresponding improvement ratios of 13.64% (FA), 12.08% (NCC), and 32.32% (blend). After soaking, ϕ increased from 26.8° (untreated) to 34.5° (10% FA), 34.6° (1.5% NCC), and 41.8° (blend), with improvement ratios of 28.73% (FA), 29.1% (NCC), and 55.97% (blend), as depicted in Fig .14.

The observed enhancements are mechanistically attributed to the synergistic physical and chemical effects of the additions. The FA helps to fill the voids of the soil and promote pozzolanic reactions that lead to the formation of more cementitious compounds, as found by Zedan et al. [8] and other reviews concerning the use of fly ash for soil stabilization [15, 16]. The NCC because of its nano scale particle size and high surface area, densifies the soil microstructure and improves interparticle interaction in agreement with the findings of Al-Obaidi et al. [9] and Emad and Salman [11]. The highest values of cohesion and friction angle in this study indicate the synergistic effect of the FA and NCC mixture. This fills a significant gap in the existing research and highlights the superiority of combined additives over the single additive method. The blend ratio of 0.5% NCC and 10% FA was selected because it represents a practical and potentially cost-effective solution, suggesting potential improvements in field compaction workability. This approach ensured enhanced shear strength without adverse effects on compaction. However, it is worth mentioning that the considerable increase in shear strength after soaking is a sign of the efficiency of the blend in compensating for the strength loss caused by water, which is a major problem for gypseous soils [9, 11]. Overall, these results indicate that the use of a combination of NCC and FA is a practical and effective way of stabilizing highly gypseous soils, particularly under varying moisture conditions.



(a) (b)

Fig. 7. Results of direct shear tests for untreated and treated soils under: (a) dry condition and (b) soaked condition



(a) (b)

Fig. 8. Physical deformation and failure patterns of gypseous soil treated with a mixture of 10% Class C fly ash and 0.5% nano-calcium carbonate after direct shear testing under (A) dry and (B) soaked conditions

Microstructural and mineralogical studies were conducted using X-ray diffraction (XRD) and scanning electron microscopy (SEM) to evaluate the effect of stabilization of gypseous soil by a mixed additive containing 0.5% nano-calcium carbonate (NCC) and 10% Class C fly ash. The XRD analysis of the untreated soil (Fig. 15) shows pronounced crystalline peaks mainly corresponding to gypsum (G) at $2\theta \approx 11.7°$, $20.7°$, and $31.1°$, confirming its dominance. Additional peaks for quartz (Q) and calcite (C) are also present, indicating their minor roles in the mineralogical composition. Upon treatment with nano calcium carbonate and fly ash (Fig. 16), the pattern undergoes significant transformation. The primary gypsum (G) peaks are notably reduced in intensity, while several new crystalline peaks emerge, especially those associated with ettringite (E) and more pronounced features for quartz (Q) and calcite (C).

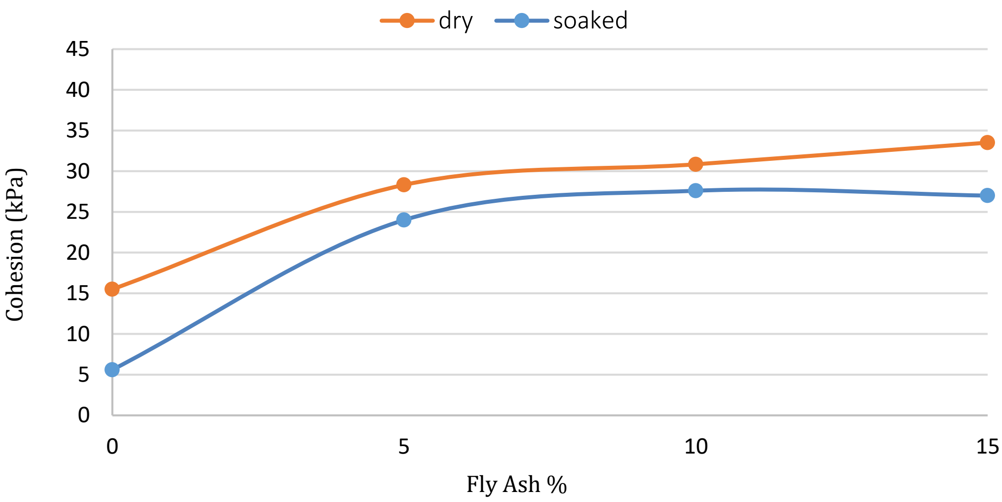


Fig. 9. Effect of fly ash content on cohesion of gypseous soil under dry and soaked conditions

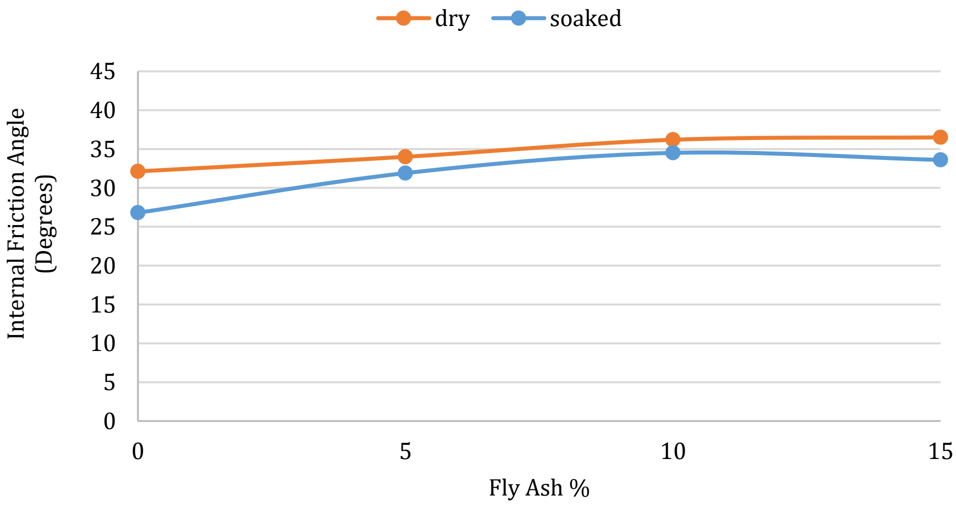


Fig. 10. Effect of fly ash content on internal friction angle of gypseous soil under dry and soaked conditions

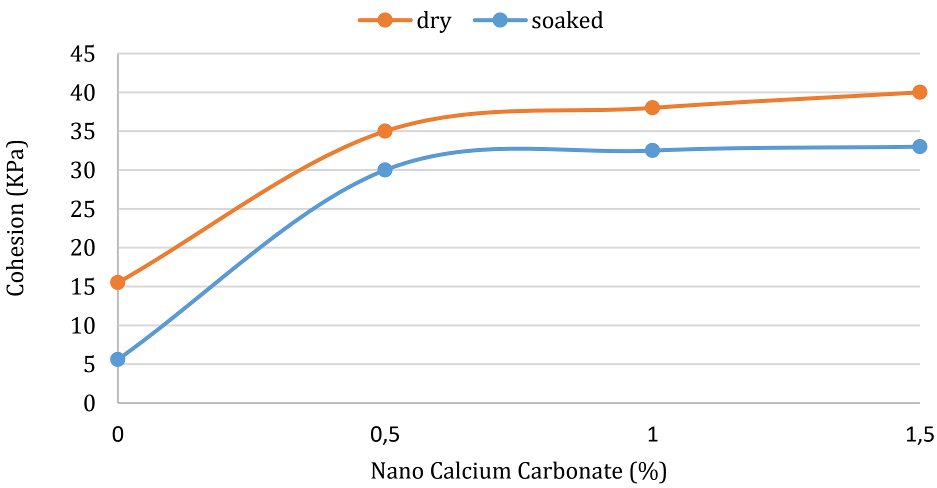


Fig. 11. Effect of nano calcium carbonate content on cohesion of gypseous soil under dry and soaked conditions

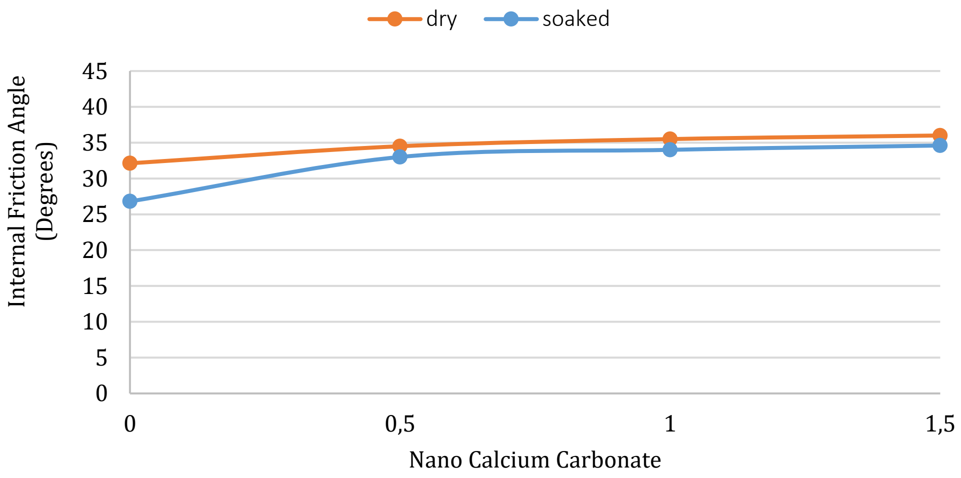


Fig. 12. Effect of nano calcium carbonate content on internal friction angle of gypseous soil under dry and soaked conditions

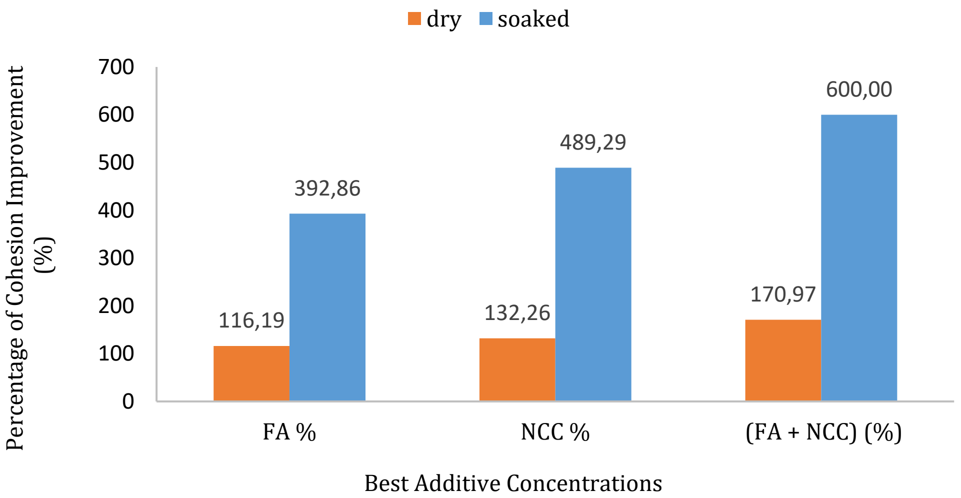


Fig. 13. Percentage improvement in cohesion for gypseous soil treated with best additive concentrations under dry and soaked conditions.

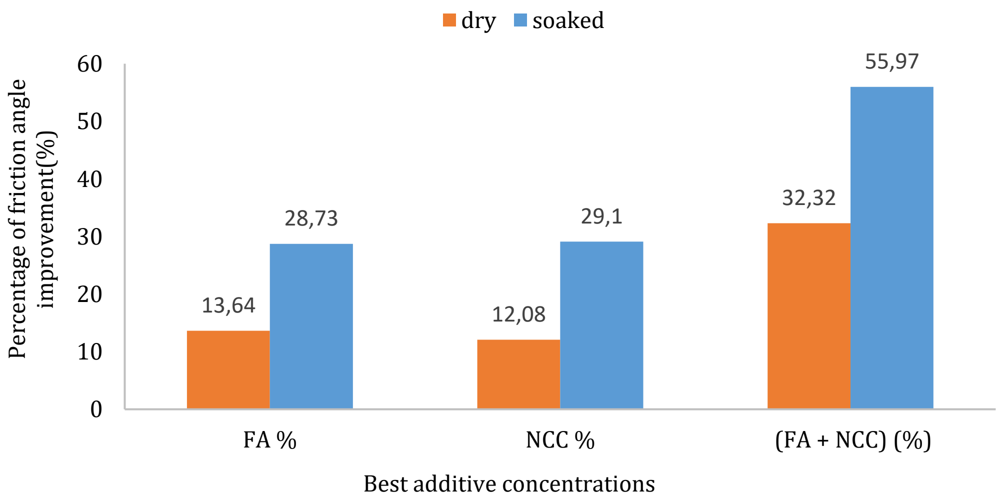


Fig. 14. Percentage improvement in internal friction angle for gypseous soil treated with best additive concentrations under dry and soaked conditions

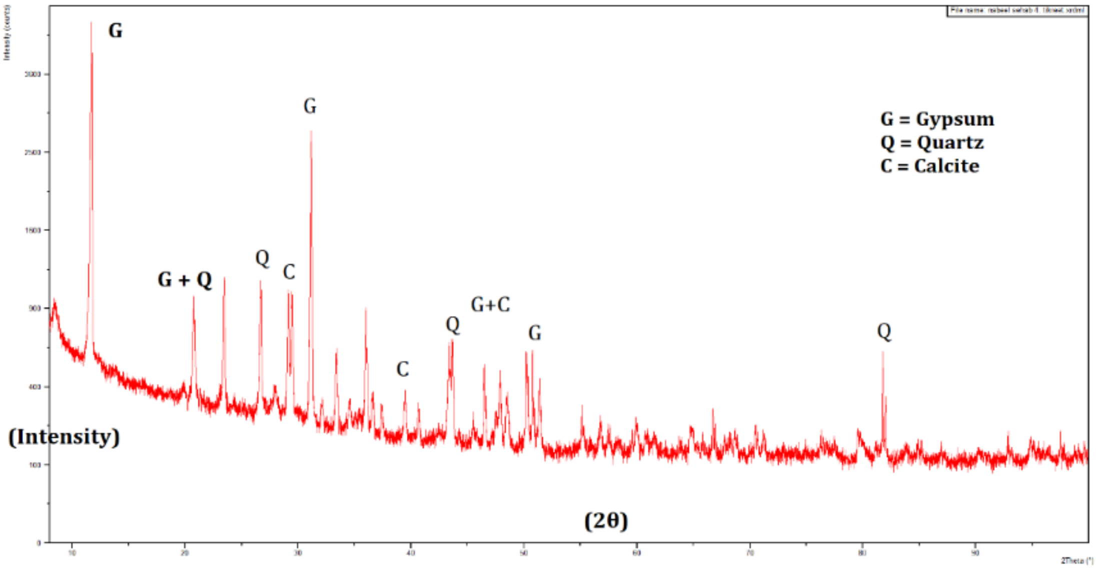


Fig. 15. XRD pattern for untreated gypseous soil

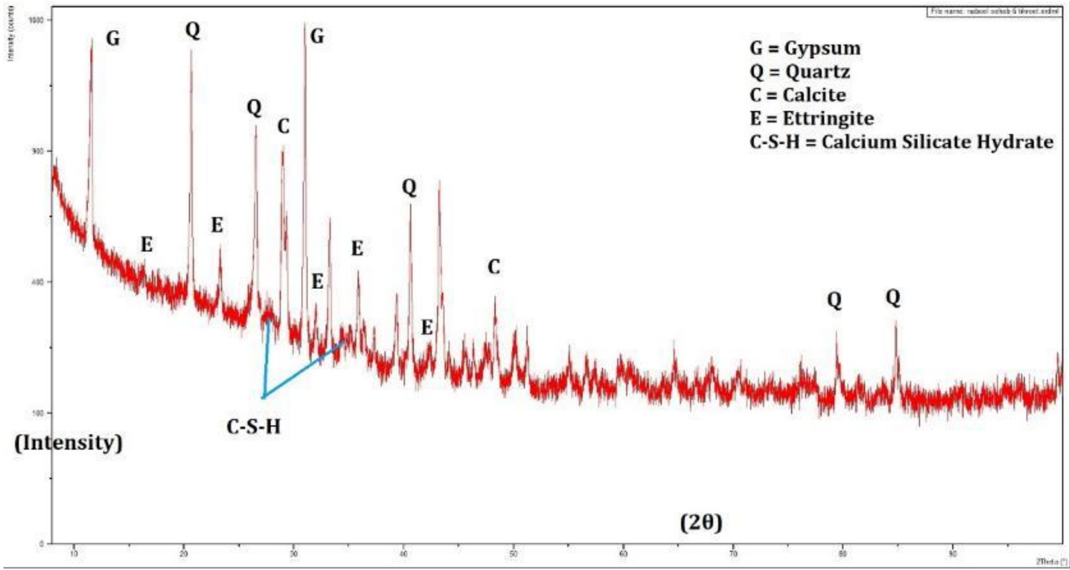


Fig. 16. XRD pattern for treated gypseous soil by 0.5% NCC + 10% FA

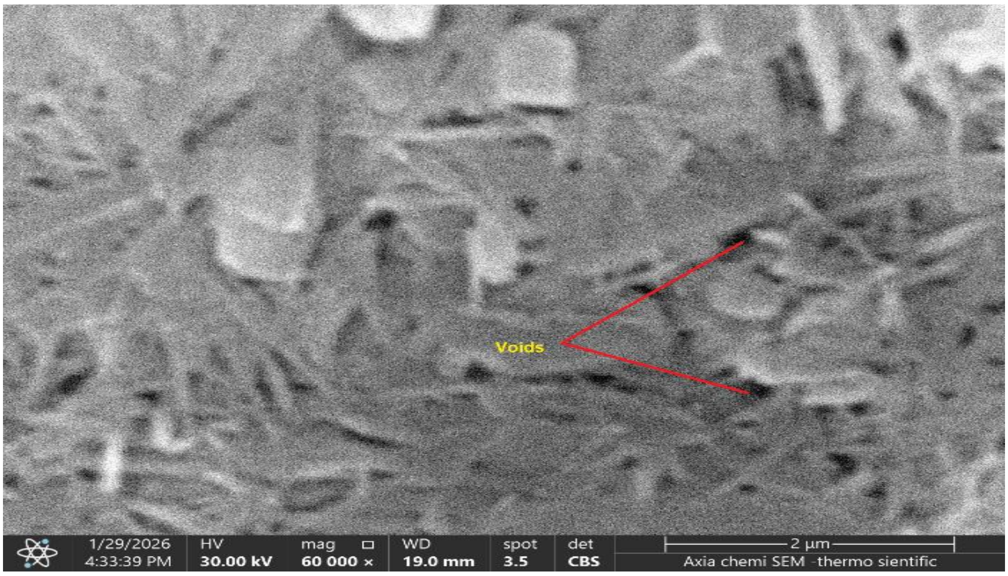


Fig. 17. SEM image of treated gypseous soil with 0.5% NCC and 10%FA

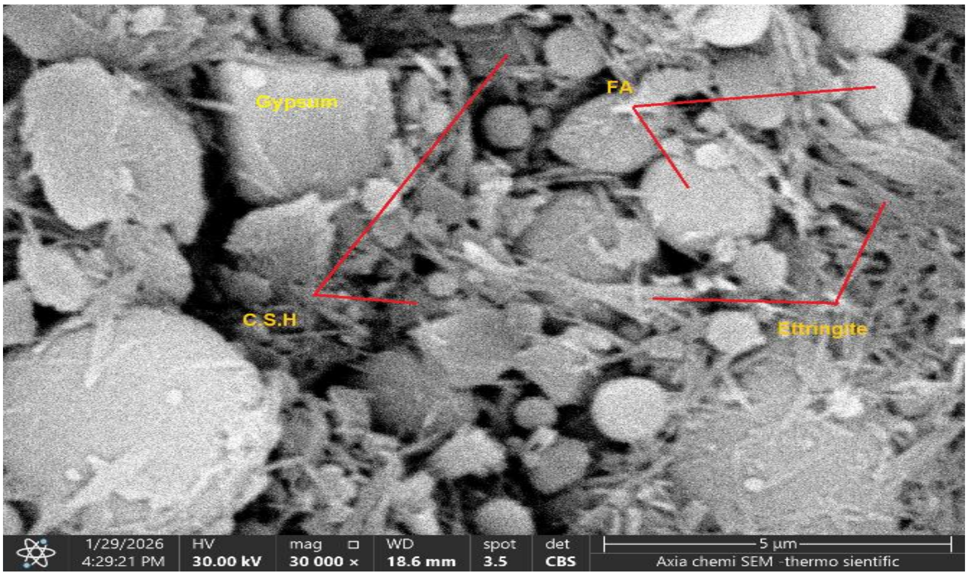


Fig. 18. SEM image of treated gypseous soil with 0.5% NCC and 10%FA

Importantly, instead of a sharp diffraction peak, the presence of calcium silicate hydrate (C-S-H) is identified by a broad amorphous hump or elevation in the baseline within the 2θ range typically associated with C-S-H. This characteristic broadening reflects its poorly crystalline or semi-amorphous structure, which is a common trait for C-S-H phases formed during pozzolanic reactions. The emergence of these features, coupled with the reduction of gypsum peaks, demonstrates extensive mineralogical transformation, with the development of a more complex and stable mixture of crystalline and amorphous phases in the treated soil. Although the formation of ettringite contributed to the observed improvement in soil strength through particle bonding and cementation, previous studies have reported that excessive or delayed ettringite formation in sulfate-rich soils may result in long-term expansion and deterioration under prolonged moisture exposure. The present study did not include long-term durability or expansion tests; therefore, the potential effects of ettringite-induced swelling on the long-term performance of the stabilized soil were beyond the scope of this work and warrant further investigation. The SEM observations confirmed the mineralogical results by showing major changes in the soil fabric. The microstructure of the untreated soil was relatively loose and discontinuous with many voids and little bonding between particles (Fig. 17). In contrast, the treated specimen (Fig. 18) showed a denser and more cohesive matrix with a marked reduction in pore spaces and improved particle interlocking. Within the treated matrix, spherical particles attributable to fly ash and acicular ettringite crystals could be distinguished, where they functioned as bridging and binding phases strengthening the contacts among soil particles. The combined evidence obtained from the XRD and SEM analyses (Figs. 15-18) provides a rational explanation for the improvement in the engineering performance of the stabilized soil, in particular the enhancement in shear strength and durability observed after treatment.

4. Conclusions

This study comprehensively evaluated the effects of nano calcium carbonate (NCC), Class C fly ash (FA), and their combination on the shear strength and compaction properties of highly gypsiferous soils (56% gypsum content). The following key conclusions are drawn:

- The addition of NCC, FA, and their blend resulted in significant improvements in both cohesion and internal friction angle, with the most pronounced percentage of relative improvement observed under soaked conditions compared to the untreated soaked state.
- The blend of 0.5% NCC and 10% FA, selected to balance mechanical gains and density effects, provided the highest observed values for cohesion (39.2 kPa) and internal friction angle (41.8°) after soaking, with improvement ratios of 600% and 55.97%, respectively. These results demonstrate the effectiveness of the blended additives in compensating for the

strength loss caused by soaking, which represents one of the most critical engineering challenges associated with highly gypseous soils.

- The treated soils exhibited marked increases in maximum dry density (MDD)with increasing NCC content, reaching a peak of 1.782 g/cm^3 at 1.5% NCC (compared to 1.715 g/cm^3 for untreated soil). This increase reflects enhanced packing and densification due to the nano-size effect of NCC.
- Conversely, increasing FA content led to a gradual decrease in maximum dry density, from 1.67 g/cm^3 at 5% FA to 1.605 g/cm^3 at 15% FA, likely due to the lower specific gravity and increased water demand for lubrication, which subsequently reduces compaction efficiency.
- The combined addition of 0.5% NCC and 10% FA achieved a maximum dry density of 1.7 g/cm^3, indicating a balanced effect—higher than FA alone, but slightly less than pure NCC—while maintaining improved shear strength characteristics.
- These results confirm that the selected blend ratios not only enhance shear strength and water-resistance but also optimize compaction behavior, suggesting potential improvements in field compaction workability.
- These improvements, confirmed by XRD and SEM analyses, are attributed to the void-filling effect of fly ash, nanoscale densification, enhanced particle bonding by NCC, and the synergistic physicochemical interaction between the two additives. Early pozzolanic reactions may also have contributed, although their full development generally requires longer curing periods.
- Although the formation of ettringite may have contributed to the observed improvement in the engineering properties of the stabilized soil, previous studies have indicated that excessive or delayed ettringite formation in sulfate-rich soils may pose a risk of long-term expansion under prolonged moisture exposure. Therefore, long-term swelling and durability tests are recommended to assess the long-term performance of the proposed stabilization method.
- Overall, this approach addresses a critical gap in the literature regarding the combined use of NCC and Class C FA for gypseous soil stabilization, and provides practical recommendations for geotechnical applications in arid and semi-arid regions.

Acknowledgements

The authors are grateful to the Department of Civil Engineering, College of Engineering, Tikrit University, for supporting this research as a part of the Postgraduate Program under University Order No. 3/7/12727, issued on 08 August 2023.

References

[1] Food and Agriculture Organization of the United Nations. Management of gypsiferous soils. Vol. 62. Rome, Italy: FAO; 1990.
[2] Al-Kaaby FS. Distribution of the gypsiferous soil in Iraq. Baghdad, Iraq; 2007.
[3] Al-Emami OH. Collapsibility of gypseous soil under fluctuation of ground water table [MSc thesis]. Baghdad, Iraq: Department of Building and Construction Engineering, University of Technology; 2007.
[4] Al-Mufty A. Effect of gypsum dissolution on the mechanical behavior of gypseous soil [PhD thesis]. Baghdad, Iraq: University of Baghdad; 1997.
[5] Mansour ZM, Chik Z, Taha MR. On the procedures of soil collapse potential evaluation. J Appl Sci. 2008;8(23):4434-4439. https://doi.org/10.3923/jas.2008.4434.4439
[6] Solis R, Zhang J. Gypsiferous soils: an engineering problem. In: Sinkholes and the Engineering and Environmental Impacts of Karst. Reston, VA, USA: ASCE; 2008. p. 742-749. https://doi.org/10.1061/41003(327)72
[7] Al-Beiruty M. Collapse potential determination of gypseous soils [MSc thesis]. Baghdad, Iraq: University of Technology; 2003.
[8] Zedan AJ, Hummadi RA, Hussein SA. Effect of Adding Mixture of (Concrete Waste and Asphalt Waste) on the Properties of Gypseous Soil. Tikrit Journal of Engineering Sciences. 2019;26(1):20-25. https://doi.org/10.25130/tjes.26.1.03
[9] Al-Obaidi MT, Al-Mukhtar MT, Al-Dikhil OM, Hannona SQ. Comparative study between silica fume and nano silica fume in improving the shear strength and collapsibility of highly gypseous soil. Tikrit J Eng Sci. 2020;27(1):72-78. https://doi.org/10.25130/tjes.27.1.10
[10] Theyab F, Muhauwiss FM, Alabdraba WM. Enhancing gypseous soil behavior using casein from milk wastes. J Mech Behav Mater. 2022;31(1):306-313. https://doi.org/10.1515/jmbm-2022-0041

[11] Emad R, Salman AD. Shear strength and collapsibility of gypseous soil improved by nanomaterials. IOP Conf Ser Earth Environ Sci. 2024;1374(1):012004. https://doi.org/10.1088/1755-1315/1374/1/012004
[12] Bağrıaçık B, Mahmutluoğlu B, Ürünveren A. A comprehensive study on the assessment of CaCO3-, Nano-CaCO3-, and glass fiber chopped strand (GFCS)-treated clay in terms of bearing capacity and settlement enhancements. Applied Sciences. 2025;15(14):7779. https://doi.org/10.3390/app15147779
[13] Mohammadi M, Rajabi AM, Khodaparast M. Experimental and numerical evaluation of the effect of nano calcium carbonate on geotechnical properties of clayey sand soil. KSCE Journal of Civil Engineering. 2022;26(1):35-46. https://doi.org/10.1007/s12205-021-1914-8
[14] Muhauwiss FM, Hussein AH, Al-Sumaiday HG. Behavior of gypseous soil treated by Arabic gum biopolymer under cycles of wetting and drying. Tikrit J Eng Sci. 2025;32(3):1-9. https://doi.org/10.25130/tjes.32.3.10
[15] Jassam MG, Abd AH, Al-Sumaiday HG. Collapsibility and strength of gypseous soil waterproofed by silane coupling agent. Res Eng Struct Mater. 2026;12(3):1174-1190. https://doi.org/10.17515/resm2026-1640ma0426rs
[16] Pandian NS. Fly ash characterization with reference to geotechnical applications. J Indian Inst Sci. 2004;84(6):189-216.
[17] Kumar P, Sharma RS. Effect of fly ash on engineering properties of expansive soils. J Geotech Geoenviron Eng. 2004;130(7):764-767. https://doi.org/10.1061/(ASCE)1090-0241(2004)130:7(764)
[18] Edil TB, Acosta HA, Benson CH. Stabilizing soft fine-grained soils with fly ash. Journal of Materials in Civil Engineering. 2006;18(2):283-294. https://doi.org/10.1061/(ASCE)0899-1561(2006)18:2(283)
[19] Ehwailat KIA, Ismail MAM, Ezreig AMA. Ettringite formation and stabilization methods of sulfate-bearing soil: A state-of-the-art review. Indian Geotechnical Journal. 2022;52:927-941. https://doi.org/10.1007/s40098-022-00619-x
[20] ASTM D6913/D6913M-17. Standard test methods for particle-size distribution (gradation) of soils using sieve analysis. West Conshohocken, PA, USA: ASTM International; 2017.
[21] Barzanji F. Gypsiferous soils of Iraq [PhD thesis]. Ghent, Belgium: Ghent University; 1973.
[22] ASTM D854-14. Standard test methods for specific gravity of soil solids by water pycnometer. West Conshohocken, PA, USA: ASTM International; 2014.
[23] ASTM D4318-17e1. Standard test methods for liquid limit, plastic limit, and plasticity index of soils. West Conshohocken, PA, USA: ASTM International; 2017.
[24] ASTM D1556/D1556M-15e1. Standard test method for density and unit weight of soil in place by sand-cone method. West Conshohocken, PA, USA: ASTM International; 2015.
[25] ASTM D1557-12e1. Standard test methods for laboratory compaction characteristics of soil using modified effort (56,000 ft-lbf/ft^3 (2,700 kN-m/m^3)). West Conshohocken, PA, USA: ASTM International; 2012.
[26] Thomas MDA. Optimizing the use of fly ash in concrete. Skokie (IL): Portland Cement Association; 2007. 1-24.
[27] Yu QL. Application of nanomaterials in alkali-activated materials. In: Nanotechnology in eco-efficient construction. Cambridge, UK: Woodhead Publishing; 2019. p. 97-121. https://doi.org/10.1016/B978-0-08-102641-0.00005-0
[28] Changizi F, Haddad A. Application of nano materials in improving geotechnical properties of soils: a review study. J Rehabil Civ Eng. 2023;11(4):138-158.
[29] ASTM D2216-19. Standard test methods for laboratory determination of water (moisture) content of soil and rock by mass. West Conshohocken, PA, USA: ASTM International; 2019.
[30] Karim, H.H., Schanz, T. and Ibrahim, A.N. Microstructural characterization of Iraqi gypseous soils. In: Proceedings of the 2nd International Conference of Buildings, Construction and Environmental Engineering (BCEE2), American University of Beirut, October 2015, vol. 17, pp. 17-18.
[31] ASTM D3080/D3080M-11. Standard test method for direct shear test of soils under consolidated drained conditions. West Conshohocken, PA, USA: ASTM International; 2011.