DEGRADATION OF RED CERAMIC WITH INCORPORATED OF PETROLEUM WASTE

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Abstract. The red ceramics bodies of the district of Campos-RJ it's have fast degradation when be at environmental construction. With the intention of reducing the degradation in these pieces, its incorporated petroleum waste in the red ceramic mass. The petroleum waste is a solid fuel, derived of petroleum, of black color and shape approximately of sand, obtained as by-product petroleum's distillation, in a process designated thermal "cracking". The material when fired at 800ºC was obtained smaller failure probability by Weibull distribution of the red ceramic pieces not submitted to the degradation. The mixtures of the ceramic specimens molded with clayey soil and waste content varying from 0 to 1,5%. After drying under 110ºC and firing to 800ºC and 900ºC, samples were submitted to flexural testing and physical properties were then evaluated, used Weibull distribution to analysis the material failure probability. The results presents improvements in the samples probability failure incorporate with petroleum waste.

Introduction

The durability questions of today takes a major Featured in the materials characteristics. Given a better performance lifelong of ceramic products, introduced the petroleum coke to the clay material [1].

Petroleum coke is a solid fossil fuel, petroleum derivative, black and approximately granular form or type "needle", which is obtained as a byproduct of petroleum distillation (at the bottom of the distillation column), a process designated "cracking "heat. This product accounts approximately 5% to 10% of the total oil entering the refinery [2].

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The Weibull distribution [4, 5] describes the probability of the material fails when submitted to a given voltage level. This probability of failure of the material is submitted to tension σ is provided by probability density function Weibull. The equation depicts the Weibull distribution: [Eq.1]:

$$F(\sigma) = 1 - \exp\left\{ -\left(\frac{\sigma - \sigma_0}{\sigma_R} \right)^m \right\} \tag{1}$$

Where σ_0 is the strain before which the material will not fail, σ_R is a referential tension value, corresponding to 0.632 probability of failure of the material is the Weibull modulus, related to the dispersion of the measurements.

In the Weibull distribution can be observed the behavior of fractures of ceramic red. This distribution connects the material failure and presence of defects which weaken. In direction, this work aims to characterize and observe how the degradation influence the technological properties of red ceramics incorporated with petroleum coke.

Materials and Methods

Materials

For this study, we used the following materials: clay used to produce red ceramics from the municipal district of Campos-RJ and Coke, of the distillation of Petroleum.

Methods

Characterization tests

Quantitative Chemical Analysis

These tests were carried out in the laboratory characterization LECIV/CCT/UENF. The samples were sieved on #200 mesh (0.075mm). The equipment used was the florescence spectrometry Philips model PW 2400, on 10g of pressed powder pallets. The loss of ignition was obtained by determining the weight difference between samples fired at 950ºC and dried at 110ºC.

Differential Thermal Analysis

The samples were carried out to Polymer Laboratory (LAMAV/CCT/UENF) for the universal equipment, V2.6D TA Instruments model and obtained the curves DTA operating nitrogen atmosphere at a heating rate of 10°C/min.

Degradation

The degradation was carried out at laboratory equipment degradation to be evaluated after the changes in the technological properties (Figure 1). This equipment submit the samples to time-controlled continuous leaching of hot distilled water (70ºC) or cold water (21ºC), with temperature control of the sample chamber, and also submits the samples to freezing up to -4ºC. Additionally, samples are subjected to vapor atmosphere every time the hot water cycle starts. The samples were submitted to degradation in equipment during 420 hours.

Fig. 1 – Laboratory equipment degradation.

Technological Tests

Sample Preparation

Samples for technological tests were prepared with clay and four different waste contents (i.e. 0, 0.5, 1.0 and 1.5%). The coke powder was sieved through #20 mesh (0.85mm). The batch mixture was performed on dried material and the extrusion moisture was calculated by Eq.2 [6]:

$$Wext. = \frac{LL}{2} + 2\% \qquad (2)$$

Where the LL is the liquid limit and $W_{ext.}$ is the extrusion moisture content.

After mixed, the samples were taken to the laminator followed by the extrusion. The ceramic bodies were, then, molded in prismatic shape of 11.0 cm x 2.7cm x 1.7cm. The process of drying and firing has involved temperatures of 110ºC, 800ºC and 900ºC, in an electronic oven. After firing, the ceramic bodies were submitted the tests according to [6] and also to a three loading point flexural strength test according to standard procedure [7]. The obtained results represent the average of 25 determinations. The three loading point flexural strength test was carried out with loading rate of 0.1mm/min with the distance of 9.0 cm between supports.

Results and Discussion

Characterization tests

Table 1 shows the chemical compositions of clay and coke.

Tab. 1. Chemical analysis of the raw materials:

raw materials	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	K_2O (%)	TiO_2 (%)	SO_3 (%)	CaO (%)	MnO (%)	V_2O_5 (%)	NiO (%)	C (%)	ZrO_2 (%)	LoI (%)
Clay	44.19	39.77	8.9	2.92	1.54	1.41	0.50	0.72	0.01	----	----	0.03	10.99
Coke	0.94	----	9.9	----	1.54	3.1	0.74	----	0.40	1.21	82,17	----	----

Table 1 shows the chemical composition and the LoI of the raw materials. The chemical composition of the clay is typical of a kaolinite-based material with low amounts of alkaline oxides and relatively high amount of Al_2O_3 (39.77%). The high percentage of LoI (10.99%) indicates an elevated fraction of clay minerals. The chemical composition of the coke, in addition to C (82.17%), confirms the calorific power potential of the coke. The amount of Fe_2O_3 in both raw materials is responsible for the reddish color of the specimen after firing.

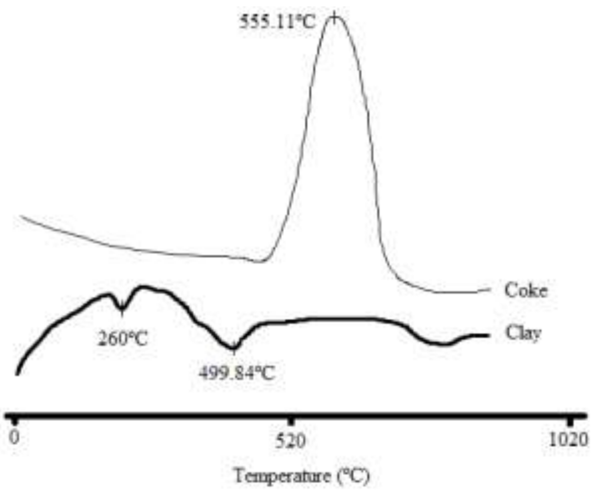
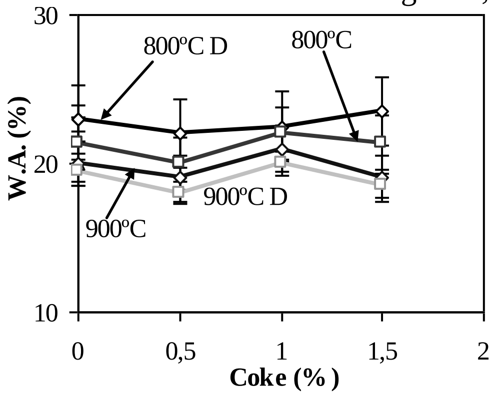


Fig. 2. ATD curves of the clay and coke.

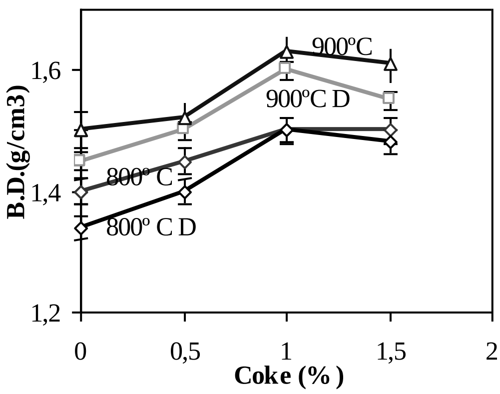
The Fig. 2 shows the DTA curves of clay and coke. The first endothermic peak in clay, from 260ºC, can be related to Al and Fe hydroxides decomposition such as gibbsite and goethite. The presence of both aluminum and iron hydroxides is undesirable since they demand an additional energy input for decomposition [10]. Moreover, the formation of the corresponding oxides, Al_2O_3+Fe_2O_3>83% (see Tab.1), increases the refractoriness of the ceramic bodies as well as their loss on ignition. The second endothermic peak, from 499.84ºC, due transformation partial hidden by the kaolinite dehydroxylation [10]. The Fig. 2 also shows the DTA curve of the coke. It can be observed that an exothermic peak occurred due to firing of fixed carbon in the coke (see Tab.1). Figure 2 also shows the DTA curve of the coke. It can be seen that there was an exothermic peak due to firing of fixed carbon (82.17%) in the coke (see Tab.1). This can be beneficial to incorporate coke red ceramic, saving energy required to fire the ceramic pieces, and also in reducing the firing time according to [11].

Technological properties

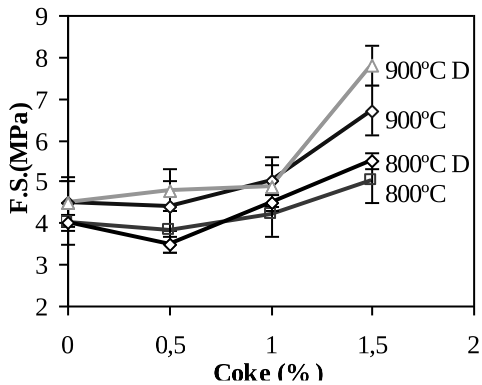
After the firing tests were carried physic-mechanical mixtures of 0% up to 1.5% of petroleum coke and the results are shown in Figure 3, before and after degradation.



(a)



(b)



(c)

Fig.2. Technological properties of the mixtures after firing and before and after degradation: (a) water absorption; (b) bulk density; (c) flexural strength.

Analyzing the results of Fig. 2(a), comparing ceramic fired at 800°C before and after accelerated degradation process in the laboratory by incorporating 0.5% to 1.5% of petroleum coke, there is an increase in water absorption. And at 900°C there is no major changes due to the degradation this property, because the standard deviations are coincident. In the bulk density (Fig. 2 (b)) shows that this property degradation decreases for pieces fired at 800°C. The same goes for pieces ceramics at 900°C. For the flexural strength (Figure 2(c)) was not observed significant differences among pieces intact and degraded at 800°C. As for the pieces with firing temperature to 900°C the addition of petroleum coke change strength after degradation. However, larger time is required for the equipment degradation to inferences regarding the effect of degradation, so it was used Weibull statistics to evaluate the mechanical strength of the ceramic pieces incorporated with coke.

Figure 3 shows the Weibull diagram for the samples with 0% petroleum coke burned at different temperatures. From this diagram is determined by the Weibull modulus **m**, which is also considered as the value from which there is a risk of rupture and characterizes the dispersion of data from stress rupture test pieces [3].

The value of **m** is in a valuable standard for the analytical quality of ceramic materials [12]. It’s desired that the value of m is as large as possible. The higher the value of **m**, the higher is the homogeneity of the batch statistical evaluated for the probability of cracks and fissures in the material. With the determination of the Weibull modulus can be evaluated which temperature is the most appropriate for a smaller dispersion of defects as shown in Fig.3.

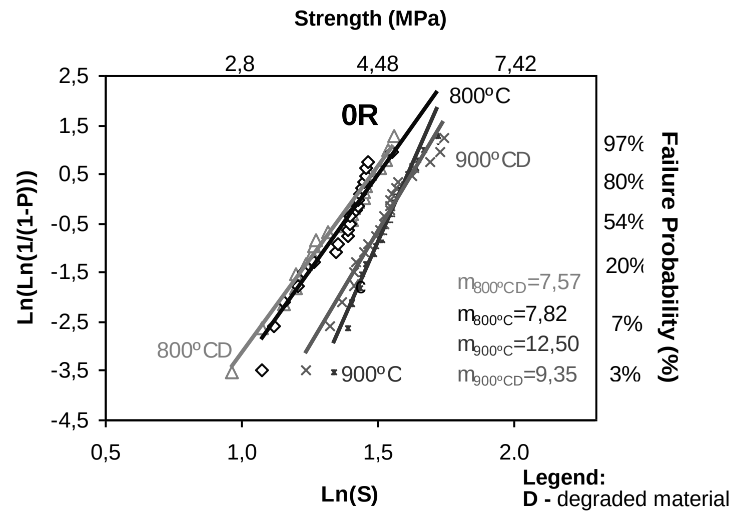


Fig. 3 - Weibull distribution to 0% petroleum coke before and after degradation.

It can be observed that the modules was larger for temperatures of 900°C, before and after the accelerated degradation process in the laboratory, meaning that this group of samples is more homogeneous and less probability to failure. Figure 4 plots, it shows the Weibull diagrams for red ceramic fired at 900°C with different additions of petroleum coke before and after degradation.

Fig. 4 plots the Weibull distribution for the red ceramic fired at 900°C with different additions of petroleum coke, before and after the accelerated degradation process in the laboratory.

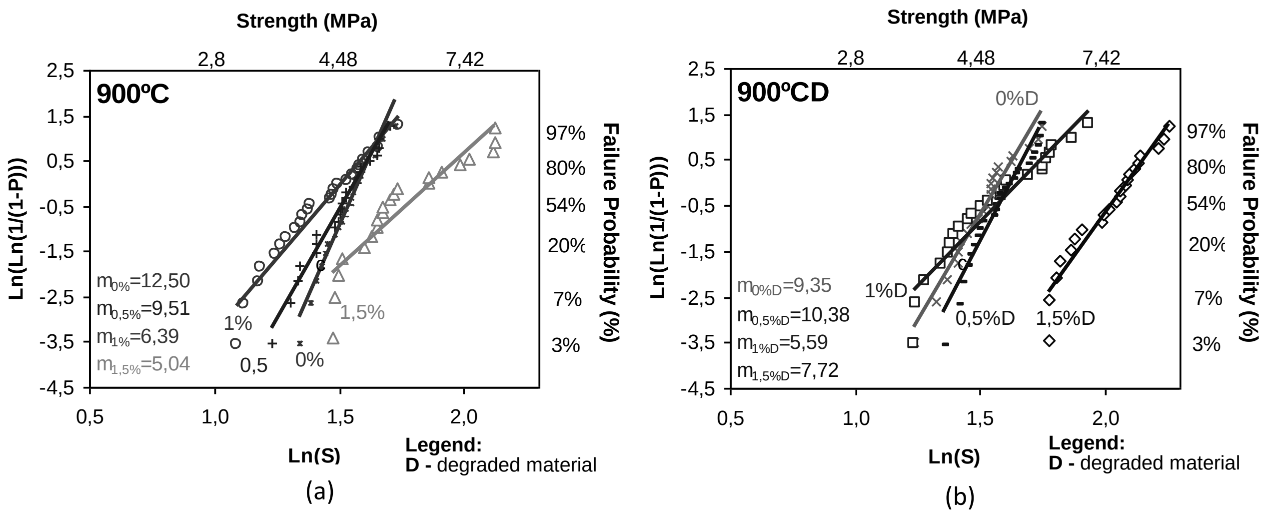


Fig. 4 - Weibull distribution to temperature of 900ºC with different additions of petroleum coke, before (a) and after accelerated degradation process in the laboratory.

Figure 7 (a) shows that the addition of petroleum coke in the clay not contributed to an improvement in Weibull modulus (m). However, it is noted that in Figure 7 (b) the ceramic pieces were submitted to accelerated degradation process in the laboratory is further recommended to add 0.5% of petroleum coke in relation to the other additions, i.e., lower failure probability in degradation, thus more reliable.

Conclusion

The incorporation of 0.5% of coke in piece clay of red ceramic leads to the conclusion that:

Due to the high amount of SiO2 and Al2O3, clay behavior refractory firing, resulting in small variations in the properties at the temperatures used in this work.

Has been observed endothermic peak at 499.84ºC to clay, characterized by partial transformation of kaolinite dehydroxylated. From this peak, no endo or exothermic event occurred according to the DTA. It can be seen that there was an exothermic peak due to firing of fixed carbon (82.17%) in the coke.

With the addition of petroleum coke burned clay at 800°C, observed an increase in water absorption of the pieces after the process of degradation. How additions above 0.5%, there is less variation among the pieces degraded, this also at 900°C.

In ceramic pieces fired at 900°C in water absorption, no significant changes between the intact and degraded. Regarding to flexural strength verified no significant difference between the intact and degraded materials. Except for parts fired at 900°C with the addition of 1.5% of petroleum coke increases the variation between the results.

The higher Weibull modulus (m = 10.38) was observed in ceramic with 0.5% of petroleum coke at 900°C, enhancing the Weibull modulus of the material degraded.

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