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Investigating the effect of pozzolanic activation on some of geotechnical properties of soil stabilized with calcium based waste materials

Hassnen Jafer[1*], Abeer Abduljabbar Al-Saeedi[2], Wathiq Al-Jabban[3]

[1] Department of Civil Engineering, College of Engineering, University of Babylon, Babylon, Iraq
[2] Collage of Material's engineering, University of Babylon, Babylon, Iraq
[3] Environmental engineering department, College of Engineering, University of Babylon, Babylon, Iraq
* Corresponding author's e-mail: eng.hassnen.mosa@uobabylon.edu.iq

ABSTRACT

This research is aimed to use two different waste materials; calcium carbide residue (CCR) and alum sludge ash (ASA) for the stabilization of a fine-grained soil. Different dosages of CCR were used (3, 6, and 9% by the soil dry mass) and mixed with varying percentages of ASA (1.5, 3, 4.5, and 6%) at different proportion to produce 13 mixtures of soil-binder along with control mixture; untreated virgin soil. The Atterberg limits, standard compaction and unconfined compressive strength (UCS) tests were utilized to evaluate the geotechnical properties. The specimens subjected to the UCS test were cured at different periods; 7, 28, and 56 days. Additionally, scanning electron microscopy (SEM) testing was conducting for microstructural investigation. Experimental results revealed significant improvement in Atterberg limits particularly after the inclusion of ASA in the used binder, which recorded 50% reduction of plasticity index. UCS increased for both soils treated with CCR and with binary binders, recording the highest value for the binder comprising 9% and 4.5% by the soil dry mass of CCR and ASA, respectively, which showed strength about 10 times of that for the untreated soil. SEM testing revealed considerable developments in microstructure of the selected binder through the age of curing confirming the gradual production of cementitious gel, such as C-S-H. hence, the more dense and coherent structure contributes to strength development of the treated soil.

Keywords: soil stabilization, waste materials, soil strength, calcium carbide residue, alum sludge ash.

INTRODUCTION

Post-construction issues in soft clays, including excessive settling, swelling, dispersivity, and erodibility, represent significant geotechnical challenges linked to these soil types. Soft clays are characterized by low strength, high compressibility, and sensitivity, which results in a reduction in strength upon disturbance [1]. Cement is utilized as an addition to stabilize soft clays, based on the premise that the base exchange and cementing properties of Portland cement with clay resemble those of lime [2]. Lime is produced with cement through the hydration process, suggesting that a key point of soil-cement interaction may be investigated by testing clay-lime reactions. Soil consolidation is a process of improving soil properties through a variety of treatments, such as compaction, addition, or chemical combination with additives, to achieve higher strength, durability, and other benefits. Soil stabilizers are generally divided into classic and nonconventional types; among the classic stabilizers, fly ash, lime, and cement are usually considered conventional stabilizers, while sulfonated oils and bitumen are considered unconventional stabilizers [1]. There are significant differences in soil-stabilizer reactions and in their actual behaviour after stabilization, which can be attributed to differences in soil chemical composition, variations in microstructure and macrostructure, and discrepancies in geologic deposits and chemical and physical interactions of the soil-stabilizer mixture [1]. Cement/lime is the stabilizer of choice for geotechnical engineers when dealing with troublesome soft soils, because it is readily available, easy to mix, relatively inexpensive, and highly improvable.

The cement industry is identified as a significant contributor to global greenhouse gases. Indeed, up to 8% of these carbon dioxide (CO_2) emissions are attributable to Portland cement (PC) production, leading to adverse environmental impacts [3]. PC production generally entails the amalgamation and thermal processing of precise ratios of iron ore, clay, silica sand, and limestone. It is predicted that approximately 1.5 tons of natural limestone and up to 0.3 tons of additional raw materials are required to manufacture 1 ton of PC [4]. A significant quantity of energy is required for operations, approximately 4–5 GJ/ton [5]. Notwithstanding the adverse effects of cement manufacturing, its annual output reached 4.1 billion tons in 2020, equating to the formulation of approximately 25–30 billion tons of concrete.

Calcium carbide residue (CCR) represents a by-product of the acetylene production process, mostly composed of calcium portlandite and calcite [6, 7]. The yearly output of calcium carbide residue is around 28 million tons, but its usage rate is just 55% [8]. The subject of interest is to the potential substitution of regular Portland cement with a composite of CCR waste and fly ash as binding agents. This results from the possible interaction between portlandite in CCR and the amorphous silica or alumina in fly ash, leading to the development of reaction products like as calcium silicate hydrate and calcium aluminate hydrate. Multiple studies have examined the individual and synergistic impacts of CCR and FA as supplementary cementitious materials in both concrete and cement uses [9–13]. This research has shown that CCR and FA may significantly enhance mechanical characteristics, long-term performance and durability. Furthermore, the use of CCR and FA may reduce the heat of hydration, hence decreasing the likelihood of thermal cracking in extensive concrete structures. The use of CCR and FA in concrete mixes reduces the heat of hydration, hence reducing the likelihood of thermal cracking in large concrete constructions. Moreover, the integration of these components may enhance resistance of the concrete to chemical assault, including sulfate and chloride penetration, which is essential for the structures subjected to harsh conditions. The objective of this study is to evaluate the efficacy of a binary waste-based binder system comprising Calcium Carbide Residue (CCR) and Alum Sludge Ash (ASA) for pozzolanic activation and stabilization of fine-grained silty clayey soils. The novelty of the work lies in the synergistic utilization of two industrial and urban wastes (CCR rich in calcium oxide and ASA highly enriched in amorphous SiO_2 and Al_2O_3) without any chemical/thermal activation beyond the pre-treatment standard. Although some studies have investigated either CCR or fly ash by itself, this paper is one of the first to study the complementary chemical interaction and microstructural development in the CCR–ASA system through a full range of tests, including Atterberg limits, standard Proctor compaction, UCS testing at several curing ages, and SEM microstructure analysis.

EXPERIMENTAL PART

Utilized materials

Soil

The grain-size distribution curve shown indicates that the soil is a fine-grained material, with a majority of fine particles, containing minimum gravel content (<5%), a minor quantity of sand (15–20%), a higher percentage of silt (40%), and considerable clay (~30%). One can observe a consistent downward trend with a comparatively steep slope in the silt and clay portion of the gradation curve, indicating a poorly graded material with a narrow particle size range. Such a gradation pattern typically correlates with low permeability and high compressibility, suggesting that the soil is likely to exhibit poor drainage characteristics and moderate to high plasticity, particularly under saturated conditions. On the basis of on the unified soil classification system (USCS), and in the absence of Atterberg limit data, the soil can be provisionally classified as CL–ML, indicating a transitional behavior between silty clay and clayey silt. Similarly, under the USDA classification framework, the soil falls within the silty clay or clay loam domain. These characteristics imply limited suitability for load-bearing applications without appropriate ground improvement measures, as the high fines content may adversely affect shear strength and volumetric stability (Figure 1).

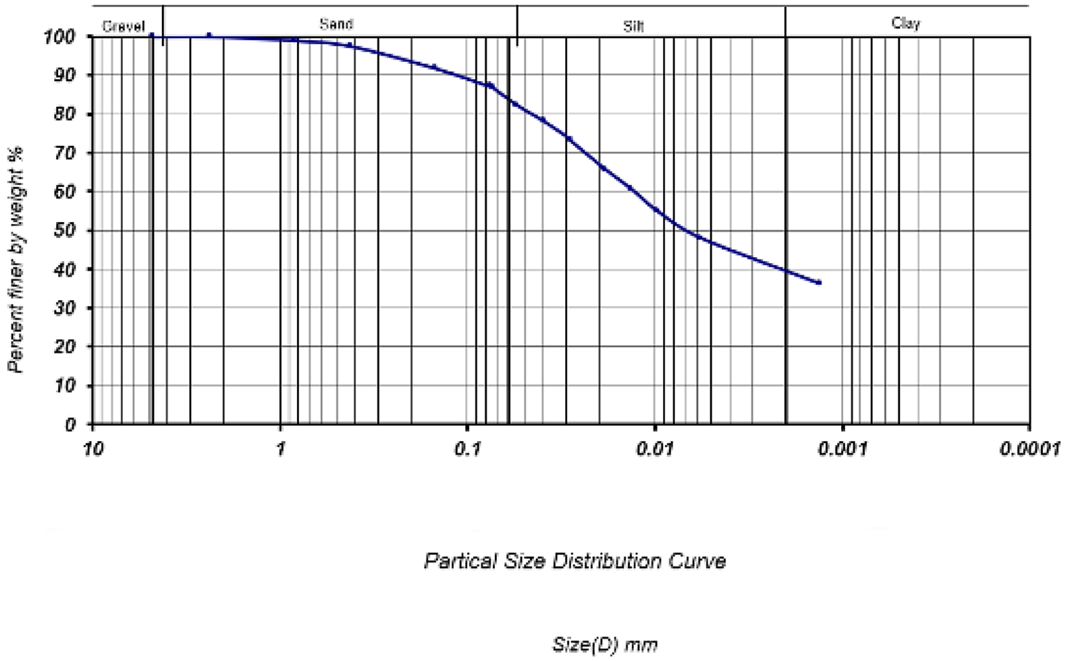


Figure 1. Particle size distribution of the utilized soil

Binder materials

In this study, calcium carbide residue and alum sludge ash were employed as sustainable binder materials to partially replace conventional cementitious components, thereby promoting circular economy practices and reducing the environmental footprint of construction materials. CCR, a by-product generated from acetylene gas production, is predominantly composed of calcium hydroxide, offering high alkalinity and pozzolanic activation potential when combined with siliceous or aluminous materials. On the contrary, ASA is produced by controlled burning of coagulation sludge from a municipal water treatment plant, using aluminum sulfate (alum) as a coagulant. The sludge, which is high in aluminum hydroxides and residual organic matter, is calcined at 450–650 °C to remove carbonaceous material and increase reactivity. The ash is then passed through a 150-mesh sieve (100 µm) to remove unburned material and achieve uniform particle size. This thermal activation converts the amorphous alumina and silica phases into reactive forms that can contribute to pozzolanic reactions.

X-ray fluorescence (XRF) indicated potential for a complementary chemical property between binder materials for pozzolanic reactions. Higher CaO (81.84%) in the calcium carbide residue and a much higher alkalinity (pH 13.10) confirmed its good reactivity as a calcium-rich activator. On the other hand, alum sludge ash contains silica (SiO_2, 46.38%) and alumina (Al2O3, 41.94%) in high amounts, which are necessary to generate pozzolanic reaction products when mixed with CCR. ASA also contains trace amounts of Fe_2O_3, K_2O, and MgO, having a slightly neutral pH level of 7.8. These observations underscore the synergy that CCR and ASA can provide in blended binder systems, with CCR serving as the activation environment for hydration and ASA providing reactive silica and alumina, as outlined in Table 1.

Through the laser diffraction measurements, the continuum of particle size distribution character is obtained, and with a lowest value for D_{50} (17.2 µm), materials being alum sludge ash having also the narrowest grading in between solid residuals, and an opposite situation for maximum values, which are registered by calcium carbide residue (D_{50}: 36.5 µm). Sharp gain in volume from 10 to 60 µm in the cumulative distribution, indicating that where 90% of the material passes under a size of 65 µm (having a larger surface) characterizes improved pozzolanic activity. Aside from CCR, it also shows a greater grading extent of up to 1000 µm, indicating that CSS is not only more uneven but also less reactive. Such a drastic change in particle size distribution is believed to significantly influence microstructural packing and early-age hydration in the binary binder system. This finding indicates the

Table 1. Chemical analysis for binder materials

Oxide content (%)	Value and description	
Chemical composition	CCR	ASA
SiO_2	14.08	46.38
Al_2O_3	0.9	41.94
Fe_2O_3	0.00	4.86
MgO	0.77	0.40
CaO	81.84	0.41
TiO_2	0.12	0.09
Na_2O	1.32	0.09
K_2O	0.20	0.99
So_3	0.77	0.10
pH value	13.10	7.8

capabilities of CCR–ASA mixtures composed of design pozzolanic matrices, specifically well-graded packing and an enhanced reactivity surface area (Figure 2).

As detailed in Table 2, the stabilization matrix comprised fifteen engineered mixes and one untreated control (VS), systematically varying the proportions of calcium carbide residue and alum sludge ash. CCR was incorporated at three levels – 3%, 6%, and 9% – by dry weight of soil, while ASA was added incrementally at 0%, 1.5%, 3%, 4.5%, and 6%, yielding total binder contents ranging from 3% to 15%. The mix IDs were coded accordingly,

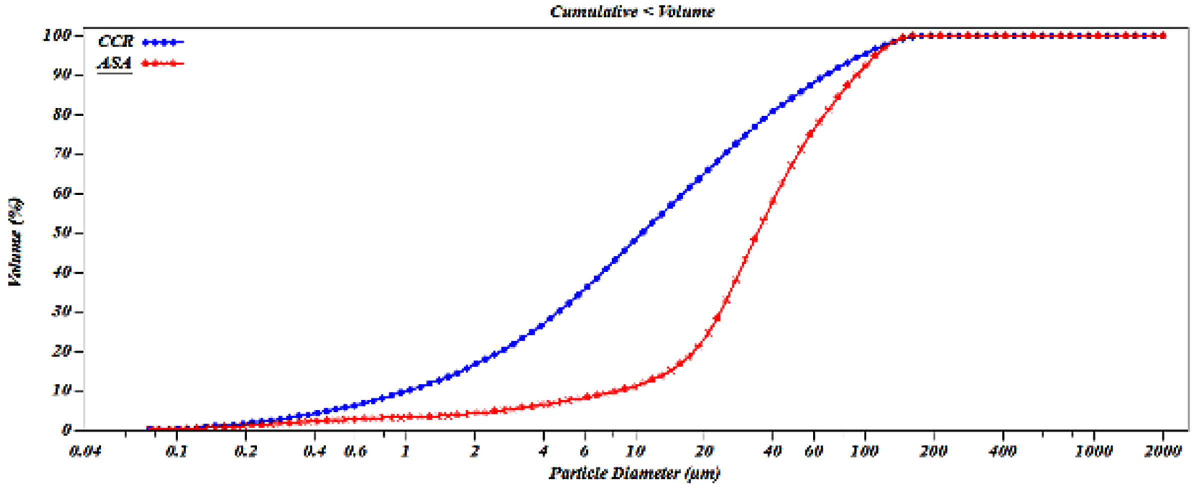


Figure 2. Particle size distribution for the selected binder materials

Table 2. Mixing proportion of supplementary cementitious material

No.	Mix ID	CCR %	ASA %	Total binder %
1	VS	0	0	0
2	CC3	3	0	3
3	CC3A1	3	1.5	4.5
4	CC3A2	3	3	6
5	CC3A3	3	4.5	7.5
6	CC3A4	3	6	9
7	CC6	6	0	6
8	CC6A1	6	1.5	7.5
9	CC6A2	6	3	9
10	CC6A3	6	4.5	10.5
9	CC6A4	6	6	12
10	CC9	9	0	9
11	CC9A1	9	1.5	10.5
12	CC9A2	9	3	12
13	CC9A3	9	4.5	13.5
14	CC9A4	9	6	15

with “CC” denoting CCR percentage and the suffix “A” representing the corresponding ASA dosage (e.g., CC6A3 indicates 6% CCR and 4.5% ASA, totaling 10.5% binder).

Experimental tests

To evaluate the influence of both CCR and ASA on some of the geotechnical and microstructural properties of the treated soil in this study, the following laboratory tests were conducted.

Atterberg limit test

In this test, liquid limit (LL), plastic limit (PL), and plasticity index (PI) tests were performed and calculated in accordance with ASTM D4318 [14]. these tests were utilized to assess the effect of added binders on the consistency of the prepared soil-binder mixtures

Compaction test

This test was conducted in order to obtain the compaction parameters for all soil mixtures represented by the maximum dry density (MDD) and optimum water content (OWC). the test procedure was in accordance with ASTM D698 [15].

Unconfined compressive strength (UCS) test

The UCS test was conducted using an electrical motorized machine to apply an incremental load on a cylindrical specimens (38 mm in diameter and 76 mm in hight) according to the standard ASTM D2166 [16]. apart from the untreated specimens, all other specimens were subjected to 7, 28, and 56 days of curing before they were tested.

Scanning electron microscopy (SEM) analysis

Scanning Electron Microscope (SEM) analysis standard imaging protocols were used for SEM for microstructure inspection. The SEM analysis was performed only on the identified samples (CC9 and CC9A3) at 7, 28, and 56 days of curing to investigate the morphology and distribution of hydration products. Gold coating of the samples was performed prior to imaging to prevent poor conductivity and inconspicuous images in a high-vacuum environment.

RESULTS AND DISCUSSION

Atterberg limits test

The Atterberg limits of stabilized soil mixtures incorporating varying percentages of calcium carbide residue and activated silica alumina reveal a consistent improvement in soil plasticity characteristics with increasing binder content. Across all CCR dosages (3%, 6%, and 9%), the addition of ASA resulted in a progressive increase in the plastic limit (PL) and a notable decrease in the plasticity index (PI), indicating enhanced soil stability and reduced moisture sensitivity. Although the liquid limit (LL) exhibited minimal fluctuations – ranging from 42% to 48% – the significant reduction in PI, particularly at higher ASA dosages, reflects the beneficial effects of

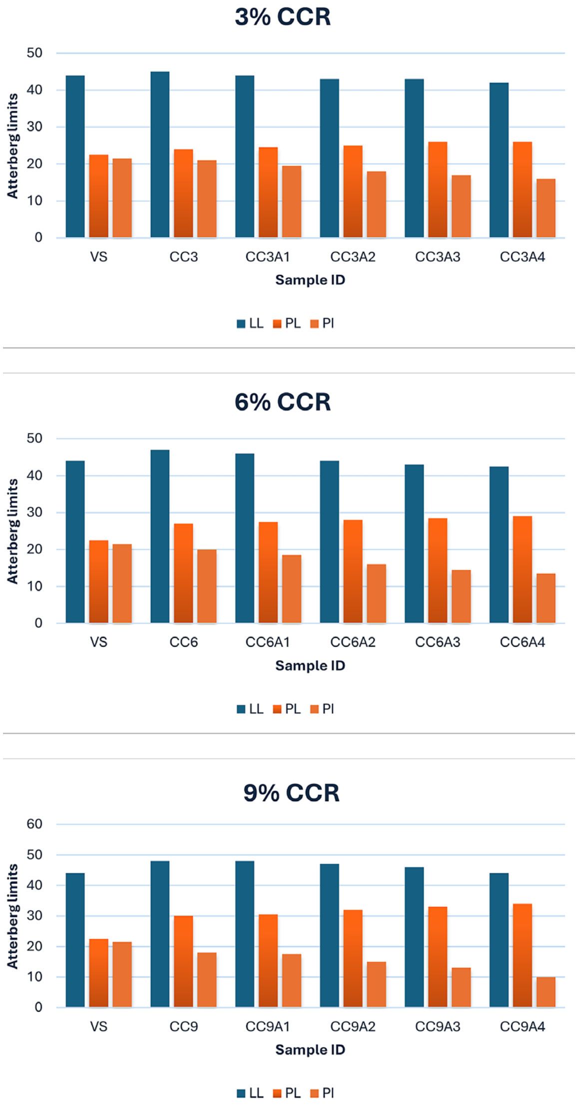


Figure 3. Atterberg limits tests results for calcium carbide residue ratios: (a) 3%, (b) 6%, (c) 9%

pozzolanic reactions in consuming free water and altering clay minerals". The best performance was observed in the CC9A4 mix (9% CCR + 6% ASA), in which PI decreased to 10, demonstrating a significant improvement in workability and shrinkage resistance.

The remarkable decrease in PI noticed with the increase of ASA content on CCR-stabilized soil can be account to synergistic pozzolanic reactions between the high calcium content of CCR and the rich silica (SiO_2 = 46.38%) and alumina (Al_2O_3 = 41.94%) contents found on ASA, which could act as an active alumina-silicate material for geotechnical applications. The chemical analysis evidence that CCR is mainly composed of calcium oxide (CaO = 81.84%) with a very high pH of approximately 13.10 is expected to promote rapid pozzolanic activity, thereby bringing about this positive transformation. When ASA is blended with ASA that can be considered an extremely reactive aluminosilicate source, the dissolved calcium ions from CCR interact with the siliceous and aluminous phases in ASA to produce secondary cementitious compounds such as calcium silicate hydrate (C–S–H) and calcium aluminate hydrate (C–A–H), proposed in [17, 18]. These hydration products fill the voids in the soil and coat the clay particles, decreasing the double-layer thickness and reducing water adsorption, resulting in less plasticity and greater consistency. Furthermore, the absence of Fe_2O_3 in CCR eliminates the potential interference with hydration reactions, allowing more efficient consumption of free lime [19]. Therefore, the enhanced plastic behavior in mixes such as CC9A4 (PI = 10) is a direct result of optimized geopolymeric interaction between the highly basic CCR and the reactive ASA matrix, substantiating their application as sustainable binders in geotechnical soil improvement (Figure 3).

Standard compaction test

The results of the Standard proctor compaction test presented in Table 3 and Figure 4 demonstrate the effect of varying CCR and ASA contents on the maximum dry density (MDD) and optimum water content (OWC) of treated soil mixtures. The reference untreated soil (VS) exhibited an MDD of 1.74 Mg/m³ and an OWC of 15.6%, which served as the baseline for comparison. Upon the addition of 3% CCR (CC3), a slight decrease in MDD to 1.72 Mg/m³ and an increase in OWC to 16.2% were observed, attributed to the flocculation and agglomeration of clay particles induced by the high calcium content in CCR. As the rate of ASA varied from 1.5 to 6% (CC3A1–CC3A4), the MDD decreased from 1.71 Mg/m³ to 1.66 Mg/m³, and the OWC increased from 16.8% to 18%. This is in line with improvements in the fine reaction product and a decrease in particle packing density, requiring higher moisture for

Table 3. Standard compaction test results

No.	Mix ID	CCR %	ASA %	MDD	OWC
1	VS	0	0	1.74	15.6
2	CC3	3	0	1.72	16.2
3	CC3A1	3	1.5	1.71	16.8
4	CC3A2	3	3	1.69	17.2
5	CC3A3	3	4.5	1.67	17.5
6	CC3A4	3	6	1.66	18
7	CC6	6	0	1.7	17
8	CC6A1	6	1.5	1.69	17.5
9	CC6A2	6	3	1.66	18
10	CC6A3	6	4.5	1.64	18.6
9	CC6A4	6	6	1.62	19.2
10	CC9	9	0	1.68	18
11	CC9A1	9	1.5	1.66	18.5
12	CC9A2	9	3	1.64	19
13	CC9A3	9	4.5	1.6	20
14	CC9A4	9	6	1.59	21

compacting at best. A similar behavior was recorded for mixes containing 6% CCR. MDD decreased from 1.70 Mg/m^3 (CC6) to 1.62 Mg/m^3 (CC6A4), the OWC increased from 17% to 19.2%. The drop in MDD reflects the microstructural transformation due to pozzolanic reactions between the CaO-rich CCR (81.84%) and the highly siliceous-aluminous ASA (SiO_2 = 46.38%, Al_2O_3 = 41.94%), resulting in increased void ratio and formation of lightweight gel phases such as C–S–H and C–A–H.

In the case of 9% CCR, MDD reduced from 1.68 Mg/m^3 (CC9) to 1.59 Mg/m^3 (CC9A4) with increasing ASA, while OWC increased notably from 18% to 21%. This consistent decline in MDD and rise in OWC across all CCR dosages with ASA incorporation can be ascribed to the increased demand for water to facilitate the pozzolanic reaction and the formation of secondary cementitious compounds, which create a more porous and flocculated soil matrix [20,21]. The variations in calcium carbide residue and activated silica alumina contents exert a significant influence on both the maximum dry density and optimum water content of treated soil, as illustrated in Figure 4. At a constant ASA dosage, MDD decreased incrementally as CCR content was increased from 0% to 9% with a corresponding upward trend of OMC attributed to relatively high specific gravity and chiefly CaO (81.84%), extensive at this particle size, aggregates that affect and enhance flocculation, which caused an increase in the pozzolanic activity. Likewise, with the increase in ASA dosage for each CCR level, the MDD continued to decrease and OWC to increase because of high SiO2 (46.38%) and A12O3 (41.94%) contents present in ASA which enables the generation of secondary cementitious compounds like calcium silicate hydrate (C–S–H) calcium aluminate hydrate (C–A–H). These reaction products elevate the void ratio and water requirement, reduce compacted

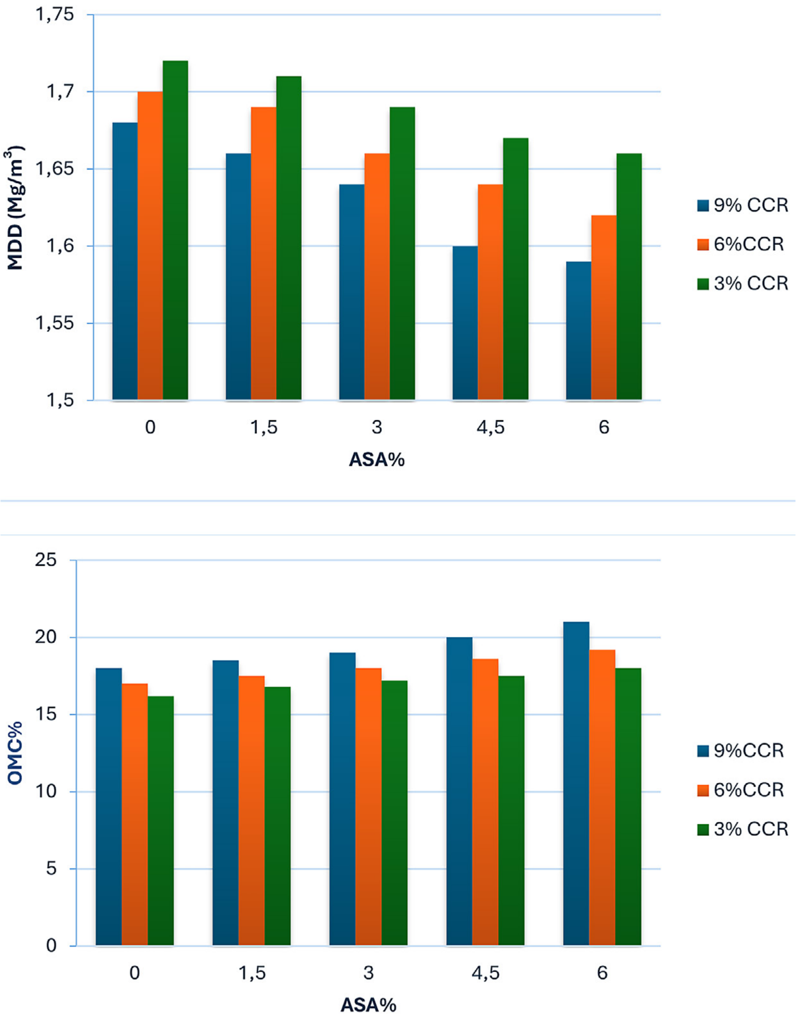


Figure 4. The variation in the a) MDD; b) OWC based on changing ASA%

density, and increase the quantity of moisture required for successful compaction work [20, 22]. Furthermore, the high alkalinity of CCR (pH = 13.1) promotes the dissolution of ASA in particle form and increases the reaction rate, thereby increasing OWC [23].

Unconfined compressive strength (UCS)

The variation in UCS with curing periods of 7, 28, and 56 days is shown in Figure 5, which highlights the significant roles of CCR and ASA in improving the strength of stabilized soils

Figure 5. The variation in UCS results at 7, 28 and 56 curing ages based on CCR: (a) 3%, (b) 6%, (c) 9%

through pozzolanic activity and microstructural densification. The untreated soil (VS) showed a minor increase in strength at all curing ages (136 kPa), whereas the incorporation of CCR alone (e.g., CC3, CC6, and CC9) resulted in a marked increase in UCS, with further enhancement observed upon the addition of ASA. The highest UCS was recorded in the CC9A3 and CC9A4 mixes, achieving 1112 kPa and 1039 kPa at 56 days, respectively, reflecting an over 8-fold improvement compared to untreated soil. This strength development is attributed to the synergistic pozzolanic reaction between the high CaO content in CCR (81.84%) and the highly reactive SiO_2 (46.38%) and Al_2O_3 (41.94%) in ASA, which promote the formation of calcium silicate hydrate (C–S–H) and calcium aluminate hydrate (C–A–H) gels that fill pore spaces and enhance interparticle bonding. According to [24], the strength gain in CCR-based systems is driven by increased formation of C–S–H due to the abundant availability of calcium, especially in the presence of external silica-rich additives. Moreover, the continuous increase in UCS with curing time supports the progressive hydration and pozzolanic reactions, consistent with the findings by [25], who noted that extended curing enhances strength due to ongoing gel formation and pore improvement in chemically stabilized soils [26].

Scanning electron microscopy (SEM) analysis

Figure 6 shows low-magnification SEM micrographs of the primary powder materials used in this study: (a) calcium carbide residue and (b) alum sludge ash. These micrographs are essential in revealing the physical morphology and surface characteristics that influence their reactivity as well as behavior in soil stabilization applications.

In Figure (a), the CCR particles exhibit an angular, blocky, and highly irregular texture, with micro-cracks and porous surfaces. This morphology is typical of the CCR powders generated as a by-product of acetylene gas production, consisting predominantly of calcium hydroxide and having a very high CaO content (81.84%) and alkaline pH (~13.1). Such structures favor the absorption and release of water and the distribution of Ca during hydration, which contribute to CCR being a better activator of the pozzolanic reaction. The microstructure promotes the early development of portlandite and ettringite [27, 28]. They also noted that the reactive shape of CCR is an important factor in the different hydration types observed in binders in blended systems.

In Figure (b), the ASA powder particles – produced by the controlled combustion of alum-based water treatment sludge – display a heterogeneous and flaky morphology, consisting of irregular plate-like particles and partially porous agglomerates. This morphology results from the incomplete burnout of organic matter and the inherent flocculated structure of the original sludge. Chemically, ASA is rich in SiO_2 (46.38%) and Al_2O_3 (41.94%), both of which are critical oxides for pozzolanic activity. Despite not being chemically or thermally activated, the fine particle size and amorphous character of ASA powder still enable it to interact effectively with the calcium ions released by CCR, forming secondary cementitious gels, such as C–S–H and C–A–H. Similar observations were reported by [29, 30].

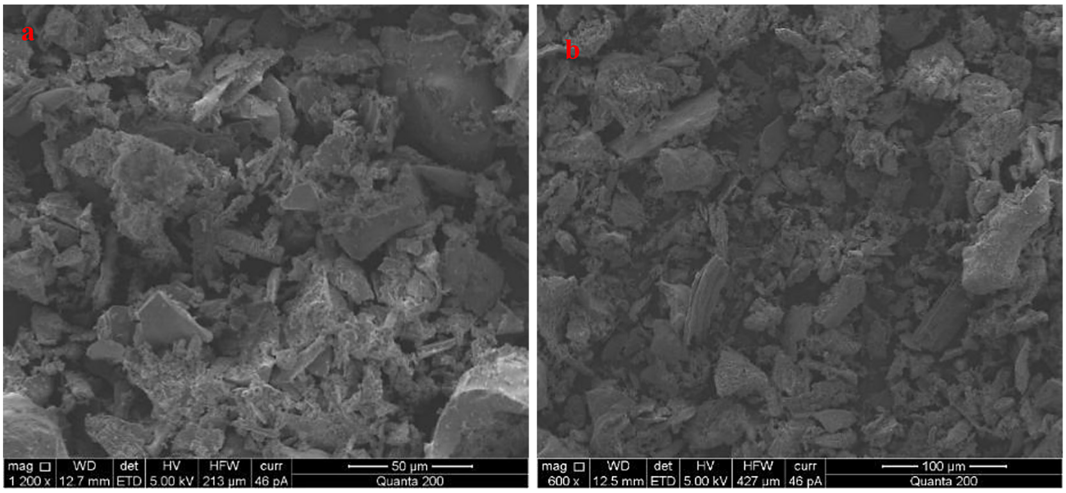
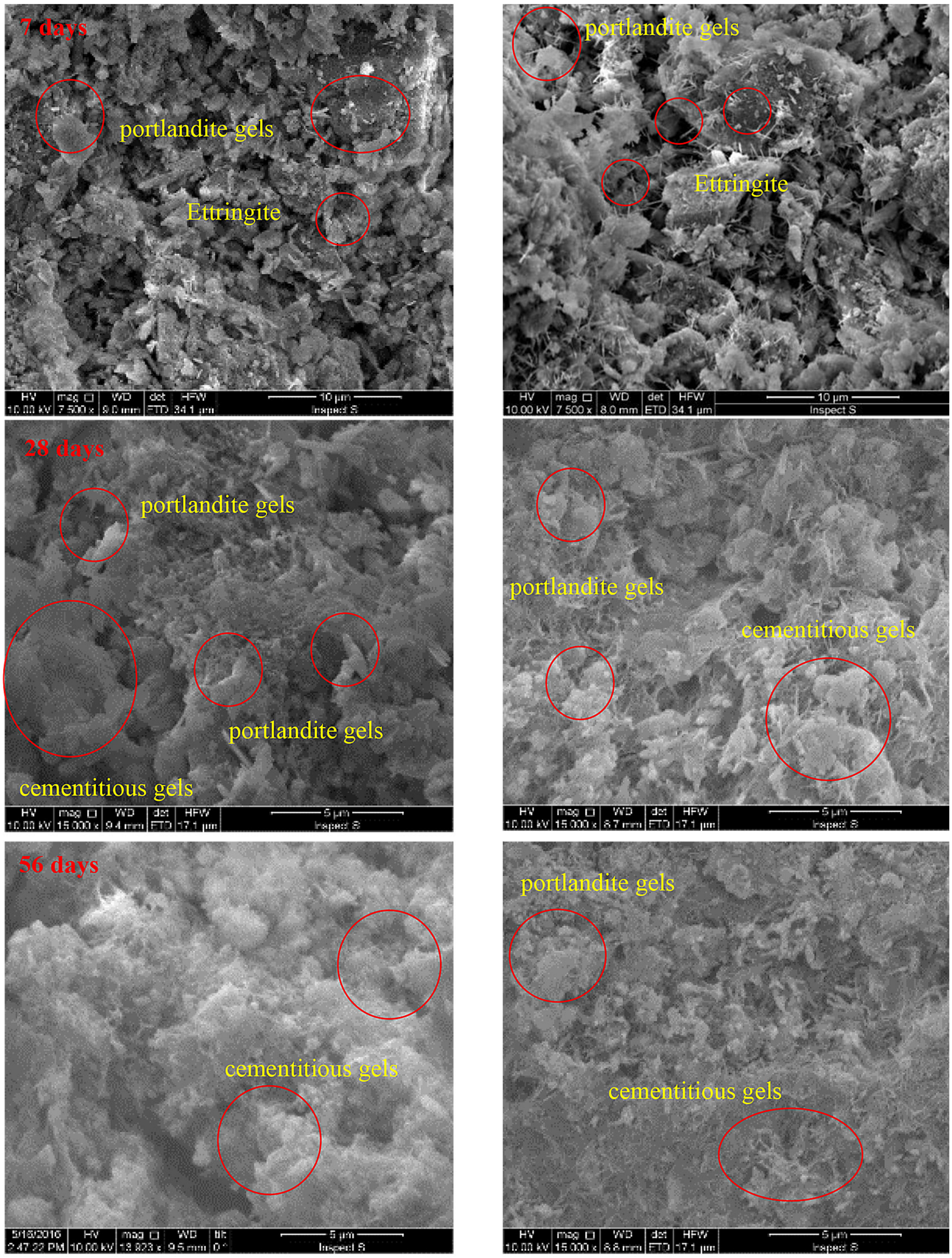


Figure 6. SEM image for (a) calcium carbide residue and (b) alum sludge ash

Figure 7 presents scanning electron microscopy micrographs of stabilized soil pastes – (a) CC9 and (b) CC9A3 – examined at different curing intervals: 7, 28, and 56 days, highlighting the microstructural evolution associated with pozzolanic activity. The left column (a) corresponds to the CC9 paste (9% CCR only), while the right column (b) represents CC9A3 paste (9% CCR + 4.5% ASA), identified as the most promising mix. At 7 days, both samples exhibit the needle-like structures characteristic of ettringite and scattered portlandite gels, reflecting the early-stage hydration of calcium oxide (CaO) from CCR and its partial reaction with reactive alumina from soil



(a) (b)

Figure 7. SEM analysis of most promising binders after 7, 28, and 56 days of curing: a) CC9 paste, b) CC9A3 paste

minerals and ASA. However, the CC9A3 paste shows more extensive formation of fibrous cementitious gels, suggesting enhanced pozzolanic reactivity due to the presence of ASA, which contains high amounts of SiO_2 (46.38%) and Al_2O_3 (41.94%), facilitating early binding reactions.

At 28 days, both systems show a more developed and denser microstructure, with the CC9A3 matrix appearing more compact and less porous, dominated by the accumulation of cementitious C–S–H and C–A–H gels, alongside reduced visibility of crystalline ettringite and portlandite particles. The transformation of portlandite into amorphous hydration products is more advanced in CC9A3, indicating a more complete pozzolanic reaction [31].

By 56 days, the microstructure of CC9A3 continues to demonstrate superior gel coverage, with uniformly distributed cementitious gels filling interparticle voids and embedding remaining portlandite phases. In contrast, the CC9 paste still retains visible portlandite crystals and a less cohesive matrix. The ongoing gel development in CC9A3 results from the extended reaction between $Ca(OH)_2$ (from CCR hydration) as well as the amorphous silica and alumina in ASA, promoting long-term strength gain [24].

CONCLUSIONS

This study has demonstrated the effective utilization of calcium carbide residue and alum sludge ash as sustainable binders for the stabilization of silty clay soils, with particular emphasis on their influence on plasticity behavior, compaction characteristics, strength development, and microstructural evolution. The results of the Atterberg limits tests revealed a significant reduction in plasticity index with increasing ASA content, attributed to the synergistic pozzolanic reaction between the CCR-derived calcium hydroxide and the reactive silica as well as alumina in ASA, resulting in the formation of cementitious C–S–H and C–A–H gels. Compaction testing showed a consistent decrease in maximum dry density and an increase in optimum moisture content with rising binder content, especially at higher CCR and ASA levels, owing to the increased demand for water during hydration and the development of a more porous, flocculated matrix. The unconfined compressive strength of the stabilized soil increased progressively with curing time, reaching a peak value of 1112 kPa at 56 days for the CC9A3 mix, which contains 9% CCR and 4.5% ASA. This strength enhancement was confirmed by SEM analysis, which showed a denser matrix and greater gel coverage over time, indicating continuous pozzolanic activity and microstructural improvement.

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