

## FORMULACIÓN Y CARACTERIZACIÓN DE PARTES CERÁMICAS PARA RECUBRIMIENTOS A PARTIR DE PRODUCTOS DE COMBUSTIÓN DE CARBÓN MINERAL



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### PALABRAS CLAVE

Carbón, baldosas cerámicas, desperdicios de carbón, productos de combustión del carbón (CCP).

El uso de carbón como combustible en diversos procesos industriales, tales como algunos de los que se llevan a cabo en: termoeléctricas y la siderurgia, genera cantidades importantes de desechos o subproductos, denominados productos de combustión del carbón (CCP). Por tanto, se estudian alternativas viables para la correcta eliminación o aplicaciones tecnológicas de este subproducto con o sin la adición de fases de refuerzo con el fin de reducir los impactos ambientales. Entre estas aplicaciones, un posible enfoque es el uso de CCP para la fabricación de baldosas cerámicas, ya que estos residuos son químicamente similares a varias cerámicas. Por lo tanto, este trabajo tuvo como objetivo evaluar algunas propiedades físicas y mecánicas de los PCC para observar la viabilidad de fabricar piezas cerámicas a partir del producto de la combustión del carbón. Los resultados confirmaron el potencial y la viabilidad de utilizar CCP para componer completamente la matriz de baldosas cerámicas.

## FORMULATION AND CHARACTERIZATION OF CERAMIC PARTS FOR COATINGS MADE FROM THE PRODUCTS OF COMBUSTION OF MINERAL COAL

### KEYWORDS

Coal, ceramic tiles, coal waste, products of coal combustion (CCP's).

### ABSTRACT

The use of coal as fuel in various industrial processes such as some of thermoelectric and steel industries generates relevant amounts of waste or by-products, called coal combustion product (CCP's). Therefore, viable alternatives for the correct disposal or technological applications of this by-product are studied with or without the addition of reinforcement phases in order to reduce the environmental impacts. Among these applications, one possible approach is the use of CCP's for the manufacture of ceramic tiles, since these residues are chemically similar to several ceramics. Therefore, this work aimed to evaluate some physical and mechanical properties of CCP's to observe the feasibility of manufacturing ceramic parts from the product of



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coal combustion. The results confirmed the potential and feasibility of using CCP's to completely compose the ceramic tiles matrix.

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## 1. INTRODUCTION

The use of waste from the most diverse industrial activities is recognized as essential for the preservation of the environment, in addition to reducing material costs and contributing to the practice of sustainable development [1]. In addition, when these residues are not reused, they go to deposits and landfills, which can cause contamination by leaching, occupying large spaces that could have other purposes [1].

Due to industrial growth, which has led to an increase in energy consumption, the use of coal-fired power plants, an energy source widely used in countries such as India, China and the United States, has been growing at a great rate [1,2].

During the burning of mineral coal in thermoelectric plants, a large amount of various types of waste and products are generated, called Mineral Coal Combustion Products or CCP's, which cause concern due to the presence of heavy metals and others potentially harmful elements, such as sulfur. Currently, such waste have been the subject of many studies, as they contain ashes whose chemical composition is similar to that of some ceramic products [1,2].

In order to study the applicability of these products, researches are carried out, contributing to the practice of sustainable development, in addition to allowing the formulation of new materials.

Although there are several studies with the incorporation of some type of coal ash as a reinforcement phase in ceramic materials, this work aims to process and characterize ceramic parts made from coal combustion products

(CCP's) for application where ceramic materials are required.

The CCP's can be defined as being all the ashes of the mineral coal in a single residue.

## 2. EXPERIMENTAL

### 2.1. Manufacture of ceramic specimens

After selected, the material went through an oven drying process at 110 °C for 24 h and was classified by sieving to obtain a particle size smaller than 200 mesh (75 µm). Subsequently, water (binder) was added in the proportion of 5% by volume in order to improve the processing conditions and consistency of the paste, in addition to providing the samples before the sintering with sufficient mechanical resistance for handling the piece.

After adding the binder, the paste was mixed manually until the entire mass had a uniform moisture throughout the volume.

To carry out this work, eight specimens (codified as CP-C1 to CP-C8) were manufactured using a circular mold with a diameter of 18.9 mm and the height 0.9 mm applying to the paste a uniaxial compression by 7 MPa. This methodology was experimentally adopted by Pinheiro [2]. After compaction, the samples were dried at 130 °C for a period of 24 h.

The sintering process was carried out in a Linn Elektro Therm electric furnace at the Ceramic Materials Laboratory belonging to the Metallurgical and Materials Engineering Department at the Federal University of Ceará. Specimens were sintered at 1170 °C for 60 min with a heating rate of 5 °C/min.



This procedure was determined experimentally by Pinheiro [2].

## 2.2. Characterization of ceramic specimens and raw materials

### 2.2.1 Chemical and mineralogical analysis

#### 2.2.1.1 X-Ray Diffraction

For the determination of the crystalline phases present in the sintered samples and the raw material they were characterized by X-ray diffraction. The measurements were carried out using a Philips® X'Pert Pro diffractometer with a radiation of CuK $\alpha$  ( $\lambda = 0.1542$  nm). The  $2\theta$  angle ranged from  $10^\circ$  to  $70^\circ$ . This range is recommended by Pinheiro who used the Rain Forest Granite tailings for the manufacture of ceramic tiles [2].

The phases were identified using the X'Pert High Score Plus program and quantified by Rietveld method.

#### 2.2.1.2 X-Ray Fluorescence

The CCP's already duly sieved until reaching a particle size of less than 200 mesh were submitted to chemical analysis at the Laboratory of X-rays of the Department of Physics at the Federal University of Ceará, by X-ray fluorescence (FRX), in Rigaku equipment, model ZSX mini II.

#### 2.2.1.3 Scanning Electron Microscopy

In order to analyze the microstructure of the samples and obtain information about the particle morphology, the samples were initially covered with a gold layer of approximately 20 nm using a vacuum deposition system. Then, the micrographs of the material were obtained from a FEI Quanta 250 Scanning Electron Microscope from the

Research and Technology in Welding Laboratory - LPTS (DEMM / UFC) with varying magnifications.

In addition, the specimens were analyzed chemically by a Energy Dispersive Spectroscopy (EDS) detector coupled to the SEM in order to obtain the specific chemical compositions in the material.

## 2.3. Physical characterization

### 2.3.1 Mass variation

The percentage variation in the mass of the samples, based on the chemical reactions that occur inside of them during the sintering process, is given by Equation 1:

$$MV(\%) = \left( \frac{m_s - m_0}{m_0} \right) \times 100\% \quad [1]$$

Where  $m_0$  is the initial mass and  $m_s$  is the mass after sintering.

### 2.3.2 Linear retraction of burning

The linear shrinkage test allows to evaluate the linear dimensional variation of specimens after submitted to the sintering process. This information is essential for the manufacture of the ceramic parts. In this work, the linear shrinkage (measured through the diameter of samples) from sintering at  $1170^\circ\text{C}$  was studied. According to NBR 9623 [3] linear shrinkage can be determined using Equation 2:

$$\Delta X(\%) = \left( \frac{x_f - x_0}{x_0} \right) \times 100\% \quad [2]$$

Where  $X_f$  is the final diameter of the specimen and  $X_0$  is the initial diameter.

### 2.3.3 Water absorption



Water absorption is a key factor that affects the durability of ceramic materials. According to the ABNT 13817 standard [4], ceramics can be classified according to the value of water absorption in order to determine their application. In addition, it can be said that ceramic coatings of low water absorption have high mechanical resistance. To determine the water absorption of the specimens, the samples were dried in oven at 110 °C for 24 h and then immediately weighed. After this stage, the specimens were immersed in a container containing distilled water for 24 h. Then, the specimens were slightly dried with a flannel and then weighed again to check their wet mass. According to NBR 13818 [5], water absorption is expressed as a percentage by Equation 3:

$$Wa = \frac{M_{wet} - M_{dry}}{M_{dry}} \times 100\% \quad [3]$$

Where  $M_{dry}$  is the mass of the dry specimen and  $M_{wet}$  is the mass of the wet specimen (saturated).

#### 2.3.4 Apparent specific mass

The tests of specific mass and apparent porosity reinforce the result of the water absorption test, as these tests show the “degree” of densification, consequently, the mechanical properties of the material.

The apparent specific mass was determined using Equation 4, where  $m_w$  is the wet mass,  $m_d$  is the dried mass,  $m_i$  is the submerged mass (for the samples) and  $\rho$  is the water density. The apparent specific mass is obtained through the following Equation [4]:

$$ASM = \frac{m_d}{m_w - m_i} \rho_{water} \quad [4]$$

#### 2.3.5 Apparent Porosity

In order to calculate the apparent porosity (PA) of the specimens, right after the water absorption test, the samples were weighed immersed in distilled water, 24 h after the moment of immersion. The apparent porosity is expressed as a percentage according to the following Equation number 5 [7].

$$PA (\%) = \left( \frac{m_w - m_D}{m_w - m_I} \right) \times 100\% \quad [5]$$

Where  $m_w$  is the wet mass,  $m_D$  is the dried mass and  $m_I$  is the submerged mass of the samples.

## 2.4. Mechanical Characterization

The methodology adopted for the mechanical tests is the same used by Rajamannan et al. [8] who used for each test a batch of 3 (three) specimens. Considering the desired application (ceramic tiles), Vickers microhardness was used.

### 2.4.1 Vickers microhardness

To determine the microhardness of the specimens, which is the resistance to localized plastic deformation, the Vickers microhardness test was used. To the assay measurements, a SHIMADZU HMV microdurometer was used in the Materials Characterization Laboratory (LACAM) belonging to the Department of Metallurgical and Materials Engineering at the Federal University of Ceará. An indentation charge of 980.7 mN for 10 s was applied.

### 2.4.2 Tensile strength by diametrical compression

The samples were tested individually using a universal mechanical testing equipment EMIC 100 kN, Cell - TRD 28 of the Laboratory of Mechanical Testing - LEM (DEMM / UFC).





The material were put to rest along a generatrix and adjusted in order to guarantee the coupling of the specimen with minimal compression. Then, the load of the equipment was applied continuously with a speed of 1 mm/min until the fracture of the samples and the maximum loads for fracture were recorded [2].

### 3. RESULT AND DISCUSSION

By visual analysis (Figure 1), it was possible to observe the surface aspect of the samples with dark coloring and characteristic brightness indicating vitrification, filling of the pores of the material by a glassy phase. The surface brightness obtained in the ceramic samples with the sintering, as well as the reddish brown color observed, results from the presence of iron oxide in the residues of the mineral coal used in the specimens manufacturing [8]. During the sintering process, the hematite ( $\text{Fe}_2\text{O}_3$ ) present in the compacted powder of the residue is thermally activated, moving atomically by diffusion in the ceramic samples reducing  $\text{Fe}_2\text{O}_3$  in  $\text{FeO}$ .

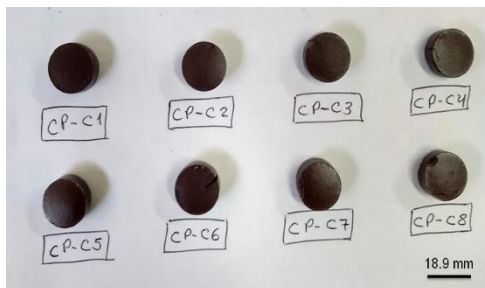


Figure 1. Top view of sintered samples manufactured from CCP's.

#### 3.1. X-Ray Diffraction

The XRD results for the CCP's are presented in the diffractogram in Figure 2. X-ray diffraction analysis revealed a quartz diffraction pattern ( $\text{SiO}_2$ ), mullite ( $\text{Al}_4\text{SiO}_8$ ), gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ).

It was observed that in approximately  $2\theta = 31.12^\circ$  an overlapping diffraction peaks corresponding to the phases ( $\text{SiO}_2$ ) [101] and ( $\text{Al}_4\text{SiO}_8$ ) [210] occurred. Also, the diffractogram shows peaks of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  and  $\text{Fe}_2\text{O}_3$ . It was revealed that the crystalline structures of quartz and mullite are hexagonal and orthorhombic, respectively.

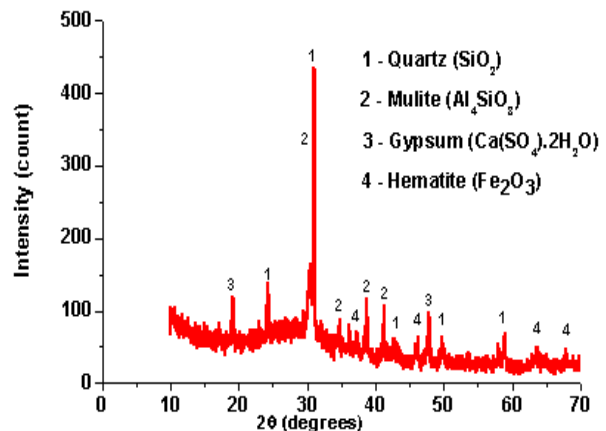


Figure 2. CCP's diffractogram.

Other phases identified in the diffractogram were gypsum and hematite. The highest intensity of the hematite peak ( $\text{Fe}_2\text{O}_3$ ) [104] was found at approximately  $2\theta = 39.1^\circ$ . The amorphous halo between  $2\theta = 10^\circ$  and  $2\theta = 50^\circ$  and  $60^\circ$  shown in the diffractogram, indicates a large amount of amorphous material, referring to the organic matter of coal [9]. The percentage of the identified crystalline phases is shown in Table 1.

The result showed in this table is close to that found in the work developed by Sabedot et al. [10] where the phases identified in the diffractogram were silica and mullite. The results prove that the CCP's components are phases of great potential for incorporation as a reinforcement phase in ceramic specimens.

**Table 1.** Semi-quantitative analysis of the phases present in the CCP's.

Phases	[wt %]
Mullite	54.5
Quartz	37.5
Gypsum	4.0
Hematite	4.0

### 3.2. X-Ray Fluorescence

The results of X-ray fluorescence analysis indicate that the main components of the products obtained from the combustion of mineral coal are: silicon (59.17%), aluminum (13.17%), iron (10.74%), potassium (6.11%) calcium (4.97%), titanium (2.91%) and sulfur (0.84%). Therefore, it is possible to observe a large amount of raw materials (Si, Ca and Al) suitable for the manufacture of ceramic pieces. This result is shown in Table 2.

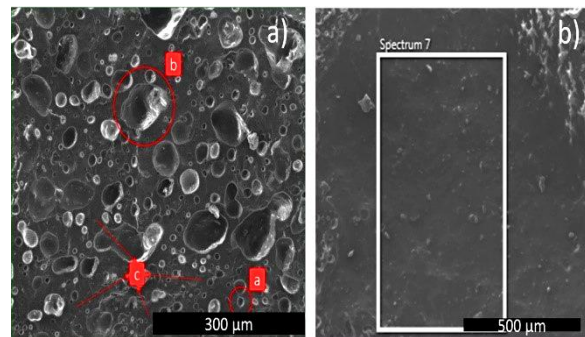
**Table 2.** Chemical composition of the analyzed CCP's.

Components	[wt. %]
Si	59.17
Al	13.17
Fe	10.74
K	6.11
Ca	4.97
Ti	2.91
S	0.84
Other elements	2.09

### 3.3. Analysis of the structure by SEM

From the analysis carried out by SEM on the cross section of the samples, it was possible to observe, the presence of dispersed pores of varying dimensions and large amounts of oxides in the middle of a dense vitrified matrix. The pores (indicated by letter *a* in Figure 3a) have an approximately rounded appearance of small diameters, in addition to a dark interior indicating an empty position on the surface of the sample. This porosity is

formed mainly by the trapping of gases that occurs due to the bloating effect studied in literature [8]. It is said that the hematite reduction reaction ( $\text{Fe}_2\text{O}_3$ ), which at high temperatures activates the atomic diffusion of iron to neighboring regions, produces as a gaseous phase, oxygen. This is the main generator of bubbles, originating pores in the sintered specimens. As a result, it is expected that, although the specimens had a good surface appearance, the volume of pores present inside the ceramic matrix should give less specific mass to the ceramic body, contributing negatively to the mechanical resistance, since these pores behave as stress concentrators.



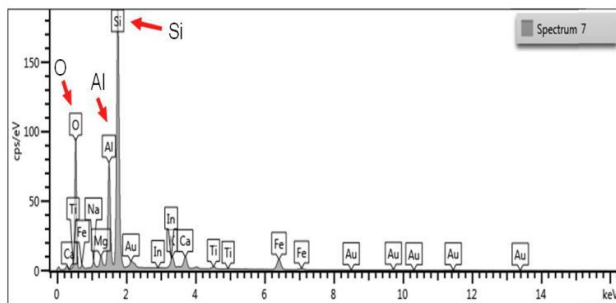
**Figure 3.** SEM images of the cross section of the sample a) after the manufacturing and b) ceramic matrix.

The oxide inclusions (indicated by letter *b* in Figure 3a), presented irregular morphology, probably because the ash is formed by oxides of different compositions [2] and more intense brightness at the ends due to the dielectric character that acts as an electrical insulator making it difficult to apply the uniform gold coating and conduct the electrons necessary for the formation of this image.

The visual absence of particle agglomerates that were resulted from the processing of the ceramic material indicates complete sintering of the samples. In addition, the smooth and low-roughness aspect of the ceramic matrix (indicated by letter *c* in Figure 3a), without the presence of visible cracks or other significant

defects, as shown in Figure 3b, highlights the matrix vitrification which is the state of desired sintering to ensure better mechanical resistance to ceramic parts.

The results of chemical composition obtained by EDS semi-quantitative analysis on the region highlighted by a white rectangle in Figure 3b is shown in Figure 4. More relevant proportions of oxygen, silicon and aluminum were detected.



**Figure 4.** Result of the energy dispersive spectroscopy (EDS) analysis performed on the ceramic matrix of the sintered specimens.

In addition to some heavy metals, very few of the metallic elements detected can be incorporated into the CPP's structure due to the ability of CPP's or coal ash to bind many heavy metal ions through complexation [11,12]. Other elements such as silicon can originate from impurities [13]. These elements compose the glassy matrix of the ceramic parts. Thus, despite the present porosity, the ceramic parts must have good mechanical performance, considering the linear relationship between the proportion of mullite and the mechanical resistance of the ceramic parts, in addition to the relatively high hardness conferred by quartz [2].

In Table 3, the results obtained for the circular samples (CP-C1 to CP-C8) regarding the mechanical and physical tests, were summarized. The abbreviations presented in

Table 3 are: tensile strength by diametrical compression (TSDC), mass variation (MV), linear retraction of burning (LRB), water absorption (WA), apparent porosity (AP), apparent specific mass (ASM), standard deviation (SD). In this table some of the results obtained for the tests carried out are not shown due to samples defects (irregularities and cracks).

### 3.4. Mass Variation(MV)

The percentage of mass variation showed an average of 10.38%. Compared to the results obtained by Pinheiro [14], the average of 10.38% of mass loss reinforces the tendency to increase this characteristic with the increase in the proportion of CCP's in the composition of the ceramic matrix, which in the case of the present work was 100%.

### 3.5. Linear Retraction of Burning (LRB)

The average of 4.02% measured for LRB on the diameter of samples is due to the fact that the CCP's have chemical elements with a low melting point, which, when subjected to high temperatures are eliminated. From the above, the glass phase will fill the volume in some pores left by the evaporated elements, causing a linear retraction of the ceramic sample. The free sulfur present in the CCP's is eliminated at approximately 445 °C Pinheiro [15] defined a trend of increasing linear shrinkage with the increase in the concentration of CCP5] defined a trend of increasing linear shrinkage with the increase in the concentration of defined a trend of increasing ceramic reinforces the tendency to increase this characteristic with the parallel between the



results of the two studies, since in this case the linear retraction of burning was determined in terms of the diameter of specimens with a circular section, while in the other work; the retraction was defined linearly in bodies of

prismatic section test. This test affirms the degree of vitrification of ceramic parts and this value is related to the mechanical properties.

**Table 3.** Results obtained for the physical and mechanical characterizations of the specimens manufactured from the CCP's.

Sample	Microhardness [HV]	TSDC [MPa]	MV [%]	LRB [%]	WA [%]	AP [%]	ASM [g/cm <sup>3</sup> ]
CP-C1	658.70	---	- 10.05	---	---	---	---
CP-C2	637.40	---	- 9.72	3.17	0.24	0.38	1.61
CP-C3	608.60	---	- 12.09	---	---	---	---
CP-C4	---	17.95	- 8.97	3.70	0.00	0.00	1.67
CP-C5	---	13.27	- 10.34	3.70	0.24	0.40	1.69
CP-C6	---	---	- 9.81	---	---	---	---
CP-C7	---	---	- 10.55	4.76	0.00	0.00	1.79
CP-C8	---	15.60	- 11.48	4.76	0.00	0.00	1.74
Average	634.90	15.61	- 10.38	4.02	0.09	0.16	1.70
SD	89.76	2.34	1.00	0.71	0.13	0.21	0.07

### 3.6. Water Absorption (WA)

These results are due to the fact that the CCP'S contain large amounts of feldspars (responsible for the densification of the material). Luz [16] states that feldspars contribute to reducing the water absorption of porcelain tiles by assigning them the desired properties.

The average of the results obtained was 0.09% with a standard deviation of 0.13%, which is similar to the water absorption of porcelain tiles, according to the NBR 13818 standard [5], which establishes that the water absorption is  $\leq 0.5\%$  for products of this type.

### 3.7. Apparent Specific Mass (ASM)

In the apparent specific mass tests an average of 1.70 g / cm<sup>3</sup> was obtained. In the sintering

process, the filling of the pores increases the density of the samples, making it possible to relate the densification behavior with the increase in the sintering temperature, since, with this, the greater the amount of glass phase penetrating and filling the pores the greater the resistance of the ceramic body [17]. In addition, elements such as Fe, K, Na, Ca and Mg contribute to the densification of ceramic materials [8].

### 3.8. Apparent Porosity (AP)

The average of the apparent porosity measured was 0.16%. The apparent porosity represents the proportion of open pores contained in the specimen, showing the degree of sintering of the ceramic samples. This porosity has a direct influence on the mechanical resistance of the samples,





considering that the pores are stress concentrating points that facilitate crack propagation, with this, in general, the lower the percentage of porosity, the greater the resistance of the ceramic body. Possibly, the pores are not interconnected and are closed pores, since the apparent porosity only takes into account the number of open pores [18].

The closed pores can be caused by the closing of open pores, due to the evolution in sintering or they can be caused by the evolution of gases in the solid phase, which are unable to leave the material. The pores tend to take on a spherical shape.

### 3.9. Vickers microhardness

The results of Vickers microhardness test show an average of 634.90 HV. The high microhardness values obtained, which represents high resistance to localized plastic deformation, must be due mainly to the levels of aluminum oxide ( $Al_2O_3$ ) in mullite phase that forms an external layer that glazes the surface of the ceramic samples. The above may have occurred by diffusion, since the pores are made up of gases [5,20] from the formation of the mullite as analyzed by Pinheiro that chemically evaluated ceramic surfaces manufactured with the addition of CCP's [2]. One of the most frequent phases in traditional ceramics, mullite is formed from the combination at high temperatures of silica ( $SiO_2$ ) and alumina ( $Al_2O_3$ ) forming the crystalline phase  $3Al_2O_3 \cdot 2SiO_2$  [19], giving good mechanical resistance properties, surface hardness and refractoriness. In addition, the high microhardness must also be associated with the presence of iron oxide ( $Fe_2O_3$ ) [20] which had the diffusion, to the surface,

activated by the high temperatures of sintering.

### 3.10. Tensile Strength by Diametrical Compression(TSDC)

From the results obtained, with an average TSDC of 15.61 MPa and standard deviation of 2.34 MPa, it is possible to notice that the good mechanical resistance of the produced ceramic specimens is not limited to the surface, where there are higher concentrations of mullite and iron oxide, to which the high Vickers microhardness values shown in the Table 3 were attributed.

## 4. CONCLUSION

According to the tests presented in this work, ceramic pieces made from mineral combustion products (CCP's) are very viable. It is worth noting that the results may differ from those presented depending on the origin of the CCP's.

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