

## QUANTITATIVE DETERMINATION OF THE AMORPHOUS AND CRYSTALLINE PHASES OF CERAMIC MATERIALS USING THE X-RAY DIFFRACTION TECHNIQUE

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Fly and bottom ashes are by-products originating from the combustion of coal in thermoelectric power stations. The fly ash residue has been used in the cement industries, while the bottom ash up to now has no industrial applications. The latter residue represents almost 50% of the total generated by the thermoelectric plants. The physical, chemical and mineralogical characteristics of bottom ash are compatible with many raw materials used in the ceramic tile industry, which indicates the possibility for the partial or total substitution of these raw materials with this by-product.

Ceramic materials have properties defined by their chemical and microstructural composition. The quantification of the amorphous and crystalline phases is a fundamental stage in the determination of the structure, properties and applications of a ceramic material. Within this context, this study's aim is the quantitative determination of the amorphous and crystalline phases of the ceramic materials developed with addition of coal bottom ash, using the X ray diffraction technique, through the methods proposed by Rietveld<sup>[1]</sup> and Ruland<sup>[2]</sup>. For the formulation of the ceramic mixtures a {3,3} simplex-lattice design was used, giving ten formulations of three components (two different types of clays and coal bottom ash), Table 1.

SAMPLES	RAW MATERIALS		
	CLAY 1 (%)	CLAY 2 (%)	BOTTOM ASH (%)
M1	47,00	41,00	12,00
M2	23,00	41,00	36,00
M3	23,00	65,00	12,00
M4	39,00	49,00	12,00
M5	31,00	57,00	12,00
M6	23,00	57,00	20,00
M7	23,00	49,00	28,00
M8	31,00	41,00	28,00
M9	39,00	41,00	20,00
M10	31,00	49,00	20,00

Table 1. Formulations of the ceramic materials with addition of coal bottom ash.

The crystalline phases identified in the ceramic materials after sintering at 1150oC during two hours are: quartz ( $\alpha$ -SiO $_2$  – JCPDS<sup>[3]</sup> 05-490), polymorph of SiO $_2$  (SiO $_2$  – JCPDS<sup>[3]</sup> 76-912), tridymite (SiO $_2$  – JCPDS<sup>[3]</sup> 75-638), mullite (Al $_{2,35}$ Si $_{0,64}$ O $_{4,82}$  – JCPDS<sup>[3]</sup> 15-776) and hematite (Fe $_2$ O $_3$  – JCPDS<sup>[3]</sup> 13-534), Figure 1.

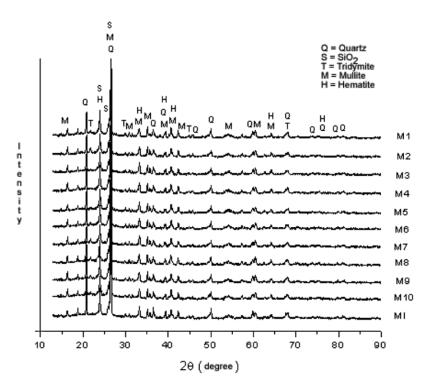


Figure 1. X-ray diffraction of the ceramic materials after sintering.

Table 2 shows the quantitative determination of the amorphous and crystalline phases of the ceramic materials. The proposed methodology for the use of the Rietveld method for the quantification relating to crystalline phases together with the Ruland method used for the determination of the crystallinity of the materials was shown to be adequate and efficient.

SAMPLE	AMORPHOUS PHASE (%)	CRYSTALLINE PHASES (%)
M1	88,10	11,90
M2	77,84	22,16
M3	74,28	25,72
M4	78,18	21,82
M5	70,71	29,29
M6	79,07	20,93
M7	84,54	15,46
M8	87,29	12,71
M9	83,05	16,95
M10	76,47	23,53

Table 2. Quantitative determination of the amorphous and crystalline phases of the ceramic materials.

## **REFERENCES**

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