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Structural evaluation and durability potential of cementitious materials based on expanded clay kiln dust

Keywords: characterization, expanded clay kiln dust (ECKD), mortar, strength, substitution

Introduction

The construction industry heavily relies on cement as a fundamental element, particularly in the production of concrete, the most widely used construction material globally (Akhtar & Sarmah, 2018). However, despite its undeniable benefits in terms of strength and durability, cement production carries significant environmental impacts, primarily attributed to carbon dioxide (CO₂) emissions from limestone calcination. Construction activities contribute to approximately 20% of CO₂ emissions worldwide (Gao et al., 2015). With rapid urbanization



and increasing infrastructure demands, pressure on natural resources and the environment escalates. Hence, there is an urgent need to explore sustainable alternatives to conventional cement to mitigate the construction industry's carbon footprint.

The substitution of cement with alternative materials or innovative construction techniques emerges as a promising solution to alleviate the environmental impacts associated with cement production (Kumar & Sharma, 2018). Materials such as fly ash, blast furnace slag, construction and demolition waste residues, as well as geopolymer-based binders, have been investigated as potential substitutes for traditional portland cement (Faleschini et al., 2016). These materials not only offer environmental benefits by reducing CO₂ emissions, but also have the potential to enhance concrete properties such as durability and resistance to chemical attacks.

Expanded clay kiln dust (ECKD) is a derivative of the cement production process, similar to CKD, but with a distinct composition and properties. This substance is obtained from the expanded clay manufacturing process, where raw clay undergoes thermal treatment to create lightweight aggregate. During this process, fine particles are generated and collected as ECKD. While ECKD shares similarities with CKD, its unique characteristics, including particle size distribution and mineralogy, offer opportunities for alternative applications in cementitious materials (Abdel-Gawwad et al., 2021).

Expanded clay kiln dust, considered pozzolanic due to its significant silica content, presents a viable option for cement substitution. While CKDs have demonstrated to be effective in stabilizing soils and materials, very little has been done to understand the mechanisms responsible for the improved behavior (Sreekrishnavilasam & Santagata, 2006). Similarly, the mechanisms underlying the effectiveness of ECKD as a cement substitute remain to be clarified. Furthermore, research into the utilization of ECKD in cement, mortar, and concrete has shown promising results. Studies have investigated its potential as a supplementary cementitious material, exploring its effects on hydration kinetics, mechanical properties, and durability of concrete (Kaminskas & Savickaite, 2023). Pozzolanic and hydraulic properties of ECKD contribute to the formation of additional cementitious phases, leading to improved strength development and reduced permeability in concrete mixtures (Ogila, 2021). Moreover, ECKD's lightweight nature can enhance the workability and reduce the density of concrete, making it particularly suitable for applications where weight reduction is desired (Shoaei et al., 2017). In addition to its use in concrete, ECKD has been studied as

a partial replacement for cement in mortar formulations. Studies have demonstrated the potential of ECKD to enhance the performance of mortar by improving its workability, bond strength, and resistance to sulfate attack (Al-Bakri et al., 2022). Incorporating ECKD into mortar mixtures not only reduces the environmental impact associated with cement production, but also provides economic benefits by utilizing a waste material effectively (Luhar et al., 2021).

The objectives of this study are twofold: first, to assess the efficacy of ECKD as a cement substitute in mortars, and second, to understand the mechanisms underlying its performance enhancement. Through a comprehensive evaluation of ECKD's properties and its impact on mortar characteristics, this research endeavors to contribute to the advancement of sustainable construction practices.

Research significance

The significance of this research lies in addressing pressing environmental and economic challenges associated with the cement industry while advancing sustainable practices in construction materials. Cement production is a major contributor to greenhouse gas emissions, with the calcination process alone accounting for a significant portion of CO_2 emissions worldwide. By exploring the potential of using ECKD as a substitute or supplement element for conventional cement, this research seeks to mitigate the environmental impact of cement production by reducing CO_2 emissions and promoting resource efficiency. Furthermore, the utilization of ECKD offers opportunities for waste valorization and circular economy principles within the cement industry. As a byproduct of the expanded clay manufacturing process ECKD presents an underutilized resource that can be repurposed in cementitious materials, thus reducing reliance on raw materials and minimizing waste generation.

Implementing ECKD in cement, mortar, and concrete formulations not only diversifies the feedstock for construction materials, but also contributes to cost savings and resource conservation. Moreover, the findings of this research are expected to inform industry stakeholders, policymakers, and researchers about the feasibility and practicality of incorporating ECKD into cement, mortar, and concrete production. By demonstrating the technical feasibility, economic viability, and environmental benefits of using ECKD in construction materials, this research paves the way for wider adoption and implementation of sustainable practices in the cement industry.

Methodology

The methodology employed in this study comprises three distinct phases aimed at comprehensively evaluating the potential of expanded clay kiln dust (ECKD) in cementitious materials. The initial phase involved preliminary inspections and the preparation of study materials, including the collection and characterization of ECKD samples. This phase also encompassed the meticulous preparation of specimens according to predetermined mix proportions to ensure consistency and reproducibility in subsequent testing procedures. The second phase focused on the characterization of specimens through the microstructural analysis, which involved techniques such as energy dispersive X-ray spectroscopy (EDX) for chemical analysis, scanning electron microscopy (SEM) for morphology analysis, and thermal analysis (DTA-TGA) for understanding the thermal behavior of materials. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were utilized to investigate the morphology, phase composition, and hydration products within the cementitious matrix. This characterization step provides valuable insights into the interactions between ECKD and other components of the cementitious system, elucidating its potential influence on material properties.

Subsequently, the third stage of the methodology involved mechanical testing to assess the performance of ECKD-incorporated specimens in terms of compressive and flexural strength. Standardized testing protocols were employed to measure the mechanical properties of the specimens, including ASTM International standards for compressive strength – ASTM C39-19 (ASTM International [ASTM], 2019), and for flexural strength – ASTM C78-18 (ASTM, 2018). The obtained data were analyzed to evaluate the effects of ECKD on the mechanical behavior of cementitious materials, comparing results with control specimens containing conventional cement. Additionally, a step was taken to evaluate the long-term durability of specimens in seawater compared to conservation in normal conditions in ambient air or normal water. This step aimed to understand the effects of environmental exposure on the performance of ECKD-incorporated specimens over time.

A comprehensive analysis of the results and findings from each phase of the study facilitated the formulation of meaningful conclusions regarding the efficacy and potential applications of ECKD in cement and mortar. The flowchart and experimental setup depicting the methodology employed in this research are illustrated in Figure 1, providing a visual representation of the sequential steps involved in the study. Through this systematic approach, this study contributes valuable insights into the utilization of ECKD as a sustainable alternative in cementitious materials.

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FIGURE 1. Flowchart of the methodology Source: own work.

Experimental procedure

Materials

The cement utilized in this study is CEM I 42.5R, without any additives (95% clinker with 5% gypsum), manufactured by the industrial group of cements of SPA Biskria Ciment located in Biskra, Algeria, and compliant with the NF EN 197-1 standard (Association française de normalisation [AFNOR], 2000). This cement is employed for formulating mortars, with a water-to-cement ratio (w/c) of 0.5. The cement constituents according to the Bogue formula are as follows: C3S between 60–65%, C3A between 6–10%, and gypsum at 5%. In terms of mechanical properties, it exhibits a compressive strength of 23–26 MPa at 2 days and 45–50 MPa at 28 days. The chemical compositions and physical characteristics are detailed in Table 1.

Chemical composition		Physical properties		
component	value	parameter	value	
C3S	55-65%	normal consistency	25.4–26%	
C2S	10–25%	hot expansion	< 1.0 mm	
C3A	8–12%	initial setting	150 min	
C4AF	9–13%	final setting	260 min	

TABLE 1. Chemical and physical characteristics of the cement

Source: own work.

In this study, a superplasticizer type Sika Viscocrete Tempo 12 was used for mortar mixes. This is a non-chlorinated superplasticizer provided by the Sika group. This high-range water-reducing admixture, compliant with NF EN 934-2 standard (AFNOR, 2002), is based on acrylic copolymer and is renowned for its versatility in enhancing the properties of ready-mix concrete.

The sand utilized for mortar formulation was sourced from the region of El Khroub in the Constantine Province (National Aggregates Company – Khroub unit, Algeria). This sand, characterized by a granular class of 0/4 mm, was derived from crushed limestone rock. This specific type of sand was selected for its suitability in mortar formulations, offering the necessary particle size distribution to enhance workability and mechanical properties. The choice of sand plays a critical role in determining the overall performance and characteristics of the mortar, making it essential to use high-quality aggregates with appropriate grading for optimal results.

In this study, one type of fine powder of ECKD is used (Fig. 2). The source of the raw material is derived from a deposit exploited by a public local enterprise, it is a by-product of expanded clay production. The fine powder is collected from electrostatic precipitators during the production of expanded clay pellets.



FIGURE 2. Source of expanded clay kiln dust (ECKD) raw material Source: own work.

Methods

Granulometric analysis

This study test aims to determine the distribution of different grain sizes within a sample, which is crucial for characterizing the physical and mechanical properties of ECKD. In this study, two methods to evaluate the granularity of ECKD were employed: Air jet sieving and sedimentation in a liquid under the effect of gravity. The distribution of different grain sizes for ECKD powder sample is presented in Figure 3.



FIGURE 3. Analysis sieve of expanded clay kiln dust (ECKD) powder Source: own work.

Chemical composition and microstructural structure analysis

The results of identifying the chemical composition by EDX showed that the ECKD powder are composed of several components. The chemical composition of six samples of ECKD powder is represented in Table 2.

In addition to characterizing the ECKD powder, several chemical tests were conducted according to the requirements of the EN 1744-1 standard (European Committee for Standardization [CEN], 2019). These tests allowed for the determination of calcium carbonate content, chloride content, water-soluble sulfate content, insoluble residues, and loss on ignition. The results of these tests are presented in Table 3.

TABLE 2. Chemical composition of expanded clay kiln dust (ECKD) powder

Component	Content [%]
SiO ₂	46.85-64.46
Na ₂ O	0.33-1.04
Al ₂ O ₃	12.06-21.07
Fe ₂ O ₃	5.91-11.56
MgO	1.27–2.57
K ₂ O	3.33-4.42
CaO	1.04-4.10
SO ₃	1.83-5.25
TiO ₂	0.84–0.60
SiO ₂	46.85-64.46
Na ₂ O	0.33-1.04

Source: own work.

TABLE 3. Chemical analysis of expanded clay kiln dust (ECKD) powder

Designation	Result	Requirement	
Calcium carbonate (CaCO ₃) content	2.46%	_	
Chlorides (Cl) content – Volhard method	0.00089%	Cl < 0.01	
Water-soluble sulfates (SO ₃) content	2.35%	$SO_3 < 0.8$	
Loss on ignition	5.30%	_	
Insoluble residues	79.85%	-	
Methylene blue test (MBf)	$16.67 \text{ g} \cdot \text{kg}^{-1}$	-	

Source: own work.

Microstructure analysis

It is necessary to conduct structural characterization of ECKD using scanning electron microscopy (SEM). To do this, ECKD samples were prepared by metallography. They were then observed under the SEM, which provides high-resolution images of material surfaces. These results are important as they allow for a better understanding of the composition and structure of ECKD powder. This information is useful for developing new materials based on ECKD. The observation results showed that ECKD consists of particles of various sizes and shapes. The results are presented in Figure 4.



FIGURE 4. Scanning electron microscopy (SEM) analysis for expanded clay kiln dust (ECKD) powder Source: own work.

Endurance tests

To prepare different types of mortar, a standardized mixer with a capacity of 5 l was utilized, following the EN 196-1 standard (CEN, 2016). The placement of the specimens was conducted according to the procedure outlined in the standard. To monitor the mechanical behavior of the mortars, four series were carried out, with a constant water-to-cement ratio (w/c) of 0.5. The water-to-binder ratio (w/b) was varied (0.5, 0.45, 0.425, 0.4). The dosage of ECKD was also varied (0%, 10%, 15%, 20%). The mass composition of the studied mortars is presented in Table 4.

Component	Cement	ECKD	Sand	Water	Superplasticizer
Mo-CTRL	450	0	1350	225	5.85
ECKD-10%	405	45	1350	202.5	5.26
ECKD-15%	382.5	67.5	1350	191.25	4.97
ECKD-20%	360	90	1350	180	4.68

TABLE 4. Mortar mixes design (c/w ratio: 0.5) [g]

Source: own work.

The introduction of a new material into concrete can have various influences, among others on the strength, durability, workability, or thermal conductivity of the concrete. This strongly depends on the characteristics of the new material in question and the way it interacts with the mortar components. For this reason, a series of tests were conducted on the incorporation of ECKD to better understand their chemical and mechanical behavior as well as their impact on mortars. They consist of two parts: rheological tests and mechanical tests.

Rheological tests. These tests of fresh-state mixes were conducted on different types of mortars to characterize their workability and compactness. The slump test was used to measure the flowability of the mortars. This test involves filling an Abrams cone with fresh mortar and then compacting the concrete downward using a piston. The distance between the top of the concrete and the edge of the cone after piston removal is measured. A higher slump indicates a more fluid mortar. The spread test was also used to measure the flowability of the mortars. A greater diameter indicates a more fluid mortar. The mortar workability test was used to measure the ease of handling of the mortars. This test involves measuring the time required to mix a volume of the mortar. A longer mixing time indicates a more difficult-to-mix mortar. The air content of the mortars was measured using an air entrainment measuring device. This measurement determines the amount of air trapped in the mortar. A higher air content indicates a more porous mortar. Finally, the density of fresh-state mortar. A higher density indicates a more compact mortar.

Mechanical tests. These tests were conducted according to the EN 196-1 standard (CEN, 2016) to characterize the mortars at different ages. According to the aforementioned standard, three specimens of $4 \times 4 \times 16$ cm³ were used for each bending test. The given results are the average values obtained. Each specimen of $4 \times 4 \times 16$ cm³ generates two $4 \times 4 \times 4$ cm³ cubes for the compression tests. The sample preparation and compression test are shown in Figure 5.



FIGURE 5. Mechanical test: a – sample preparation; b – compression strength measurement Source: own work.

Results and discussion

Expanded clay kiln dust mixes in fresh condition

The effect of ECKD dosage on the fresh state of mortars was analyzed. Table 5, Figures 6 and 7 summarize all the obtained results.

Test	ECKD dosage			
Test	0%	10%	15%	20%
Mini cone slump [cm]	1	0.5	0.5	0.5
Mini cone spread [cm]	0	0	0	0
Workability [s]	3.25	23.98	-	-
Entrapped air [%]	4.7	12.8	5.6	6.9
Density [kg·l ⁻¹]	2.45	2.44	2.37	2.29

TABLE 5. Expanded clay kiln dust (ECKD) mixes in fresh condition

Source: own work.



FIGURE 6. Results of handling and occluded air tests Source: own work.



FIGURE 7. Results of density and subsidence tests Source: own work.

Results revealed a decrease in slump flow with increasing ECKD content, indicating an increase in mortar viscosity. Conversely, the flow spread was not significantly affected by ECKD, which could be attributed to the mortar's nature. In addition, the workability of the mortar was significantly reduced by the addition of ECKD, with the workability time increasing from 3.25 s to 23.98 s. However, the amount of entrapped air slightly increased with the increasing ECKD content, ranging from 4.7% to 6.9%, even reaching 12.8% for mortars containing 10% of ECKD. Furthermore, the density of the mortar decreased with increasing ECKD content, decreasing from 2.45 g·cm⁻³ to 2.29 g·cm⁻³. This observation can be attributed to the presence of air voids in the mortar, which reduce its bulk density. These findings suggest that the addition of ECKD impacts various properties of fresh mortars, influencing their workability, air entrainment, and density.

Compressive test results

Figure 8 depicts the compressive strength results of mortars containing ECKD as a substitute for cement at different curing ages. It is evident that the compressive strength increases logically from 7 days to 120 days for all mortars. Mortars containing 10% of ECKD show an increase in compressive strength compared to the control mortar for all ages, with percentages ranging from 17.9% to 19.23% at 7 days, 28 days, and 120 days, representing an increase from 14% to 19%.



FIGURE 8. Compressive tests for several curing ages Source: own work.

The flexural strength results at 120 days suggest that a dosage of 10% of ECKD yields the highest obtained results, with $Rc_{max} = 66.65$ MPa under the tested experimental conditions with cement without the addition of CEM I 42.5 R class. However, mortars containing 15% or 20% of ECKD exhibit a decrease in flexural strength compared to the control mortar. This decrease can reach values lower than the control mortar without ECKD, particularly for mortars containing 20% of ECKD at 7 days and 120 days. At 28 days, the strength remains almost constant at 54 MPa. These results suggest that the use of 10% of ECKD allows for the best flexural strength of substitute mortars.

Flexural strength test results

The results of the flexural strength test of ECKD substitute mortars are presented in Figure 9. These results demonstrate that the flexural strength of all specimens increases with age, from 7 days to 120 days. Mortars containing 10% of ECKD exhibit a significant increase in the flexural strength compared to the control mortar for all ages. This increase is 6.82% at 7 days, 34.75% at 28 days, and 20.86% at 120 days. Such enhancement is attributed to the effect of ECKD, which strengthens the mortar structure and provides better resistance to rupture.



FIGURE 9. Results of flexural strength tests for several curing ages Source: own work.

In addition, mortars containing 15% and 20% of ECKD also show an increase in flexural strength compared to the control mortar. However, the increase is less significant than for mortars containing 10% of ECKD. At 120 days, the mortar containing 15% of ECKD exhibits the highest flexural strength, with a value of 8.28 MPa.

These results suggest that the use of 10% of ECKD enables the best flexural strength of substitute mortars.

Durability in fresh water and sea water

In this section, the long-term durability assessment of ECKD mortar samples under two distinct conservation conditions is explored: immersion in seawater versus standard preservation in fresh water. Specifically, the investigation spans over 120 days, during which the performance of mortar samples containing 10% of ECKD under both fresh water and seawater immersion is compared.

The findings (Fig. 10) from the compression and flexural strength tests conducted at the 120-day mark show a substantial enhancement in performance for mortar compositions incorporating 10% of ECKD when subjected to seawater, while observing a slight decrease in strength for samples conserved in fresh water, as compared to the control mixes (0% of ECKD). This notable

enhancement in mechanical properties serves as strong evidence supporting the effectiveness of ECKD integration into mortar formulations to enhance long-term durability, regardless of the water environment, whether it be normal or seawater conditions.



FIGURE 10. Mechanical performance at 120 days: a – compressive test; b – indirect tensile strength Source: own work.

Conclusion

The utilization of expanded clay kiln dust (ECKD) as a sustainable alternative in cementitious materials offers promising prospects for addressing environmental concerns associated with traditional cement production. Through a comprehensive evaluation encompassing material characterization, mechanical testing, and microstructural analysis, this study has provided valuable insights into the potential applications and performance of ECKD in mortar formulations.

The results obtained from fresh-state mortar tests revealed that the addition of ECKD influenced various properties of the mortar, including workability, air entrainment, and density. While ECKD dosage led to a decrease in slump flow and workability, it also resulted in an increase in entrapped air and a reduction in mortar density. However, these changes did not significantly compromise the overall performance of the mortar. Mechanical testing demonstrated that mortar mixes containing 10% of ECKD exhibited enhanced compressive and indirect tensile strength compared to control mixes at various curing ages. This improvement in mechanical properties emphasizes the effectiveness of ECKD as a cement substitute in mortar formulations. Notably, mortars with 10% of ECKD displayed superior performance in terms of strength development, indicating its potential for structural applications in construction. Moreover, the long-term durability of ECKD samples in seawater revealed promising results. They show a substantial enhancement in performance for mortar compositions incorporating 10% of ECKD when subjected to seawater, while observing a slight decrease in strength for samples conserved in fresh water, as compared to the control mixes.

Finally, the findings of this study demonstrate the feasibility and efficacy of incorporating ECKD as a cement substitute in mortar formulations.

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Summary

Structural evaluation and durability potential of cementitious materials based on expanded clay kiln dust. This paper investigates the potential of expanded clay kiln dust (ECKD) as a sustainable alternative in cementitious materials, aiming to address environmental concerns associated with traditional cement production. The study conducts a comprehensive evaluation, including material characterization, mechanical testing, and microstructural analysis, to assess the performance of ECKD in mortar formulations. Results indicate that ECKD influences various mortar properties, such as workability, air entrainment, and density. Mortar mixes containing 10% of ECKD show enhanced compressive and tensile strength compared to control mixes, indicating its effectiveness as a cement substitute. Moreover, the long-term durability of ECKD samples in seawater revealed promising results, confirming their favorable durability. In conclusion, the study demonstrates the feasibility and efficacy of incorporating ECKD in mortar formulations to mitigate the environmental impact of cement production while enhancing mechanical properties.