



## Coal ash Portland Cement Mortars Sulphate Resistance

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Received 01 September 2020; Revised 21 November 2020; Accepted 22 December 2020; Published 01 January 2021

### Abstract

Coal fly ash (CFA), coal bottom ash (CBA) are residues produced in thermo-electrical power stations as result of the coal combustion in the same boiler. Therefore, some characteristics of the coal fly ash (CFA) are comparable with those of the coal bottom ash (CBA). Nevertheless, coal bottom ash size is larger than coal fly ash one. Consequently, it was found that it is necessary to grind the coal bottom ash (CBA) to reach a similar size to that one of the CFA. The objective of this paper is to evaluate the performance of Portland cement mortars made with coal fly ash (CFA), coal bottom ash (CBA) or mixes (CFA+CBA), against sulphate attack. The methodology is based on the expansion of slender bars submerged in a sodium sulphate solution (5%) according to the ASTM C-1012/C1012-13 standard. It has been found that mortars elaborated with CEM I 42.5 N (without ashes) presented the largest expansion (0.09%) after a testing period of 330 days. Mortars made with CEM II/A-V exhibited lower expansion (0.03%). Summing up, it can be established that mortar expansion decreases when the coal ash amount increases, independently of the type of coal ash employed. The novelty of this paper relies on the comparison between the performances of Portland cement mortars made with coal fly ash (CFA) or coal bottom ash (CBA) exposed to external sulphate attack.

*Keywords:* Mortars; Sulphate Resistance; Durability; Coal Ash; Portland Cement.

## 1. Introduction

Portland cement is the material most widely used in the construction industry worldwide and it is estimated that its production releases approximately 7.4% of global anthropogenic emissions of carbon dioxide [1]. The cement industry follows several levers to reduce the unavoidable environmental effects of producing Portland cement, but probably, among all of them, the increase of the period of service-life of the cement-based materials is most important, for instance, by crack reduction [2]. Kulkarni et al. (2020) used the technology named “microbiologically induced calcium carbonate precipitation (MICP)” to repair cracks and they found a reduction in permeability in the range from 65 to 85% for cracks ranging from 0.12 to 1.3 mm [2]. The second one could be the production of blended cements. Therefore, the cement industry has an important role in reducing carbon dioxide emissions to minimize the climate change [3].

Coal fly ash is a waste of coal which annual worldwide generation is over 900 million tons (China 580 Mt; India: 169.25 Mt; USA: 43.5; Mt; Europe: 26 Mt; Australia: 14) [4]. Nevertheless, coal fly ash use is still estimated to be just

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<http://dx.doi.org/10.28991/cej-2021-03091640>



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about half of the production level, mainly as cement and concrete constituent with replacement amounts between 15 and 25% [5]. Coal fly ash mitigates the damage of sulphate attack by reducing tricalcium aluminate hydrate, consuming  $\text{Ca}(\text{OH})_2$  in the pozzolanic reaction and providing a denser matrix [7]. Then, concrete containing coal fly ash improves the resistance against sulphate attack of concrete made with plain concrete with 30% replacements [7]. Better performances with 60% cement replacements has been found, however, these concretes have low early age strengths [4]. In order to increase the reactivity and hydration rate, higher the fineness of fly ash should be provided [4]. In addition, water-binder ratio is a control factor regarding the damage of concrete subjected to sulphate attack [7].

Sulphate attack was first studied by Candlot in the 1890's. He attributed the expansion of cements to the ettringite crystals formation, which was named as "cement bacillus" [8]. During the XX century, sulphate attack to mortars and concretes has been extensively reported, but none of them contained ground coal bottom.

Sulphate attack to the cementitious materials is a series of chemical reactions and physical processes that can act in a combined manner. Two main chemical mechanisms are involved in the sulphate attack: gypsum formation and ettringite formation and the usual physical form of the attack is salt crystallization. In general, it may be defined as a kind of deterioration in which any sulphate salt interacts with a cementitious material [9-11]. Given that, formation of thaumasite ( $\text{CaSiO}_3 \cdot \text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}$ ) is a product of the sulphate attack [12].

Calcium hydroxide and alumina-bearing constituents are very vulnerable to sulphate attack [13]. Some effects of sulphate attack are spalling, cracking, paste softening and loss of mechanical properties, among others, but the most harmful sulphate reactions are the formation of ettringite and gypsum.

Harmful effects on the cementitious material depend on the type of sulphate solution, pore network, type of cement and so on. Therefore, sulphate attack testing methods may not be realistic. In fact, laboratory testing is not representative of field conditions [14]. Consequently, they may not be adequately interpreted.

There are several testing methods to induce a sulphate reaction in a cement-based material. However, differences in results can be reached depending on the sulphate solution concentration, type of the cation, temperature, experimental setup and so on. The most commonly used standardized testing methods are defined in the ASTM C 1012 [15] and ASTM C 452 [16]. Both of them have been extensively criticized due to their inadequacy in simulating field conditions. For instance, sulphate solution concentration in the testing method and in the field, and other exposure parameters, are different.

The aim of this work is to study the effect of sulphates attack in ground coal bottom ash mortars by using the bar expansion method. The results are compared with the ones recorded for fly ash mortars. Accordingly, the structure of the article is divided in Experimental (materials used to prepare the tested mixes and testing method), Results and discussion (characteristics of the coal ashes, length change and  $\text{CaO}/\text{SiO}_2$  ratio) and Conclusion (Figure 1).

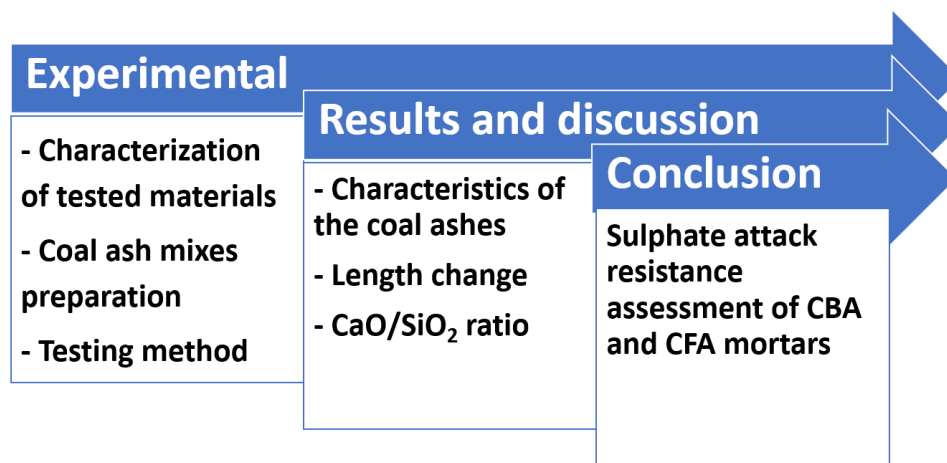


Figure 1. Represent flow diagram of the overall process

## 2. Experimental

### 2.1. Materials and Mixes

The materials used to make mortar prismatic specimens subjected to sulphate attack were ground coal bottom ash, coal fly ash, Portland cement, water and aggregates. Figure 1 shows the flow diagram of the overall process followed. The Portland cement used in the preparation of all mortar mixtures is designated as CEM I 42.5 N according to the European standard EN 197-1:2011 [5], which is considered to be of general use. Table 1 shows its chemical composition. Fineness for the cement, determined by means of the specific surface Blaine, was  $4050 \text{ m}^2/\text{kg}$ .

Coal bottom and fly ashes were collected from the same coal-fired power plant. Coal bottom ash was ground in a ball mill. Chemical characteristics of both ashes are given in Table 1. Fineness for the fly ash and ground coal bottom ash were 3976 m<sup>2</sup>/kg and 3463 m<sup>2</sup>/kg (5-8% residue on 45 μm sieve). They were mixed in the proportions given in Table 2. β-samples has 100% of fly ash, whereas Ω-samples has 100% of bottom ash.

CEM I 42.5 N was partially replaced by the ash mixes. Mixtures were selected to produce blended cements with similar compositions to CEM II/A-V, CEM II/B-V and CEM IV/A(V) standardized cements according to the European Standard EN 197-1:2011 [10]. Mortars were fabricated with cement/sand ratio of 1:3 and "water:cement" ratio of 0.5. Distilled water and standard sand were also utilized according to the EN 196-1:2016 [17]. The mortar preparation process, i.e. mixing, molding and curing, are described in the European standard EN 196-1:2016.

Sixteen different compositions were tested to assess the effect of the sulphate attack on the mortars made with the three main constituents considered in the present research study: ground coal bottom ash, coal fly ash and Portland cement clinker.

**Table 1. Chemical composition of bottom ash, fly ash and cement CEM I 42.5 N (%)**

Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Ti <sub>2</sub> O <sub>5</sub>	P <sub>2</sub> O <sub>5</sub>	LOI	IR*	Cl <sup>-</sup>
Coal fly ash	50.5	28.9	4.7	5.0	1.8	0.21	0.8	1.56	0.76	3.6	71.3	0.000
Coal bottom ash	52.2	27.5	6.0	5.9	1.7	0.13	0.6	1.53	0.74	1.8	75.7	0.001
Portland cement	20.9	4.3	3.5	62.7	1.9	3.4	0.9	0.25	0.10	3.7	1.04	0.023

\*Insoluble residue determined by the Na<sub>2</sub>CO<sub>3</sub> method (European standard EN 196-2:2013).

## 2.2. Testing Method

One of the most used testing method to assess the sulphate resistance of any new cement constituent is the standardized slender bar tests defined in the ASTM C-1012 [15], where prismatic specimens are submerged in a 5% sodium sulphate solution for six months. Length change is due to expansion of the material under attack. Then, the difference in length is measured along the time. This result is the way to evaluate the chemical resistance of such new cement constituent in sulphate environments.

**Table 2. Coal bottom ash, coal fly ash and CEM I 42.5 N mixes**

Cement mix	Raw material	Fly ash + bottom ash mix codification (%)					
		α	β	γ	δ	λ	Ω
CEM I	Coal fly ash	0					
	Coal bottom ash	0					
	Cement	100					
CEM II/A-V	Coal fly ash		10	9	8	5	0
	Coal bottom ash		0	1	2	5	10
	Cement		90	90	90	90	90
CEM II/B-V	Coal fly ash		25	22.5	20	12.5	0
	Coal bottom ash		0	2.5	5	12.5	25
	Cement		75	75	75	75	75
CEM IV/A(V)	Coal fly ash		35	31.5	28	17.5	0
	Coal bottom ash		0	3.5	7	17.5	35
	Cement		65	65	65	65	65

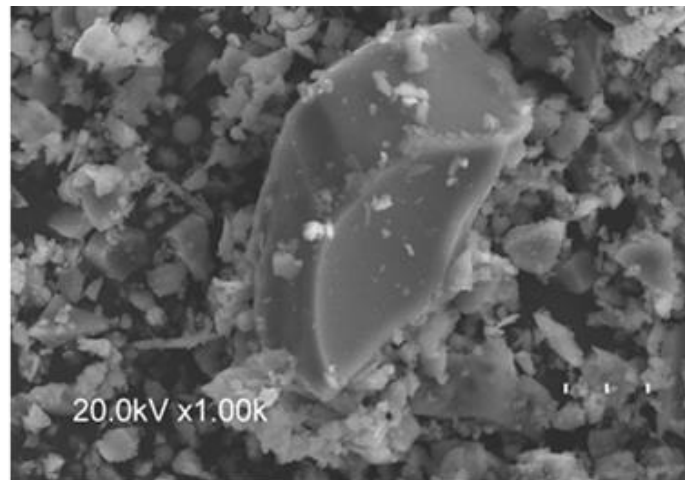
## 3. Results and Discussion

### 3.1. Characteristics of the Coal Ashes

Portland cement and coal ashes belong to CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Fe<sub>2</sub>O<sub>3</sub> system (Table 1). Coal ashes have more than 50% of silicon oxide, while Portland cement only about 20%. Also, Coal ashes have more than 25% of aluminum oxide, while Portland cement only about (5-10) %. On the other hand, Portland cement has more than 60% of calcium oxide, while coal ashes only have about 5%.

Both types of ashes are formed in the same boiler. Consequently, it is expected a similar chemical composition between them. However, slight differences in CaO, SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> can be seen. The main differences are the size and shape (Figure 2). In addition, coal bottom ash is denser than coal fly ash and its surface has irregular particles [16]. In

contrast, coal fly ash has a uniform distribution of spherical particulates with a fairly smooth surface. Such spherical shape is probably produced as result of the shrinkage of the liquid phase in the boiler.



**Figure 2. SEM photograph of the ground coal bottom**

### 3.2. Length Change

Mortar bar expansion is considered an adequate parameter to evaluate the sulphate attack of submerged specimens. However, the effect on the mortar bar expansion is discussed to assess the investigation's outcome. This procedure is followed in the ASTM C 1012 [15] and ASTM C 452 [16] to determine the resistance to sulphate attack of cement-based materials. In this method, the expansion of mortar samples is measured with the help of a standardized length comparator. Then, sample length is recorded along the time. It establishes the external sulphate resistance of the mortar with relation to an exposure class corresponding to the field environment. However, this testing procedure has been criticized due to its inadequacy in simulating some specific parameters found in field exposure. For instance, ASTM C-1012 [15] standard prescribes a 5% sodium sulphate solution to perform the sulphate attack. Accordingly, testing conditions are too severe due to the high sulphate concentration level, which is not representative of the sulphate concentrations found in the field in the majority of cases. Sulphate attack mechanism changes with modifying the sulphate concentration, adjusting the reaction products [19]. In this respect, the main product of attack is ettringite at low concentration level of sodium sulphate, i.e. below 1000 ppm  $\text{SO}_4^{2-}$  [20]. On the other hand, only gypsum is formed for high concentrations, i.e. above 8000 ppm  $\text{SO}_4^{2-}$ . Between both concentrations, ettringite and gypsum are encountered [21]. In addition, the sulphate concentration for a continuously immersed condition diminishes in the solution along with its pH with time. Thus, another major criticism of the testing method concerns with respect to the variation of the pH with the time. pH lowering promotes the degradation of mortars bars immersed in sulphate solutions. Therefore, it could be argued that mortar degradation of bars, immersed in sulphate solution, arises prior to expansion occurs. Aforementioned sulphate concentration level is mainly used for accelerating the laboratory testing method. Accordingly, ongoing research into sulphate attack is still subject of great discussion and debate about the type of chemical reactions taking place, source of the sulphate ion, external versus internal attack, deterioration mechanism, and so on. In this paper, its potential use to check the coal ash resistance to sulphate attack is assessed.

Summing up, the expansion of mortar test is another accelerated laboratory method which is rather controversial due to the lack of correlation with field conditions. Nevertheless, this method has been selected to compare the performance of coal bottom ash and coal fly ash as shown in Figure 2. During the first 28 days, all the mortars exhibited a great development of the slender bar expansion due to the sulphate attack. After this 28-days period, a second period from 28 to 90 days was observed. Finally, a third step can be observed in which only CEM I 42.5 N mortars keep on expanding.

Reference mortar made with Portland cement CEM I showed higher expansion than blended ones. In addition, increasing the amount of coal bottom ash or coal fly ash, a lower the expansion was recorded. According to ASTM C1012 [15], the limit for the expansion is 0.01% at one year of testing for Portland cements without additions. This limit increases up to 0.35%, for coal ash cements.

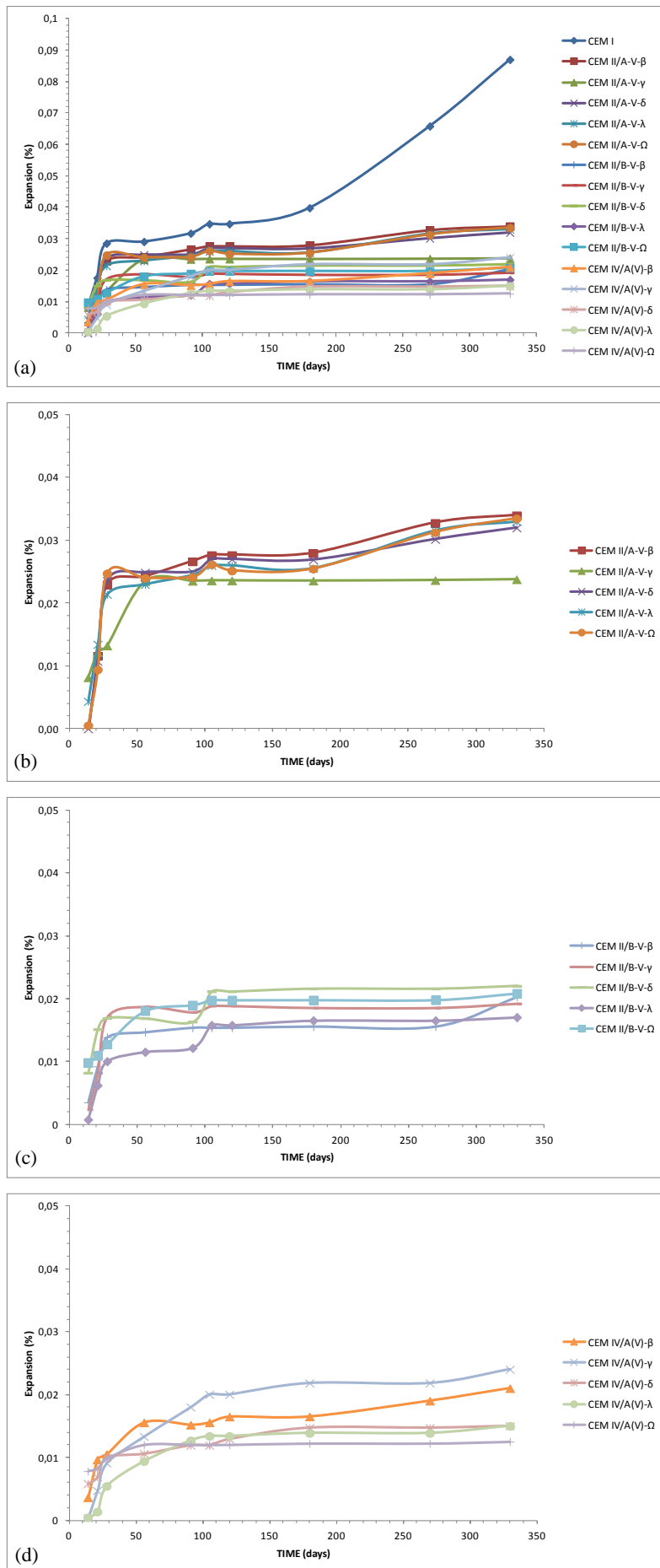


Figure 3. Mortar expansion due to sulphate attack: a) All the mixes; b) CEM A-V; c) CEM B-V; d) CEM IV/A (V)

The positive effect of ground coal bottom ash or coal fly ash utilization is evident. This finding has been largely consistent over time with other papers [13, 22-28]. However, it is quite convenient to consider that wet-dry conditions found in exposure under field conditions will trigger worse results in ground coal bottom ash or coal fly ash mortars as because of their fine microstructure [26], which might enhance capillary absorption [29, 30].

It is well-known that the more harmful reaction of sulphates with cement-based materials is the formation of ettringite, when they react with alumina-bearing phases (Equation 1-3). Another process is the salt crystallization, which is formed on the mortar surface [31]. However, salt crystallization is not evaluated by ASTM C1012 [21]. In particular, tricalcium aluminate (C<sub>3</sub>A) and calcium hydroxide are very sensitive to sulphate attack [13]. Tricalcium aluminate (C<sub>3</sub>A) is one of the main constituents of Portland cement and usually is restricted in many standards in order to avoid the ettringite salt formation. Another way to improve the resistance to the sulphate attack in cement-based material is by increasing the amount of Portland cement. In addition, compact concretes provide high sulphate attack resistance.

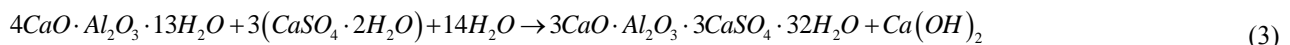
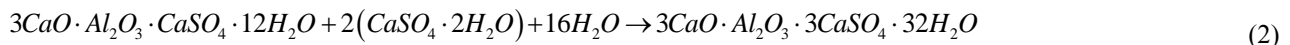
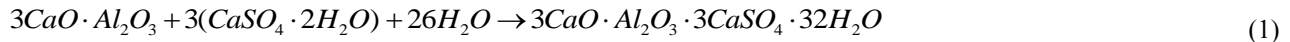
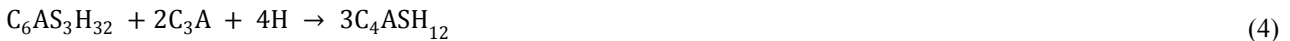


Figure 3 shows that the presence of coal ash, fly or bottom, improve the mortar resistance to the sulphate attack. This fact is attributed to several factors:

- Dilution of the C<sub>3</sub>A amount because the degradation rate is directly related to the C<sub>3</sub>A amount in the Portland cement;
- Partial Ca(OH)<sub>2</sub> consumption by the pozzolanic reaction.

Dilution of the C<sub>3</sub>A content allows a lower ettringite formation and the pozzolanic reaction triggers gel C-S-H formation with lower Ca/Si ratio, which is more durable than gel C-S-H formed in the Portland cement hydration. Calcium sulphate, normally in the form of gypsum, is intentionally added to Portland cement to form calcium sulfoaluminate or ettringite to regulate early hydration reactions to prevent flash setting, improve compressive strength development and reduce drying shrinkage. This primary ettringite arise within the first few hours after mixing with water and is not detrimental to mortar performance. This type of ettringite is transformed into calcium aluminate monosulphate hydrates, AF<sub>m</sub>, over time (Equation 4).



Secondary ettringite may be formed by dissolution and recrystallization from Ca<sub>4</sub>Al<sub>2</sub>O<sub>26</sub>H<sub>38</sub>, Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>, Ca<sub>4</sub>Al<sub>2</sub>Fe<sub>2</sub>O<sub>10</sub>, among other alumina-bearing phases. The conversion from monosulphate to trisulphate promotes the mortar expansion because ettringite has a molar volume from 3 to 8 times higher than the original material. In addition, ettringite has a density of 1.73 g/cm<sup>3</sup>, which is higher than the rest of the hydration products (2.50 g/cm<sup>3</sup>) (Equation 2). However, it has been reported that there is no variation in volume between calcium sulphate and calcium aluminate [32].

The relationship between the ash content and the slender mortar bar expansion promoted by the sulphate attack follows a decreasing trend (Figure 4). Therefore, CEM II/B-V and CEM IV/A (V) cements, with 25% and 35% of coal ash, exhibited an outstanding result with regard to the sulphate resistance. Also, CEM II/A-V had a good sulphate resistance, but less than CEM II/B-V and CEM IV/A (V).

As mentioned before, the reference cement, CEM I 42.5 N, displayed the longest expansion, approximately 4% after six months of testing and almost 10% after one year, i.e. three times higher than in the case of CEM II/A-V. This huge difference could be attributed to the lower permeability of the coal ash mortars.

Lignite combustion produces coal ashes with a high amount of SO<sub>3</sub> and CaO, which are not adequate to be used in cement-based materials exposed to sulphate environments [33]. Contrary to expectations, some papers reported a good resistance to sulphate attack of cement-based materials with coal fly ash presenting a high SO<sub>3</sub> content [28].

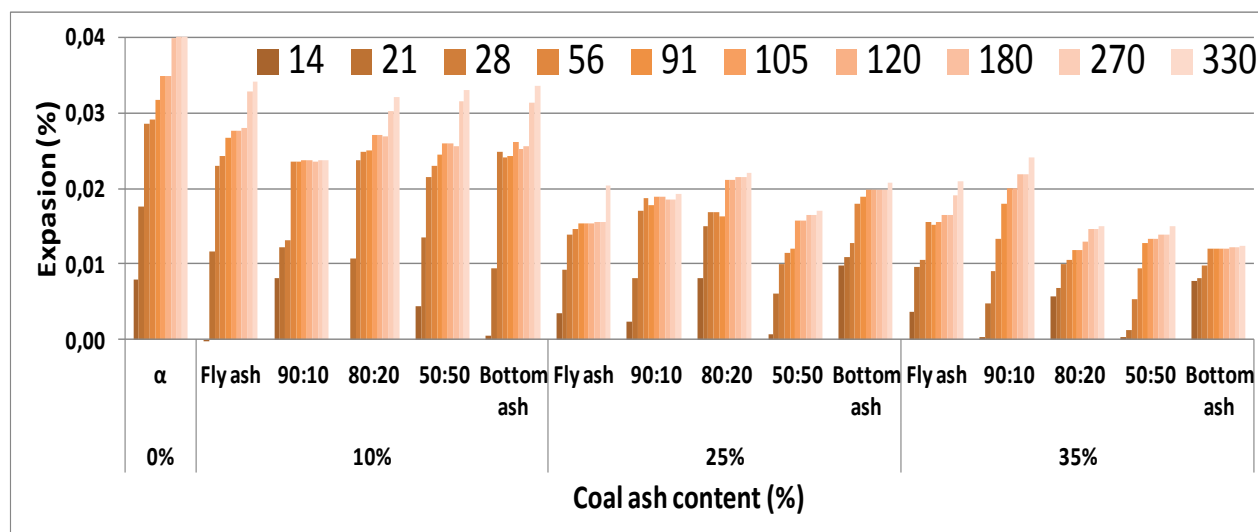


Figure 4. Relationship between the amount of coal ash and the mortar bar expansion due to the sulphate attack

In addition, a sulphate attack resistance parameter, R, according to Equation 5, has been proposed to evaluate the coal ashes [33]:

- $R < 1.5$ , the coal ash can be used in sulphate-resistant cements;
- $R > 3$ , the coal ash cannot be used in sulphate-resistant cements.

The sulphate attack resistance for coal ashes, R, was calculated for the ground coal bottom ash and coal fly ash (Table 3). The sulphate attack resistance parameter, R, was below 1 for both coal ashes. Thus, they are eligible to be utilized in cement-based materials exposed to sulphate environments.

$$R = \frac{CaO - 5}{Fe_2O_3} \tag{5}$$

### 3.3. CaO/SiO<sub>2</sub> ratio

Sulphate attack encompasses a series of physico-chemical interactions that occur between sulphates and hardened Portland cement paste. Several indicators have been proposed to assess the mortar resistance to the sulphate attack. For instance, the CaO/SiO<sub>2</sub> ratio is also utilized as criterion to evaluate the adequacy of the coal ashes with relation to the sulphate attack resistance [34]. Given that:

- $CaO/SiO_2 < 0.25$ , the coal ash is apt;
- $CaO/SiO_2 > 0.60$ , the coal ash is inadequate.

The CaO/SiO<sub>2</sub> ratio was calculated for the ground coal bottom ash and coal fly ash (Table 3) and was below 0.25 for both coal ashes. Thus, they can be considered as suitable to be utilized in cement-based materials subjected to sulphate environments. Accordingly, ground coal bottom ash and/or coal fly ash mortars provided an important enhancement with relation to the sulphate attack mostly due to the pozzolanic reaction where portlandite is consumed and Portland cement paste dilution.

Contrary to what is written before, it is thought that the mineralogical composition of the coal ashes could have a greater effect than the chemical composition on the sulphate attack resistance [35].

Table 3. Sulphate attack resistance, R, calculated according to Equation (5) for coal bottom ash and coal fly ash

Parameter	Coal bottom ash	Coal fly ash
SiO <sub>2</sub> (%)	48.12	46.84
CaO (%)	7.07	5.55
Fe <sub>2</sub> O <sub>3</sub> (%)	5.86	4.72
$R = CaO - 5 / Fe_2O_3$	0.35	0.12
CaO/SiO <sub>2</sub>	0.15	0.12



## 4. Conclusion

Sulphate attack encompasses several physico-chemical interactions that occur between sulphates and hardened Portland cement paste. From the analysis performed in this study to different mortar samples made with coal ashes, the following conclusions can be drawn. For the first time, it has already been stated that ground coal bottom ash mortars exhibited higher sulphate attack resistance than the coal fly ash ones, and much higher than the reference mortars without ashes. Nevertheless, ground coal bottom ash and/or coal fly ash mortars provided an important enhancement with relation to the sulphate attack mostly due to the pozzolanic reaction consuming the previously diluted  $\text{Ca}(\text{OH})_2$  content. The more the coal ash amount, the greater resistance to sulphate attack. A minimum coal ash content of 25% can effectively lead to enhanced resistance against sulphate attack.

## 5. Declarations

### 5.1. Data Availability Statement

The data presented in this study are available in article.

### 5.2. Funding

This research was funded by the CSIC (Intramurales Projects PIE 201660E054 y PIE 202060E176).

### 5.3. Conflicts of Interest

The authors declare no conflict of interest.

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