Microstructural Analysis of Stabilized Rammed Earth Using XRD And SEM Methods

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Abstract
The microstructure analysis of stabilized rammed earth (SRE) is crucial for understanding its mechanical properties, durability, and long-term performance. This literature review paper investigates the application of X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) in analyzing the microstructural characteristics of SRE. XRD is employed to identify the crystalline phases present in the material, providing insights into the mineralogical composition and the effects of stabilization agents. SEM offers high-resolution images that reveal the surface morphology, particle distribution, and bonding mechanisms within the stabilized matrix. By synthesizing recent studies, this review highlights the advancements in microstructural analysis techniques and their implications for the optimization of SRE in construction.

The findings from XRD and SEM analyses are integrated to present a comprehensive understanding of the microstructure-property relationships in SRE. The literature suggests that stabilization methods, such as the addition of cement, lime, or other additives, significantly influence the crystallinity, particle interlocking, and pore structure of the rammed earth. This review discusses the correlation between microstructural features observed through XRD and SEM and the resulting mechanical properties, such as compressive strength and durability. The paper concludes by identifying gaps in the current research and proposing directions for future studies, emphasizing the need for standardized testing protocols and the exploration of novel stabilization materials to enhance the performance of SRE.
Keywords: X-Ray Diffraction, Scanning Electron Microscopy, Energy Dispersive X-Ray Analysis

Introduction

Rammed earth (RE) is an ancient construction technique that has recently gained renewed interest due to its environmental benefits, such as low carbon footprint and use of locally available materials. Traditional RE involves compacting layers of damp earth within formwork to create solid structural walls. To enhance the mechanical properties and durability of RE, stabilizers are often added, resulting in stabilized rammed earth (SRE). This study focuses on the microstructure analysis of SRE using different soil types—red soil, laterite soil, and manufactured sand (M-sand)—stabilized with specific additives and examined using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) methods.

In this investigation, the SRE mixtures are stabilized with 9% cement, 2% Bagasse Ash (BA) , and 0.4% Glass Fibers (with a length of 12mm). A dry density of 1.9 g/cc is consistently maintained across all samples. The samples are prepared using unconfined compressive strength (UCS) molds to cast cylinders, ensuring uniformity in testing and analysis. XRD is utilized to identify and quantify the crystalline phases present in the stabilized mixtures, providing insights into the mineralogical composition and the transformations induced by the stabilizers. This technique helps in identifying key compounds such as calcium silicate hydrates (CSH), which are crucial for enhancing the mechanical properties and durability of SRE.

SEM analysis offers detailed imagery of the microstructure, revealing surface morphology, particle size distribution, pore structure, and the nature of bonding between particles. These images provide critical insights into how the stabilizers—cement, bagasse ash, and glass fibers—affect the microstructural properties. The addition of bagasse ash, a pozzolanic material, and glass fibers is intended to improve the bonding and reduce porosity, thereby enhancing compressive strength, tensile strength, and overall durability.

By synthesizing findings from various studies, this literature review aims to elucidate the relationships between microstructural characteristics and macroscopic properties of SRE made from different soil types. The review discusses how stabilization techniques alter microstructural features such as porosity, particle interlocking, and the formation of binding compounds. It also highlights the importance of standardized testing protocols to ensure consistent and reliable results. Furthermore, the review explores the potential of novel stabilization materials, emphasizing their impact on the sustainable development and performance optimization of SRE in modern construction.

Literature Review

Soil Properties

The soil used for stabilized rammed earth should mainly consist of sand and fine gravel, with enoughclay to provide cohesive strength and a percentage of silt to act as a void filler. The clay content should be between 20% and 35%, while sand should be between 50% and 75%[1]. soil used for construction was classified as fine to coarse sand (SW) based on the Unified Soil ClassificationSystem. The particle size distribution was determined according to the Spanish Standard, showing aslightly excessive portion of gravel and a lack of silt and clay[2]. The proportions of the fractions play a crucial role in determining the performance and characteristics of the rammed earth material.[3] The distribution affects the porosity and permeability of the soil. Higher clay content typically results in higher water adsorption capacity due to the smaller particle size and increased surface area for water retention. On the other hand, sand and gravel content can influence the strengthand stability of the rammed earth material[4]. A balanced particle size distribution is essential for achieving the desired properties in rammed earth construction[5].

The plasticity index (PI) of 8.3 indicates a moderate plasticity range for the soil. Soils with a higherPI tend to exhibit more cohesive and workable properties. The Atterberg limits, including the liquidlimit and plastic limit, provide insights into the workability and consistency of the soil. The values obtained from the test soils help in understanding how the soil will behave during construction processes such as ramming[2]. According to The Australian Earth Building Handbook, when usinglime as stabilizer the ideal soil should have a plasticity index from 20% to 30% and liquid limit between 25 and 50, so lime would be particularly appropriate for stabilization of expansive soils[3] Atterberg limits are consistent at depth greater then 10m, with an average liquid limit (WL) of 47, plastic limit (WP) of 27, and plasticity index (IP) of 20[4]

The moisture content during manufacturing is known to be an important factor for the strength development of RE. Generally, a value close to the optimum moisture content (OMC), which allowsthe maximum dry density of the soil for a certain compaction energy, is chosen[1]. This OMC is determined in most of the studies via Standard or Modified Proctor tests. The Modified Proctor testuses higher compaction energy so the OMC obtained is slightly lower, which, according to some authors would be closer to the compaction effort applied in the construction of a real wall by mechanics means. Because of the presence of gravel, the moisture content of rammed earth is typically lower than other earthen construction techniques such as cob or adobe[2]. It must also be noted that the obtained OMC is lower than usual values for rammed earth. This result can be explained by the relative high portion of gravel and low portion of silt and clay, as illustrated in. As the proportion ofgravel increases, the OMC is expected to decrease, since the specific surface area of the soil grains decrease.[8]

The density of rammed earth depends on the distribution of small particles, humidity during compaction, and pressure magnitude. Keable et al.[9] noted that soil tends to stick to the frame- work if compressed to the optimum limit and thus recommended using a lower humidity than values reported in the literature. The density ranges of modern and ancient rammed earth structures are 1700–2200 kg/m3 and 1770–1990 kg/m3, respectively.[10]

Houben and Avrami [19] stated that water has a critical effect on the decay and adhesion of rammedearth structures. Water behavior within a structure (e.g., absorption, adsorption) is difficult to distinguish and the decay process remains poorly understood. Water leakage is a destructive factor that leads to mold formation. The use of materials such as cement, lime, silica, and acrylic coatingshas been reported to effectively mitigate water leakage because they allow more adhesion between particles [20]. Another aspect is to determine the effects of humidity on the mechanical properties of rammed earth walls. To further address this question, Bui et al. [21]calculated the compressive strength, elastic modulus, and Poisson ratio of rammed earth walls and found a Poisson ratio of 0.2for dry samples and 0.37 for wet samples. Hall and Djerbib[22] also studied the effects of small particle distribution on water absorption and capillaries under pressure and the moisture absorptionof rammed earth samples.

Effect of Stabilizers on Soil Properties

Cement and lime were used as traditional stabilizers to chemically interact with the soil particles andimprove the overall performance of the rammed earth. The stabilizers played a crucial role in enhancing the properties of the soil mixture, making it suitable for construction applications. [22] Stabilizers, particularly cement, can increase the shear strength of the rammed earth material. The main hydrate of cement, CSH gel, has strong adsorption activity that can close the gaps between aggregates and improve the shear strength of the soil.[23]

Testing program has revealed that the best category of soil are those with linear shrinkage (LS) <6.0 and clay and silt content ≤ 20% or with LS < 6.0 and clay and silt content 21 – 35%. These twocategories have shown the highest stabilization success. (B. Steve). The composition of 5-20% gravel, 45-60% sand, 20-35% silt and clay has been recommended for better performance of wall construction. Composition can be checked by performing jar test (C. Jayasinghe and N. Kamaladasa). From the results it is concluded that the Compressive strength of Cement Stabilized Rammed Earth wall panels in cement and soil combination 1:8 are, were for sandy soil compressive strength is 3.06 to 3.99, for gravel compressive Strength is 1.84 to 2.09, and for clay soil compressive strength is 1.98 to 2.03.[24]

Kenneth Mak et al. 2014, in this research paper author have worked on replacement of cement with metal oxide and permazyme presented no development in compressive strength, whereas the additional of cement with resin systems showed an increase in capacity ranging from 52% to 220%. For the analysis of effect they have conducted following tests, like Compressive testsof specimens were completed on a universal testing machine and water absorption by RILEM[22]. Sachin N. Bhavsar et al, 2014, in this research paper authors concluded that the impactof brick dust on black cotton soil is positive. By changing soil by half of its dry weight by brick dustit gives maximum improvement in the engineering assets of black cotton soil. So use of brick dustis preferable for stabilization because it gives positive results as stabilizer and also it is a waste consumption. Effects of burnt brick dust on black cotton soil by replacement with burnt brick dust as a stabilizer by 30%, 40% and 50% proportions respectively [23] By stabilization of black cottonsoil for 30% replacement by marble powder and brick dust the properties related to soil are improved. For marble powder Liquid limit, Plastic limit, Plasticity index values are decreasing.

Microstructure Analysis

The study examines phase evolution in Portland cements with calcium carbonate, comparing in-situand ex-situ XRD techniques for hydration phase analysis. It highlights the impact of cement mineralogy on hydration and properties, with C1 cement showing higher initial strength gain than C2, where C1 and C2 are Commercial samples of two cements, from different suppliers. The cements selected differ not only in the geographic location of manufacture but also in the composition of raw materials, with different contents of major crystallographic phases and sources of gypsum, as shown in Fig.1. The study suggests improved methods for phase quantification in in-situ analysis[25].

X-ray Fluorescence Analysis, g/100 g			Rietveld Quantitative Phase Analysis, g/100 g		
Oxides	*C1*	*C2*	***Phases***	*C1*	*C2*
SiO_2	16.8	16.1	Alite	54.7	40.9
Al_2O_3	4.3	3.9	Belite (β-C_2S)	8.6	19.4
Fe_2O_3	2.9	3.9	Aluminate cub.	5.5	2.8
CaO	60.1	58.8	Ferrite	6.7	10.0
MgO	5.7	5.3	Gypsum	2.9	4.7
K_2O	0.8	0.2	Bassanite	2.1	4.0
SrO	—*	0.8	Calcite	10.4	9.5
SO_3	1.9	2.5	Dolomite	1.5	2.7
TiO_2	0.2	0.5	Periclase	4.7	4.2
P_2O_5	0.2	0.8	Other phases (inert)	3.0	1.8
L.O.I.	6.9	7.1	Density (g/cm^3)	3.061	3.096

* Below the detection limit.
** Particle size characteristic: C1 (D10, 1.83 μm; D50, 11.31 μm; D90, 24.18 μm). C2 (D10, 1.89 μm; D50, 10.72 μm; D90, 23.12 μm).

Fig.1 X-ray fluorescence and Rietveld quantitative phase analyses of the cements. Density determined by gas pycnometry and particle size distribution by laser diffraction.

The studyexplores the transformation of fly ash into C-S-H material for cement, highlighting its impact on cement hydration and properties. The material improves compressive strength, reduces viscosity, and expands solidified cement volume. The process involves thermal activation and hydrothermal reaction to enhance chemical activity. The integration of C-S-H material advances cement utilizationand properties[26]. A new low-carbon cement from alkaline solid waste has been developed with optimized curing methods. The optimal Carbide Slag (CS) : Soda Residue (SR) : Red Mud (RM) : Fly Ash (FA) ratio enhances compressive strength by 147.7%, while high-temperature curing at 80°C boosts early strength by 10.7/22.1 MPa. The material also has a unique hydration product structure, with N-A-S-H and C-(A)-S-H gels as primaryhydration products[27]. Study explores the use of coal gasification slag and fly ash as eco-friendly cementitious materials. It highlights the benefits of ternary systems, such as enhanced workability and mechanical properties, and the potential for reducing CO_2 emissions in construction. The study also highlights the synergistic effect of Coal Gasification Slag (CGS) and Circulating Fluidized Bed Fly Ash (CFBFA) on hydration behavior[28]. It was found that fly ash and metakaolin improved compressive strength, reduced Ca/Si ratio, and promoted gel absorption in the Ordinary Portland cement (OPC)-Metakaolin (MK) system. The study also explored the feasibility of using fly ash to modify the OPC-MK system, examining its effects on cement properties and morphological features[29].

It found that heat treatment increased glass phase content but decreased cementitious activity. The study also examined the effects of heat treatment on Fly Ash properties, including its impact oncementation activity and strength. Results showed that heat-treated FA-OPC showed greater strengththan pure cement[30]. The study investigates the use of silicomanganese slag in eco-friendly hybridcement production, revealing that Portland cement and higher NaOH molarity can improve compressive strength. It also highlights the potential of SiMn slag as pozzolanic materials in construction, emphasizing the need for balance.[31] The paper examines the production of activatedcarbon from sago wastes through acid modification. It reveals differences in surface morphology and crystallinity, with $ZnCl_2$ activation yielding high metal absorption. The study also includes water content and ash content analysis, FTIR, SEM, and XRD analysis for characterization[32]. The study examined the impact of slurry treatment on recycled concrete aggregate properties, finding that prolonged soaking of 72 hours resulted in dominant performance. The study also examined the microstructure and engineering properties of Recycled Concrete Aggregate (RCA) from different parent concretes, with longer treatment duration enhancing hydration products and coating layers. Further research isneeded to optimize slurry treatment for industrial applications [33]. Pervious concrete with Reactive MgO cement (RMC) enhances strength through carbonation, promoting CO_2 diffusion and carbonation. It addresses urban flooding and drainage issues due to its high void ratio. RMC-based pervious concrete shows improved mechanical properties and carbon sequestration. It can act as a CO_2 sink with high void content. The study also shows that RMC-based pervious concrete has lowernet CO_2 emissions due to its low binder content and sequestration capacity[34].

The study explored Friedel's salt formation in concrete and its impact on pozzolans in cement blends.It identified origins and chemical reactions, and examined the role of reactive alumina and silica. The study found that pozzolans have varying pozzolanic activity based on their mineralogical composition, and the formation coincides with the physical state of reactive alumina in pozzolan[35]. The study explores the synthesis of Calcium Ferrite from Fe_2O_3 and $CaCO_3$ using XRD and SEM-EDX analysis. It focuses on the formation of $Ca_2Fe_2O_5$ and $Ca_2Fe_9O_{13}$ phases, which are nanosized particles with magnetic properties. The study also compares Calcium with Barium and Strontium for Ferrite formation. The synthesis of Calcium Ferrite was confirmed through XRD andSEM-EDX analysis. The EDAX results show that the elements making up the phase are Ca, Fe, O, and there is little impurity, Si. The EDAX results confirm or explain the background roughness level from the XRD test results as shown in Fig.1,2,3,4 respectively. [36]. The study uses XRD-Rietveld method to evaluate the leaching resistanceof hardened cement paste. Results show that resistance changes with $CaCO_3$ and $Ca(OH)_2$ content.The addition of silica fume improves compactness, strength, and frost resistance. However, flexuralstrength decreases with age and flow rates. Flow rate has no significant effect on leaching within 90days[37]. A new CO_2-resistant calcium aluminate cement has been developed for well sealing, enhancing compressive strength and reducing porosity, and highlighting the importance of robust cement for CO_2 injection wells[38].

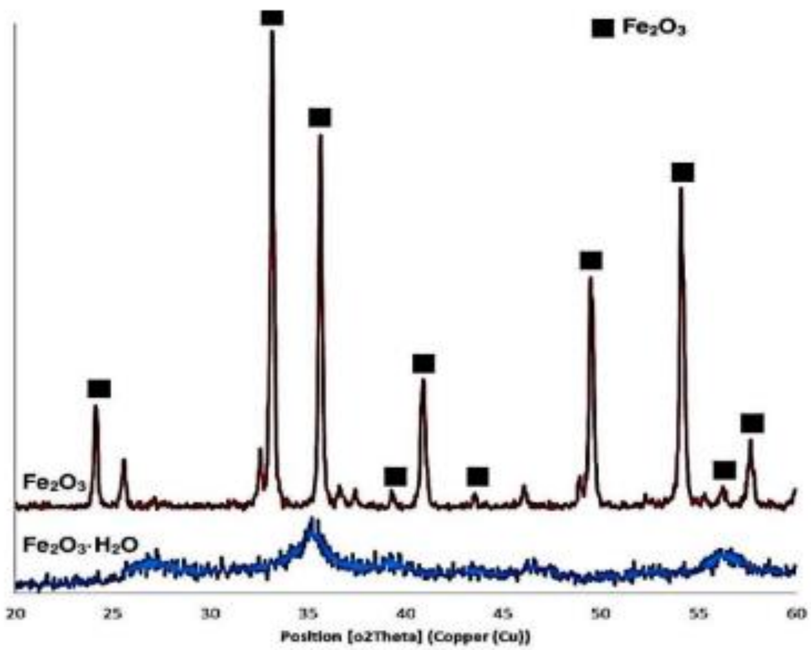

Fig.2 X-ray diffraction pattern of Fe_2O_3 synthesized from iron sand

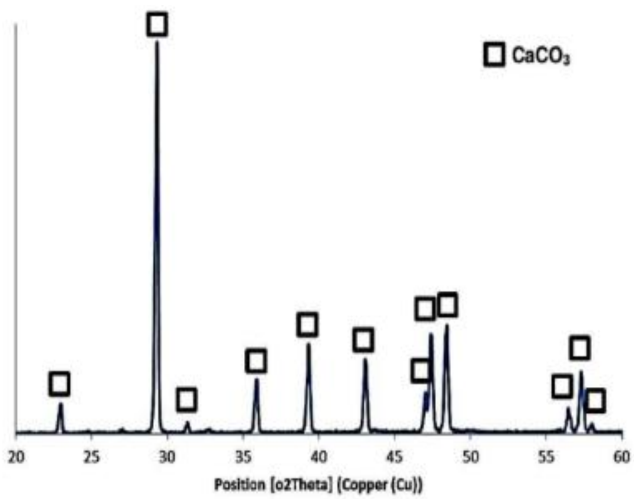

Fig.3 X-ray diffraction pattern of $CaCO_3$ synthesized from limestone

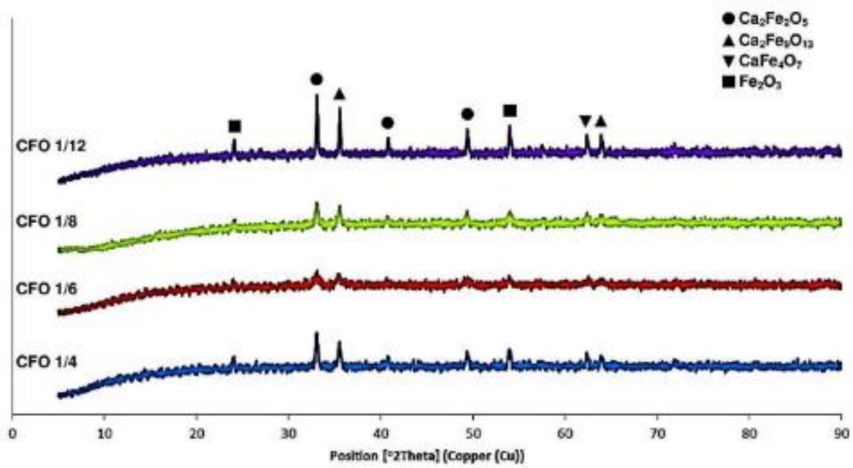

Fig.4 X-ray diffraction pattern of Calcium Ferrite synthesized by coprecipitation method

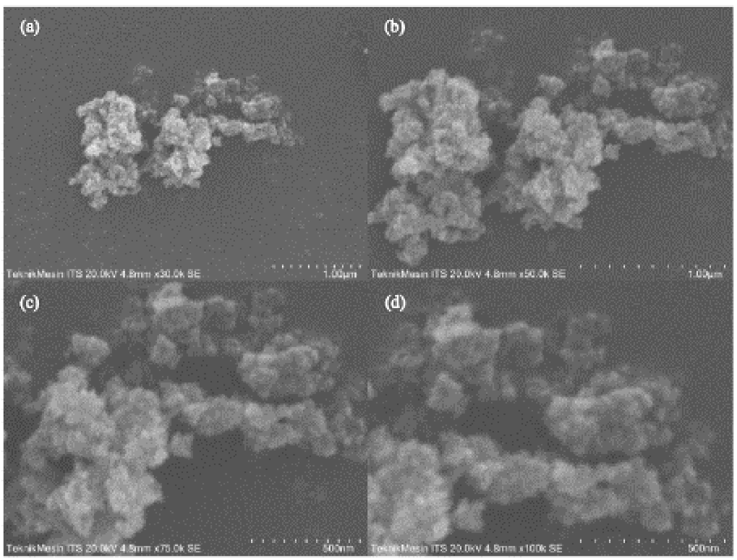

Fig.5 SEM of Calcium Ferrite samples with a ratio of 1/6

Waste Clay Brick Powder (WCBP), a common geopolymer precursor, improves geopolymer mortar properties by promoting geopolymer gel formation. Up to 40% WCBP in geopolymer mortars provides high compressive strengths. Flow reduction is observed with increased WCBP content, but workability remains. WCBP leads to a compact structure, reduced porosity, and improved mechanical properties. Future studies will focus on the long-term durability of geopolymer mixtures, evaluating fly ash alternativesand examining microstructural properties[39]. The study uses nanoindentation technique to analyzethe concrete transition zone with metakaolin. Results show that metakaolin reduces Interfacial Transition Zone (ITZ) thickness, increases CSH frequency, and stabilizes hardness. It also has a discrete influence on concretes with high cement consumption. The technique is valid for analyzing nanomechanical properties of cementitious materials and improves concretes with Portland cement in macro, micro, and nano-structural scope[40]. The study analyzed Portland cement's microstructural hydration with natural seawater using microscopy techniques and Energy Dispersive Spectrometry (EDS) mapping. The researchers developed algorithms forself-correction, denoising, and imaging calibration. EDS mapping provided distribution maps of cement phases, with seawater (SW) samples showing higher hydrate phase content with lower porosity[41].

It examined the mechanical properties of low concentration Phosphoric Acid (PA)-activated Fly Ash (FA) geopolymer for construction materials, comparing it with NaOH-activated geopolymer. It found that PA concentration and Liquid/FA ratio significantly influenced geopolymer formation. The study also examinedthe impact of PA concentration, L/F ratio, and curing temperature on Phosphoric acid-activated fly ash Geopolymer (PAFG's) strength, microstructure, CO_2 emissions, and economic impact. The results showed that PA concentration and L/F ratio significantly influenced FA dealumination and polymerization[42]. The effects of activator type and concentration on SiMn slag pastes, revealing C-S-H as the main reaction product. It concluded that SiMn slag could be used as an alternative binder. The formation of phases depends on activator type, concentration, slag structure, and curing conditions. Mechanical behavior aligns with hydration advancement and activator concentration[43]. Using X-ray Diffraction and SEM-based image analysis, the study looks into the measurement of cement paste in recycled aggregates. The findingsindicate that natural aggregates make up 80% of fine recycled aggregates, with 96% of them being paste-free. The study also looks at the connection between mineral composition and chemical analysis, finding that products with higher densities have more carbonates and less cement paste. Improving the quality of recycled aggregate is the goal of this study[44]. Zeolite-poor rock and sawdust are being explored as eco-friendly alternatives for clay bricks. Sawdust addition enhances thermal insulation, reduces bulk density, increases porosity, and lowers compressive strength. The optimal quantity should be less than 10%. The study shows potential for practical application in eco-friendly construction, with samples showing potential for high-quality composite bricks with 8% sawdust. The addition of sawdust affects water absorption, compressive strength, and thermal insulation[45].

With an emphasis on pore size, shape, hydraulic radius, and roundness, the study uses Backscattered Electron Scanning Electron Microscope (SEM-BSE) toinvestigate the pore morphology in hardened cement paste. It demonstrates a relationship betweenthe ratio of water content to pore size and area. The study also shows that irregular pore shapes and higher total porosity are caused by raising the water content ratio[46]. Through the use of cutting- edge methods including X-ray computed tomography (XCT) and Mercury Intrusion Porosimetry (MIP), the study investigates the microstructure and mechanical characteristics of Graphene Oxide (GO) cement. It demonstrates how GO improves cement paste's macro-and micromechanical qualities. The study also emphasizes how GO nanosheets, which are plentiful in oxygen functional groups, can be used to refine the pore structure and optimize the microstructureof cement paste[47]. Researchers used SEM image analysis and isothermal calorimetry to accuratelydetermine anhydrous cement proportion in materials. The MATLAB program for SEM image analysis showed high precision. Isothermal calorimetry confirmed the reactivity of powder by- products and their effect on hydration. Mechanical properties of standard mortars with by-productswere significantly measured. The proposed method can be used for all by-products with consistent results[48]. Waste shells, such as cockles, oysters, and murex, can be used as fine aggregate substitutes in cement mortars without significant strength loss. These shells are used for environmental benefits and have been studied for their physical, mechanical, and microstructural properties. Further research is needed to explore their effects on compressive strength, durability, and other properties of cement based composites[49].

Summary

This literature review paper examines the microstructure analysis of stabilized rammed earth using X-ray diffraction (XRD) and scanning electron microscopy (SEM) methods. It synthesizes findings from various studies on rammed earth and microstructural investigations of different construction materials, including cement and concrete. Despite extensive research on the microstructure of these materials, a notable gap exists in the analysis of stabilized rammed earth, particularly using XRD and SEM techniques. This review highlights the lack of specific studies focusing on the microstructural characteristics of stabilized rammed earth, which is critical for understanding its performance and durability. By analyzing the microstructure of rammed earth stabilized with red and laterite soils, and using unconfined compressive strength (UCS) cylindrical molds, this paper aims to fill this research gap. The investigation will provide insights into the stabilization mechanisms and contribute to the development of more sustainable and durable construction materials.

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