eVitroKhem. 2026; 5:396
doi: 10.56294/evk2026396

ORIGINAL

Design and Fabrication of Concrete Mixtures Using Metakaolin Calcined at Different Temperatures

Diseño y Fabricación de Mezclas De Concretos Empleando Metacaolín Calcinado A Diferentes Temperatura

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Cite as: Domínguez Pompa CR, Bernal Camacho JM, Diaz Romero Y, Martínez García VM. Design and Fabrication of Concrete Mixtures Using Metakaolin Calcined at Different Temperatures. eVitroKhem. 2026; 5:396. https://doi.org/10.56294/evk2026396

Submitted: 15-07-2025 **Revised:** 02-09-2025 **Accepted:** 08-11-2025 **Published:** 01-01-2026

Editor: Prof. Dr. Javier Gonzalez-Argote

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ABSTRACT

The present research is aimed at exploring formulations of concrete mixes that incorporate metakaolin (MK), derived from kaolin calcined at different temperatures (700 °C and 800 °C). The metakaolin was generated in a laboratory setting from pure kaolin extracted in the Magistral de Copala development area, Concordia, Sinaloa. The experimental design encompasses five variants of concrete mixes, including a reference standard and options with 15 % and 20 % cement replacement with metakaolin at 700 °C, as well as mixes with the same substitution percentage using metakaolin at 800 °C. The collected results indicate that fluctuations in the calcination temperature do not exert a substantial impact on the mechanical and deterioration resistance characteristics of concrete, as both temperatures promote the formation of metakaolins with high pozzolanic activity. The formulation highlighted for its superior performance in terms of mechanical strength (compression) and durability (microstructural parameters, electrical resistivity, chloride migration) is the one using MK calcined at 800 °C, replacing 15 % of the weight of the cement. These findings underscore the possibility of obtaining environmentally friendly mineral additions by substituting significant amounts of cement, thus contributing to reducing the carbon footprint associated with its manufacturing.

Keywords: Kaolin; Metakaolin; Concrete; Durability; Cement Replacement.

RESUMEN

La presente investigación se orienta hacia la exploración de formulaciones de mezclas de concreto que integran metacaolín (MK), derivado de caolín calcinado a diferentes temperaturas (700 °C y 800 °C). El metacaolín se generó en un entorno de laboratorio a partir de caolín puro extraído en el área de desarrollo Magistral de Copala, Concordia, Sinaloa. El diseño experimental engloba cinco variantes de mezclas de concreto, que incluyen un estándar de referencia y opciones con sustitución del 15 % y 20 % del cemento por metacaolín a 700 °C, así como mezclas con el mismo porcentaje de sustitución usando metacaolín a 800 °C. Los resultados recabados señalan que las fluctuaciones en la temperatura de calcinación no ejercen un impacto sustancial en las propiedades física y químicas de los metacaolines obtenidos, ya que al momento de ser empleados como sustituto parcial del cemento no se observaron cambios importantes en las características mecánicas y durables del concreto. La formulación destacada por su rendimiento superior en términos de resistencia mecánica (compresión) y durabilidad (parámetros microestructurales, resistividad eléctrica, migración de cloruros) corresponde a la que utiliza MK calcinado a 800°C, sustituyendo el 15 % del peso del cemento. Estos resultados subrayan la posibilidad de obtener adiciones minerales respetuosos con el entorno al sustituir cantidades significativas de cemento, contribuyendo así a reducir la huella de carbono vinculada a su manufactura.

Palabras clave: Caolín; Metacaolín; Concreto; Durabilidad; Reemplazo de Cemento.

INTRODUCTION

The main objective of the research was to explore new alternatives to meet the demand for materials in the construction sector. The research focused on the production of concrete samples with added metakaolin (MK), a calcined derivative of kaolin ($Al_2Si_2O_5(OH)_4$). These samples consisted of test tubes 10 cm in diameter (Ø) and 20 cm in length (e).

The properties evaluated included compressive strength, electrical resistivity, chloride migration, density, and porosity. A total of 60 concrete specimens with different percentages of metakaolin and different calcination times were produced.

Today, the excessive production of Portland cement (PC) has been identified as one of the primary sources of greenhouse gas (GHG) emissions, making it necessary to explore sustainable alternatives to this material.

Metakaolin, as a mineral additive, emerges as a viable option to replace cement in concrete manufacturing partially. Previous research has shown that the effectiveness of this material is linked to the calcination temperature of kaolin and the percentage of replacement in relation to the cement weight. Therefore, in this context, the definition of these variables is crucial for optimizing the final product.

This situational analysis underscores the necessity of exploring and developing sustainable alternatives in concrete production, particularly through the examination of mineral additives such as metakaolin. Understanding the factors that impact its effectiveness is a fundamental step towards more environmentally friendly construction that is less dependent on polluting materials.

Cement manufacturing is one of the primary sources of environmental impact.[1] Portland cement is used as a binder in concrete production. It is obtained by mixing selected raw materials (clay and lime) in a specific proportion, grinding them, and heating them to 1500°C. During the production of 1 ton of cement, fossil fuels and electricity are consumed, of which between 5 % and 7 % of artificial carbon dioxide (CO_2) emissions can be attributed to the cement manufacturing industry. It is estimated that global annual cement production will reach approximately 5,9 billion tons in the coming years, equivalent to 4,8 billion tons of CO_2.[2]

The depletion of raw materials extracted from natural geological structures for cement manufacturing has led to the exploration of alternatives, including natural and artificial waste. Various pozzolanic materials, such as fly ash, silica fume, and metakaolin, can be blended with ordinary Portland cement, thereby enhancing the cementing and mechanical properties of the resulting cement.[3]

Given this situation, this study proposes the manufacture of metakaolin from kaolin by subjecting it to different temperatures. The resulting metakaolin will be used in different percentages as a partial substitute for cement in concrete mixtures. The primary objective is to reduce greenhouse gas emissions, enhance sustainability, and improve the physical and durable properties of concrete.

THEORETICAL FRAMEWORK

Active admixtures are those that can react with water and calcium hydroxide in hydrated Portland cement at room temperature (pozzolanic admixtures) or react directly with water (hydraulic admixtures) to form cohesive and stable products. The reaction capacity of additives containing amorphous silica is similar to that utilized by the Romans with their materials at the dawn of concrete history.

The concept of supplementary cementitious material also encompasses materials rich in calcium oxide and other minerals that can react with water and solidify, similar to hydraulic cement. Therefore, they are referred to as hydraulic materials, as is the case with blast furnace slag or calcareous fly ash.[4]

Metakaolin (MK), classified as a class N pozzolanic material according to ASTM-C-618, is the dehydroxylated form of kaolinite clay, which is subjected to a calcination process at temperatures ranging from 500°C to 800°C. Between 100 and 200°C, clay minerals experience a substantial loss of adsorbed water, and between 500 and 800°C, kaolinite undergoes dehydroxylation, releasing water.

The dehydroxylation of kaolin to metakaolin is an endothermic process due to the large amount of energy required to remove chemically bound hydroxyl ions. Above this temperature range, kaolinite is converted to metakaolin, characterized by a two-dimensional order in its crystal structure.[5]

LEGAL FRAMEWORK

Table 1. Summary of the legal framework

Standard	Test or Classification
NMX-C-083-ONNCCE	Determination of the compressive strength of cylindrical concrete specimens such as molded cylinders and drilled cores.
NMX-C-159-ONNCCE	This Mexican Standard establishes the procedures for preparing and curing, either on site or in the laboratory, the concrete specimens used for the required tests.
NMX-C-156-ONNCCE	This Mexican standard establishes the procedures for determining the consistency of fresh hydraulic concrete.
NMX-C-486-ONNCCE	Contains specifications for controlling the properties of concrete in both its hardened and fresh states.
ACI-211	Provides guidelines for the proportioning of mixtures for these types of concrete.
ASTM C642	Determination of density, water absorption, and voids in hardened concrete.
NMX-C-077	Determination of the different particle sizes that make up gravel.
NMX-C-030	Aggregate sampling is done in a representative manner.
NMX-C-170	For reduction to test size or crushing.
NMX C-164	For determining the quality index of gravel for the preparation of concrete mixtures.
NMX-C-166	To determine the total moisture content to ensure quality and uniformity when producing hydraulic concrete mixes.
UNE-83988-1	Durability of concrete. Test methods. Determination of electrical resistivity.

METHOD

Materials

Kaolin extracted from an unnamed mine located in the Copala master development was used for calcination. X-ray fluorescence (XRF) studies were conducted to determine the chemical composition of the material.

The chemical composition of the metakaolins obtained with X-ray fluorescence (XRF) is shown in the following table.

Table 2. Chemical composition of metakaolin at 700°C and 800°C

Chemical composition of MK		
Component	**MK 700°C (%)**	**MK 800°C (%)**
SiO_2	56,70	56,90
Al_2O_3	20,50	20,50
K_2O	4,70	4,75
Fe_2O_3	1,74	1,74 %
MgO	0,35 %	0,34
TiO_2	0,20	0,21
CaO	0,15	0,16

Before calcination, the kaolin sample was manually ground to avoid diffusion problems and homogenize heat transfer during heat treatment. The sample was then placed in a granite crucible and calcined in a MARLA J-01 muffle furnace at two different temperatures: 700°C and 800°C, with a heating rate of 10°C per minute.

Concrete mix design using the ACI-211 method

In order to achieve a more effective application of concrete in various conditions and uses, a pumpable concrete will be prepared, establishing a maximum aggregate size (MAS) of 3/4" gravel. The minimum slump for all pumpable concrete will be set at 14 cm.

The methodology addressed the design of the concrete mix for a capacity of 1 m^3, generating the results detailed below:

Table 3. Concrete mix design

Mix design and material calculation using the ACI 211 method			
Cement type-class-brand: CEMEX CPO cement		Concrete placement: Pumpable	
Compressive strength (f´c): 250 kg/cm^2		Working conditions: Normal	
Sample age in days: 28		Type of additive: none	
Shrinkage: 14cm		Maximum aggregate size: 3/4	
Aggregate characteristics			
Quality	Gravel	Sand	Cement
P.V.S.S. (kg/cm^3)	1678	1603	-
P.V.S.C. (kg/cm^3)	1693	1708	-
Density	2,575	2,532	-
%Absorption	1,945	2,4	-
Maximum size mm-in	3/4"	#4	-
Finnesse module	-	2,54	-
Calculation data			
Liters of water/m^3: 210		Cement (kg/cm^3): LTS/AC: 339	
Water-cement ratio: 0,62		Liters of water/m^3 (A): 210	
Volume of aggregates: 0,65		Gravel (kg/cm^3): Volume of aggregate PVCG: 1100	
P. Vol. Fresh concrete (kg/cm^3) (P.V.C.F.): 2355		Sand (kg/cm^3): P.V.C.F.-C-A-G: 706	

Table 4. Dosage for the experimental campaign

Experimental campaign						
	C1-R	**MK700-15 %**	**MK700-20 %**	**MK800-15 %**	**MK800-20 %**	**Total**
Cement	8,52	7,24	6,81	7,24	6,81	36,62
Water	4,80	4,80	4,80	4,80	4,80	23,98
Sand	16,12	16,12	16,12	16,12	16,12	80,61
Gravel	25,12	25,12	25,12	25,12	25,12	125,60
MK 700	-	1,28	1,70	-	-	2,98
MK 800	-	-	-	1,28	1,70	2,98
A/C	0,62	0,62	0,62	0,62	0,62	

After meticulously calculating the dosage for one cubic meter of concrete, this process was extended to determine the quantities of materials needed based on the specific samples required for the experimental campaign.

Tests to determine mechanical properties.

A pitch test was carried out on the test specimens using neoprene pads, following the guidelines established by ASTM-C-1231, with precision and meticulousness.

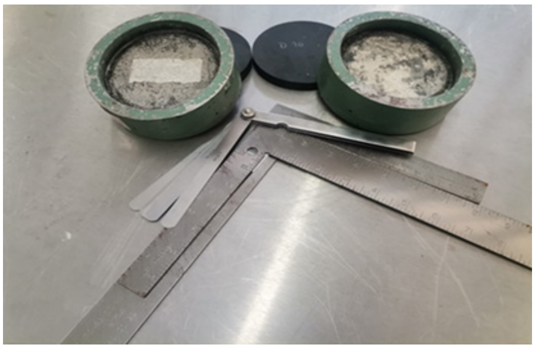

Figure 1. Equipment required for the endorsement of concrete cylinders in accordance with ASTM-C-1231

To begin, the dimensions of each of the concrete test specimens to be tested were taken, which are 10 cm x 20 cm. With these dimensions, the area of each of the test specimens was calculated. For the test, the specimen was first placed on the plate of the hydraulic press, aligning the axis of the sample with the center of thrust of the press, as shown in figure 2. A constant load was then applied, recording the readings in the computer system until the sample failed. The following equation was used to calculate the compressive strength of the test specimens.

$$f'c = \frac{P}{A}$$

Where:

f´c= Compressive strength in kg/cm^2

P= Breaking load, kg

A= Sample area in cm^2

Figure 2. Alignment of the sample axis with the center of thrust of the press

RESULTS

The results indicated a slight decrease in slump as the amount of MK increased, which is attributed to the greater fineness and absorption of the material. In terms of compressive strength, the MK800-15 % mixture demonstrated the best performance, achieving 98 % of the reference concrete strength at 28 days and surpassing it at 90 days. These results demonstrate the effectiveness of calcination at 800˚C to maximize the pozzolanic activity of the material.

The electrical resistivity of the mixtures with MK increased by up to 22 %, reflecting a more compact structure and lower permeability. Likewise, the chloride migration test revealed an 18 % reduction in ion penetration, demonstrating improved durability in aggressive environments.

Microstructural analysis using X-ray fluorescence (XRF) showed a decrease in $Ca(OH)_2$ content and an increase in C-S-H gel formation, which supports the mechanical and durability behavior observed experimentally.

CONCLUSIONS

Metakaolin calcined at 800˚C, used in a proportion of 15 % as a partial replacement for Portland cement, significantly improves the strength, durability, and electrical resistivity of concrete. Its incorporation contributes to reducing environmental impact and represents a viable alternative for developing sustainable concrete.

It is recommended to evaluate combinations of metakaolin with other additives, such as silica fume or fly ash, analyze their behavior under adverse environmental conditions, and conduct full-scale projects to validate their structural and economic performance.

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FUNDING

The authors did not receive funding for the development of this research.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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