

Production of plates based on coal fly ash for their use as insulating materials in doors and fire break walls

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1. INTRODUCTION

In the field of passive fire protection, compartmentalization consists of establishing physical limits for a certain space, so that a fire can be contained within it. Creating fire resistant divisions or compartments allows us to set up an effective barrier between the fire and the elements we want to protect, thus preventing the fire from spreading to other areas. Among the most widely-adopted solutions for protection are fire doors, ceilings and divisions with plates or panels.

Thanks to the mechanical properties of panels, very high divisions can be built by reinforcing the panels with other construction elements. For the construction sector in general, panels offer a variety of solutions for compartmentalization and are relatively thin, easy to install, resistant to moisture and highly resistant to fire.

It is well-known that many commercial materials used as insulators or fire-proof products that provide passive fire protection—both plate-conformed and gunitable (calcium silicate, perlite or vermiculite)—have a chemical composition and physical properties similar to the mixtures of fly ash and other inorganic components used in this study¹⁻⁴.

With the aim of finding products based on fly ash and other wastes that can be used as alternatives to existing products used for compartmentalization with functions similar to those described above, different products have been developed through the CEFYR⁵ Project. The results from one of the compositions tested are presented in this paper.

2. EXPERIMENTAL

2.1 Manufacture of the plates

The plates were manufactured according to the following steps:

- The components of the mixture—comprised mainly of fly ash (60% w/w) and vermiculite along with ordinary Portland cement and other additives—were weighed.
- A small amount of water was put into a mixing tank, and each of the components of the mixture was added a little at a time; we tried to use enough water so that the mixture was homogenous.
- The mixture was kept in the tank for two minutes at a slow mixing speed and for one minute at a fast mixing speed.
- The mass was poured into 28 x 18 cm molds with different thicknesses; the plates were taken out of the molds after being allowed to set for 24 hours at ambient temperature.

2.2 Equipment used

To study the insulating capacity of the plates, the oven shown in Figure 1 was designed.



Figure 1. Oven for standardized tests

As Figure 1 shows, the oven door could be replaced with a special door in which the plates to be tested were inserted. This oven allowed us to record the surface temperature of the exposed face of the plate by means of a type S thermopar (inside the oven), which was used to regulate the power of the oven by means of a proportional controller, so that the standard temperature curve was produced.

The standard fire resistance test described in Spanish Regulation UNE-23.093⁶, the one most widely-used internationally, is the result of the observation and analysis of several actual fires, and it corresponds to the equation:

$$T = 20 + 345 \cdot \log_{10}(8t + 1)$$

where T is the oven temperature for the tests in °C and t is the time in minutes from the beginning of the test.

On the unexposed face, the temperature is registered by means of a Pt-100 probe with a stainless steel contact surface. At the same time, the oven has an output of 0-10 V, proportional to the power used, which allowed us to measure this parameter.

The technical specifications of the oven are the following:

- Power: 11 kW
- Type of electrical resistance: superkandal
- Interior dimensions (height x width x depth): 300 x 200 x 300 mm
- Volume: 18 L
- Maximum service temperature: 1350°C
- Control system: possibility of 20 programs with 16 segments

Furthermore, the resistance to compression and bending of the probes was measured in the Suzpecar test machine, model MEM-102/50t. The scanning electronic microscope (SEM) study was carried out using a JEOL JSM-5400 apparatus. Finally, for the mercury porosimetry, a Micromeritics 9320 unit was used.

3. RESULTS

The criterion for the insulating behavior of the materials tested is based on the general idea for insulation presented in Regulation UNE-23093-1⁶.

With that regulation in mind, in this study we considered the insulating capability as well as the time needed for the unexposed face to reach 180°C when the exposed face was regulated and controlled in such a way that the temperature-time relationship that establishes the standardized heating curve was always in effect.

Moreover, the apparent mean thermal conductivity was estimated using the oven's power output, which was registered by means of a data acquisition card. To do this, the heat flow of the oven was registered from the beginning to the end of the test for a specific sample; the heat flow of the oven with the door shut during that time and with the same heating program (blank) was then subtracted from that value. The difference between these two mean values was considered the heat flow (Q in W) that passed through the sample.

Thus, knowing the mean temperatures of the exposed (T_e) and unexposed (T_{ne}) sides and the dimensions of the plate (A, exposed area, and $x=E$, thickness), we can estimate the apparent mean thermal conductivity, k, according to Fourier's Law.

$$k = \frac{Q \cdot E}{A \cdot (T_{in} - T_{out})}$$

To determine Q, T_e and T_{ne} , mean values were recorded during the entire test period for one of the samples tested, as shown in Figure 2.

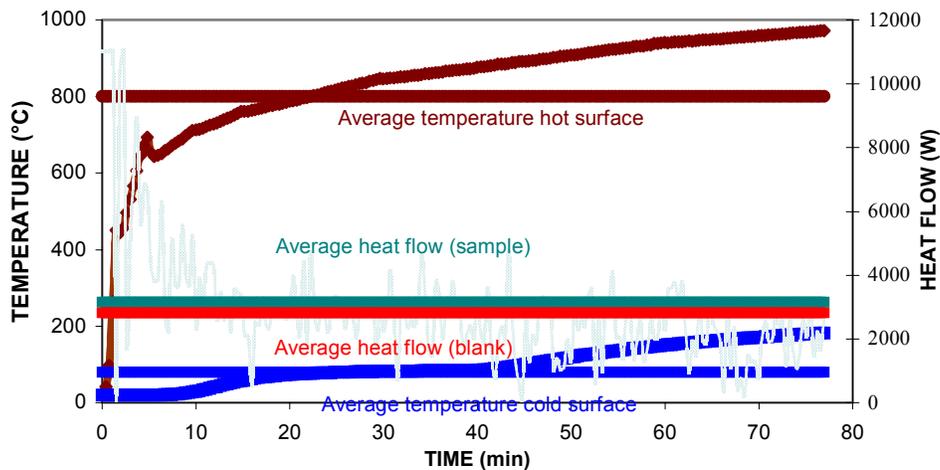


Figure 2. Determining thermal conductivity

Figure 3 shows the results obtained for thicknesses of 20 and 33 mm.

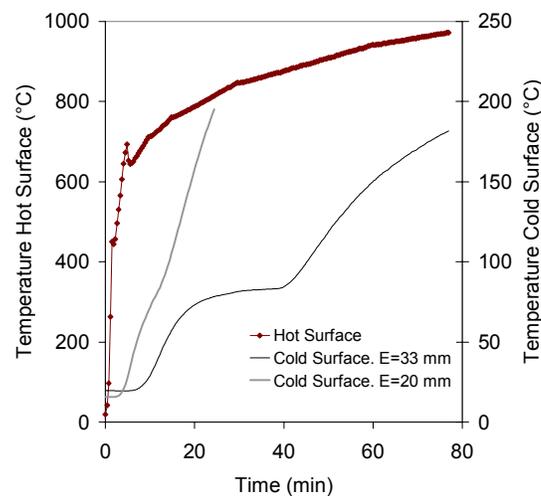


Figure 3. Thermal behavior of the product

The following observations can be made in regard to the above figure:

- There was an evaporation plateau of about 90° in the thicker plate, due to the formation of a front of water that runs through the entire plate and that is produced by the continuous evaporation and condensation of the water on the plate, that is, free moisture, water from hydration or water from the vermiculite which moves toward the unexposed face as a result of the pressure gradient generated in the plate due to the exposure to high temperatures⁷⁻⁹.
- The thickness of the samples greatly affected their insulating capacity

- It takes more than 75 min for the unexposed face of the 33-mm-thick plates to reach 180°C, which means that it is competitive with commercial plates of the same thickness that have been subjected to the same test.
- The mean conductivity of the product yielded a value of 0.28 W/m·K, which is particularly important considering that it was calculated based on standardized test conditions.

Furthermore, the density of the product was measured (around 500 kg/m³), as was its mechanical resistance to compression (σ_C) and bending (σ_F), before (0) and after the test (1). Table 1 shows the results. It can be seen that the product maintains satisfactory mechanical properties after testing.

	$\sigma_C(0)$ (kg/cm ²)	$\sigma_C(1)$ (kg/cm ²)	$\sigma_F(0)$ (kg/cm ²)	$\sigma_F(1)$ (kg/cm ²)
Product	6.6	3.9	6.3	1.8

Table 1. Mechanical parameters of the product

3.1 Explanation of the insulating behavior

In order to analyze the reasons for the good insulating behavior of the material, a scanning electronic microscope study was carried out on some samples. Figure 4 shows the results from some of unexposed faces from them.

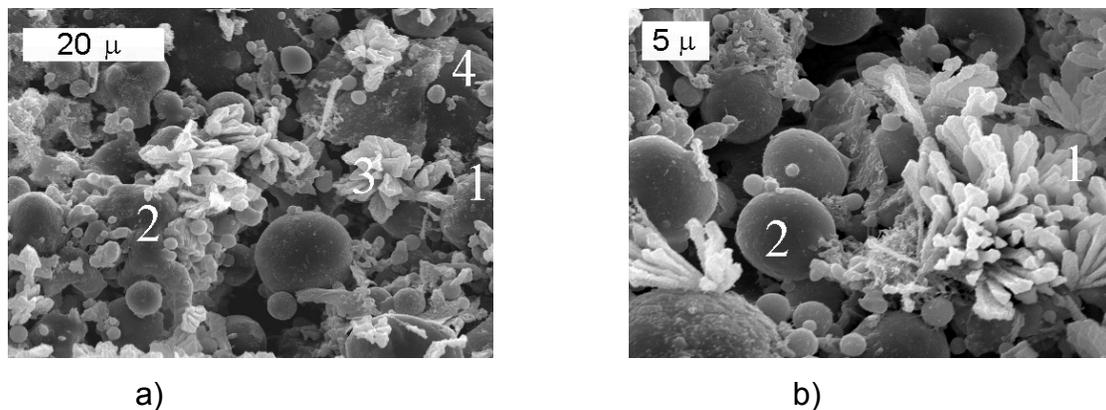


Figure 4. SEM analysis of the product: Unexposed face

In regard to image a) from the above figure, the first thing to note is how difficult it is to see whether the FA reacts with the lime produced in the hydration of the OPC. It seems that the FA serves as a contact surface, upon which the precipitation of the cement hydration products (acting as precipitation nuclei) is produced. The cement hydration products are responsible for the connection of the FA particles and the agglomeration of

the product. The fact that little cement is used means that few hydration products are produced, which in turn means that the resulting material is very porous, as can be seen in the above images. This is consistent with the porosity test for the material, in which diameters of 0.7-10 μm were obtained for most pores.

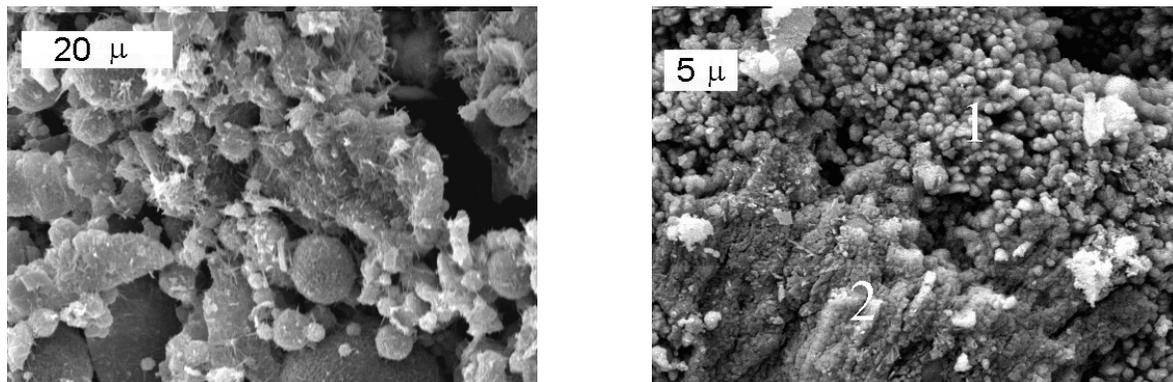
The components of the sample can be deduced with the help of the superficial microanalyses done on each of the points mentioned in Figure 4. Thus, for image a), the following can be assumed:

<u>Point</u>	<u>Component</u>
1	FA
2	Possibly Mullite
3	Hydration phases
4	Vermiculite

and we can confirm that the components of the samples remain unaltered and the hydration products serve as a bridge between them.

A more precise analysis of the above information can be seen in Figure 4b, where we can more clearly see how the surface of some of the FA particles serves as a basis for the development of the hydration products. Furthermore, the formation of the crystals characteristic of cement hydration (possibly crystals from the AF_m phases in point 1) and the FA particles (in point 2) can also be observed.

The effect of heating the material can be seen in Figure 5, where micrographs of the exposed face of the material are shown.



a) b)
Figure 5. SEM analysis of the product: Exposed face

The micrographs show an acicular structure caused by thermal transformations, with a disappearance of the hydration phases and the possible appearance of ettringite. There is also an agglomeration of material with superficial links between the particles, in such a way that it becomes difficult to distinguish each of the material's original components.

Finally, Figure 5b) shows another micrograph in which a general analysis only detected Ca, O, and S, with two different areas in point 1 (without S) and point 2 (with S). Since the sample was exposed for more than one hour to temperatures of approximately 1000°C, calcium sulphate and calcium oxide were probably produced as a result of reactions at high temperatures. The presence of CaSO₄ could have something to do with the loss of consistency in the sample on the unexposed face and with the reduction of the above-mentioned mechanical properties that took place as a result of heating.

SUMMARY AND CONCLUSIONS

The aim of this study was to obtain products, comprised mainly of coal fly ash, that can serve as alternatives to commercial calcium silicate-based insulating products. The results obtained show that some of the compositions tested have potential applications in the field of passive fire protection as insulating compartmentalization elements. In the tests, fire resistance levels similar to or better than some commercial products were achieved.

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