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Effect of curing conditions on compressive strength behavior on alkali-activated ceramic wastes

Efecto de las condiciones de curado en el comportamiento de resistencia a la compresión en residuos cerámicos activados alcalinamente

Efeito das condições de cura no comportamento da resistência à compressão em resíduos de cerâmica ativados por álcali

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Abstract

The industrial sector is responsible for the generation of a large amount of solid waste, of which some is partially recycled, but the majority is deposited in landfills or landfills causing various negative impacts on the environment. Alkaline cements are attracting growing interest for their potential to allow the industry to operate within the constraints imposed on CO2 emissions. The objective of this research was to know the effect of different curing conditions on the compressive strength behavior of alkaline activated ceramic residues. As a result, it was determined that an alkali-activated matrix is significantly influenced when cured at a temperature of 70 ° C, reaching, at 90 days of age, a compressive strength of up to 39.3 MPa in contrast to 27.08 MPa. when curing was carried out in environmental conditions of 20 ° C (± 0.5 ° C) and 60% (± 5%) of relative humidity (RH). This work was complemented with a microstructural analysis that included Scanning Electron Microscopy (SEM) and X-ray Energy Dispersion Analyzer (EDX).

Keywords: Ceramic waste; alkali activation; compressive strength.

Resumen

El sector industrial es responsable de la generación de una gran cantidad de residuos sólidos, de los cuales algunos son parcialmente reciclados, pero la mayoría son depositados en vertederos o rellenos sanitarios provocando diversos impactos negativos en el medio ambiente. Los cementos alcalinos están atrayendo un interés creciente por su potencial para permitir que la industria opere dentro de las limitaciones impuestas a las emisiones de CO2. La presente investigación tuvo como objetivo conocer el efecto de diferentes condiciones de curado sobre el comportamiento de resistencia a compresión de los residuos cerámicos activados alcalinamente. Como resultado se determinó que una matriz álcali-activada es significativamente influenciada cuando se cura con una temperatura de 70°C alcanzando, a los 90 días de edad, una resistencia a la compresión de hasta 39,3 MPa en contraste con 27,08 MPa cuando el curado se realizó en condiciones ambientales de 20°C (± 0,5 ° C) y 60% (± 5%) de humedad relativa (RH). Este trabajo se complementó con un análisis microestructural que incluyó Microscopía electrónica de barrido (SEM) y Analizador de dispersión de energía de rayos X (EDX).

Palabras clave: Residuos cerámicos; activación de álcalis; resistencia a la compresión.

Resumo

O setor industrial é responsável pela geração de grande quantidade de resíduos sólidos, alguns deles parcialmente reciclados, mas a maior parte é depositada em aterros ou aterros sanitários causando diversos impactos negativos ao meio ambiente. Os cimentos alcalinos estão atraindo cada vez mais interesse por seu potencial de permitir que a indústria opere dentro das restrições impostas às emissões de CO2. O objetivo desta pesquisa foi conhecer o efeito de diferentes condições de cura no comportamento da resistência à compressão de resíduos cerâmicos alcalinos ativados. Como resultado, determinou-se que uma matriz alcalina ativada é significativamente influenciada quando curada a uma temperatura de 70 ° C, atingindo, aos 90 dias de idade, uma resistência à compressão de até 39,3 MPa em contraste com 27,08 MPa. quando a cura foi realizada em condições ambientais de 20 ° C (± 0,5 ° C) e 60% (± 5%) de umidade relativa (UR). Este trabalho foi complementado com uma análise microestrutural que incluiu Microscopia Eletrônica de Varredura (MEV) e Analisador de Dispersão de Energia de Raios-X (EDX).

Palavras-chave: Ceramic waste; ativação alcalina; força compressiva.

Introduction

Currently, industrial waste generation is a significant concern in terms of the environment, health, and its final disposal. Recycling and using such wastes in innovative construction materials emerges to be a feasible solution not only to the pollution issue but also to an economical alternative in the construction sector by contributing a potentially sustainable source (Gaibor et al., 2019, p. 593). Recently, alkali-activated mortars (AAm)/concretes have been introduced as a new sustainable construction material to replace Ordinary Portland Cement (OPC) in the construction industry. It has been estimated that the production of AAm could allow the decrease of greenhouse gas emissions by nearly 70% in comparison with the production of OPC, which makes an environmentally friendly approach (Villaquirán-Caicedo & de Gutiérrez, 2018, p. 303) The ceramic industry manages great amounts of financial resources around the world and generates large numbers of jobs, being an important part of the general production chain (Azevedo et al., 2020. On the other hand, it is also known that the ceramic industry generates and disposal

to landfill significant amounts of wastes without any further treatment. Annually, the global production of ceramic tiles is more than 10 million square meters. China and Spain are among the largest ceramic producers in the world, with a production of 10.23 billion m2 in 2015, occupying more than half of all ceramic production globally (Wang et al., 2018), and 600 million m2 in the last years (Puertas et al., 2006, p. 1), respectively. It has been estimated in a survey that about 15-30% of production goes as waste in the ceramic industry (Senthamarai & Devadas Manoharan, 2005).According to the World Bank, in Latin America, 160 million tons of solid waste are produced per year, with an average per capita value of 1.1 kg/day, and only 3% reused or recycled. However, by 2030, with a predicted population increase of 17%, totaling 705 million, waste generation per capita will increase above 45%, reaching 1.6 kg/day. Moreover, in Latin America, up 60% of the waste ends up in improperly controlled landfills. It is known that solid waste composition in Latin America is mostly organic, although it is expected it will change, becoming mostly non-biodegradable (ECCA,2017).

For alkali-activated mortars, ceramic waste (CW) has been suggested as a significant silica and alumina source, which plays an important role in gel configuration and strengthening (Reig et al., 2013). Some advantages of using CW as the main starting material are the conservation of natural resources, energy, and lower cost, aside from reductions in CO2 emissions and other greenhouse gases. On the other hand, it is also known that AAm with a high calcium content will generate a calcium aluminum silicate hydrate (C-A-S-H) gel forms as the main reaction product. This lets to have a dense matrix with acceptable mechanical properties (Collins & Sanjayan, 2001). In this way, the ladle furnace slag (LFS), a calcium-rich aluminosilicate, which has been incorporated in the present work as a complementary precursor. Besides, the advantages regarding this material are great durability performance (particularly against acid and sulfate attack), fast setting and hardening, low hydration heat, high-temperature resistance, and lower CO2 emissions compared to OPC (Arbi et al., 2016).

Curing conditions also have an important effect on microstructural and mechanical strength development in most cementitious systems. Mild curing temperatures determine AFt and AFm phase formation on OPC, for example. Such conditions should, therefore, be expected to affect the setting and hardening of cement with an alkaline pre-zeolitic gel, since a structure of the synthesizing zeolites is known to be very sensitive to the synthesis conditions (Kovalchuk et al.,

2007). Some studies of different curing conditions (Krivenko, & Kovalchuk,2002), (Krivenko, & Kovalchuk, 2002b) showed that temperature and humidity play a key role in the development of the microstructure and consequently the properties of AAm. Improper curing conditions can cause carbonation at a very early stage, dropping pH levels, and resulting in a considerable delay in the precursors' activation rate and mechanical strength development.

The development of sustainable alkali-activated building materials and the possible use of ceramic waste from different sources either construction and demolition waste (CDW) and/or from ceramic industry waste has been studied by various authors. To name some, roof tiles production (Azevedo et al., 2020), (Azevedo et al., 2020), bricks manufacturing (Seco et al., 2018); (Amin et al., 2017) ceramic materials from eco-friendly geopolymer precursors (Villaquirán-Caicedo & de Gutiérrez, 2018, p. 303),development of high-strength alkali-activated pastes (Hwang et al., 2019, p. 520), among others. In Ecuador, there is a study about the sanitaryware ceramic waste reuse from one industry called “EDESA”, in the attempt of being incorporated in pavements. It is mentioned that 350 to 450 tons per month are generated and disposal to the landfill of the capital city, Quito (Simons, 2015). Thus, recycling these wastes in the construction sector, e.g through alkali activation, many large-scale waste streams can be converted into sustainable materials and at the same time relieved the ceramic industry waste problem, suggesting a win-win situation (Huseien et al., 2019). The building sector is the main customer of ceramic products plays an essential role to overcome some of the environmental issues. There is still room to introduce different types of industrial wastes and by-products in the production chain.

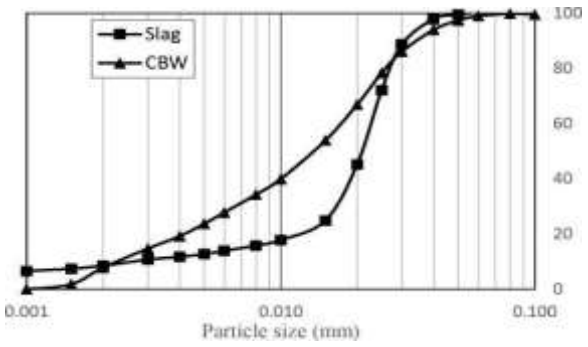
During alkaline activation, precursors are dissolved, and certain aluminosilicate gels are formed. These results are based on a series of intrinsic and extrinsic variables, including particle size, starting materials chemical composition, pH of the activating solution, nature and concentration of the activator, and curing time and temperature. The aim pursued in this research was to determine the effect of different curing conditions and mixture compositions on compressive strength behavior to optimize the polymerization reactions befall during the alkali activation of ceramic wastes. The research was complemented by a microstructural analysis, including Scanning Electron Microscopy (SEM), and X-ray Energy Dispersive Analysis (EDX).

Materials and methods

Materials description

Ceramic waste (CBW) from a Portuguese bricks manufacturing company and ladle furnace slag (LFS) supplied by the Portuguese ironwork company (Megasa) were used as precursors in this study. Originally, CBW had a diameter that fell within the 40-160 mm range which evidenced to be a poorly graded material and in the way to be used in the alkali activation process, CBW was ground in a ball mill with the help of 12 balls of 0.41 kg each one for 0.33 h/ 1000 rotations resulting on well-graded material where about 40% of the particles were under 0.075 mm (fine/course reference line). The LFS (as received in the laboratory) was sieved with a nominal mesh of 250 pm to be incorporated in the mixture. Gradation or particle size distribution (PSD) curves of precursors were determined by X-ray (Serigraph), Figure 1. The sodium silicate (SS) (Na2SiO3) was used in its commercial presentation as the main alkaline activator.

Figure 1: Particle size distribution (PSD)



The chemical composition of the starting materials was obtained by X-ray fluorescence (XRF) and it is presented in Table 1. CBW is characterized to be rich in silicon dioxide (54.89%) and aluminum oxide (26.28%). The LFS is a calcium-rich aluminosilicate (64.23%) and with an important percentage of silica (19.68%).

Table 2: Chemical composition of the CR, and LFS (% wt)

Precursor	Na2	MgO	AI2O	SiO2	P2O	SO3	CI2	K2O	CaO	TiO2	&2O	MnO	Fe2O	ZnO
CBW	0.13	1.30	26.28	54.89	0.42	0.59	0.12	3.97	0.64	1.40	0.30	0.86	9.10	-
LFS	0.31	3.54	4.86	19.68	-	4.69	-	-	64.23	0.31	-	0.35	1.39	0.64

Binders preparation

The mixture composition was based on the better results in compression strength from previous experimental work (Gaibor et al., 2019) However, it was modified by adding water and/or superplasticizer where the variation of the solid/liquid ratio allowed to determine the most efficient ratio, concerning the workability of the blends. Pastes characterization are presented in Table 2.

Table 2: Identification and characterization of the tested pastes

Paste ID	Precursor		Precursor/ Activ.	SP/ precursor.	Water/SS			SiO2 / Al2O3	SiO2 / Na2O
	CBW	LFS	SS	(wt. ratio)	(wt. ratio)				
M1	72	28	0.48	0.02	-	0.32	0.32	2.84	8.96
M2	75	25	0.45	0.02	0.05	0.29	0.29	2.76	9.59

The preparation of M1 consisted of homogenizing the CR, LFS, SS, and SP in an industrial mixer at minimum speed for three minutes. In case of M2, the mixing process took place in two stages, the precursors and the activator were first mixed for 1 minute in an industrial mixer at minimum speed, immediately the SP and water were added and mixed for two more minutes at maximum speed and it was finished with a second homogenization of the paste for three additional minutes at maximum speed. The flow properties of the fresh mortar were determined through the slump-flow test (BS EN 12350-8., 2010). During this test, the spread of the diameter passed from 100mm to 150 mm and from 110mm to 165 mm, for M1 and M2, respectively. No segregation was observed in any of the mixtures M1 or M2, this can be associated with the use of optimum precursor/activator ratio, beyond using the superplasticizer, which acts as micro-rollers and significantly reduces the friction and the flow resistance of the paste (ASTM C39 / C39M – 18, 2018). In both cases, the homogenized paste was transferred to a cubic stainless- steel mold with nominal dimensions of 50 x 50 x 100 mm3. Mechanical vibration was then applied for 2 minutes. M1 specimens were cured inside an oven, at 70°C for the initial 24 h, while M2 samples were cured in a climatic chamber (Fitoclima 28000 EDTU) with constant ambient conditions of 20°C (±0.5°C) and 60% (±5%) relative humidity (RH). After the 24 hours, both M1 and M2 specimens were demoulded and left to cure in the climatic chamber for the remaining 14, 28, and 90 days curing.

Uniaxial compressive strength (UCS) test

The uniaxial compression test was conducted at the civil engineering laboratory of the University of Minho at 14, 28, and 90 days curing time. Four replicates of nominal dimensions of 50 x 50 x 100mm3 were prepared and each of them was measured and weighed before testing. The UCS was adapted from the procedure described in ASTM C39/C39M-18 (Duxson et al., 2006)using a servo-hydraulic testing machine with an actuator of load capacity of 300 kN. Tests were carried out under monotonic displacement control, at a rate of 0.002 mm/min, and both the peak load and displacement were obtained from each test result. Stress-strain curves were plotted for all tests performed.

Mineralogical and microstructural characterization

The present study is complemented by a microstructural analysis of the initial materials and the different mixtures at 14, 28, and 90 days. After mechanical testing, a small hardened portion of a specimen from each batch was immersed in acetone to stop the chemical reactions, and later they were milled to get a fine powder (<0.45 ^m) to be analyzed by X-ray diffraction (XRD). The results were obtained from a PANalytical X'Pert Pro diffractometer, fitted with an X'Celerator detector and a secondary monochromator, with a CuKa radiation setting of 40kV and 30mA, a nominal step size of 0.017°, a rate of 100 s/step and a 20 range between 10 and 85°. The chemical compounds and energy dispersion were ascertained employing a FEI Quanta 400 scanning electron microscopy (SEM) and X-ray spectroscopy (EDS) from EDAX, respectively, using the same spectrum acquisition time and a ZAF correction model.

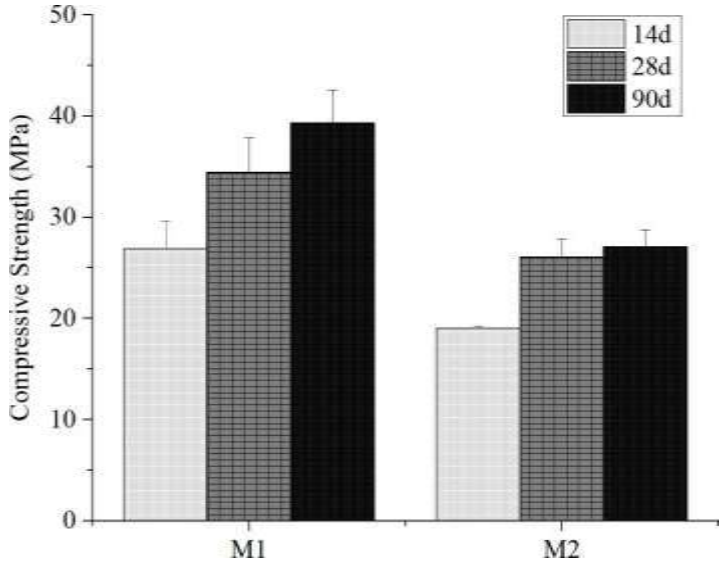
Results and discussion

Compression strength

Generally speaking, curing time increases the strength of the samples, results are shown in Figure 2 from which it can be inferred that curing conditions have a significant impact on the development of mechanical strength, notwithstanding values also vary due to the composition of mixtures. At 28 days curing, M1 attained higher strength (24.27%) than M2. The mechanical strengths of alkaline cement increase as the curing temperature increases, especially during the

first hours of reaction. This is due to an increase in temperature affects the kinetics of the reaction, accelerating it. However, it can be adopted until a certain point from which, the increasing mechanical resistance as a consequence of increasing the temperature, is no longer significant (Rivera et al., 2018)

Figure 2: Uniaxial compression strength of M1 and M2 (70°C and ambient T curing, respectively) pastes at 14, 28,



and 90 days curing

Mineralogical and microstructural characterization

Figure 3 shows the SEM characterization (300x magnification) of the microstructure of alkali cement synthesized from CBW and LFS. At 28 days curing age, M1 evidenced a denser compact matrix than M2 which indicates that the dissolution of the precursors was satisfactory, although in both cases unreacted small particles covered with reaction products and embedded in the matrix are observed. It is known that a greater solubility of precursor materials produces a greater amount of cementitious gel, which confers the mechanical properties to the synthesized material (Shoaei et al., 2019)This result accords with the compressive strength achieved for M1 (34.44 MPa) in comparison with M2 (26.08 MPa). Besides, one factor that could contribute to greater solubility of precursors in the alkaline-activated mix is the curing temperature, which was 70°C for 24 hours in the case of M1. In this regard, some authors (Kovalchuk et al., 2007), note that an increment

in the temperature of the alkaline activation process contributes to the dissolution of a greater quantity of the small particles (< 45 ^m) of precursors since at room temperature the rate of dissolution of this type of materials is considerably slow. That is reflected in the obtained results (Figure 2).

Regarding gel developed, the blend of CBW and LFS (as a complementary precursor) resulted in C- A-S-H gel type characterized for its calcium content. The general chemical composition of the two studied mixtures is presented in Table 3.

Figure 3: SEM images of M1 and M2 (70°C and ambient T curing, respectively), after 28 days curing time

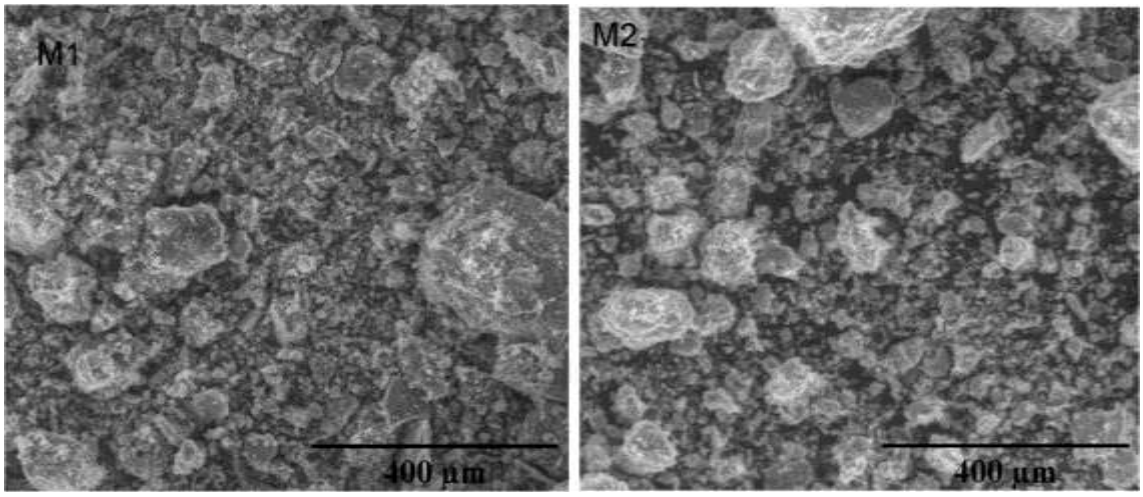


Table 3: Chemical composition of mixtures at 28 days curing time, (%Wt)

	M1 70°C	M2 ambient T
Na2O	7.01	7.61
MgO	1.63	1.69
AI2O3	15.36	15.64
SiO2	55.62	57.38
P2O5	0.96	0.76
SO3	2.12	1.76
Cl2O	0.27	0.39
K2O	2.50	2.26
CaO	10.70	9.59
TiO2	0.79	0.63

MnO	0.21	0.11
Fe2O3	2.83	2.18
CaO/SiO2	0.19	0.17
NaO/Al2O3	0.46	0.49
SiO2/Al2O3	3.62	3.67
SiO2/Na2O	7.94	7.54

Conclusions

This paper aimed to know the effect of curing conditions on alkali-activated ceramic wastes mechanical properties (compressive strength) since this is a key step in the industrial manufacture of these materials.

The hardening of an alkali-activated matrix was significantly influenced by the curing conditions which determine the water available during the first hours of the reaction. Therefore, it performs an essential role in the kinetics and degree of reaction, the development of the microstructure (such as porosity and phase composition), and the mechanical performance (mechanical strength, shrinkage, elasticity modulus, among others) of alkaline cement with silicoaluminium base. The highest compressive strength (39.3 MPa) was reached when samples were subjected at 70°C in the early 24h in contrast to ambient temperature curing (27.08 MPa) at 90 days age.

M1 and M2 mixtures are calcium-rich aluminosilicates which are determined by the precursors or starting materials, this positively affected the strength of the samples.

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