

BENTONITE CONTAINING PELLETS MECHANICAL PROPERTIES VARIATION CAUSED BY OILY WASTES INCLUSION

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ABSTRACT

Oily wastes are usual byproducts generated by oil industry. New and stricter environmental regulations had established that a permanent and sound solution must be found to these residues storage. Bentonite powder has been used to microencapsulate oily residues. However this new material containing residues cannot be buried or just piled up. The new material was found to be useful for the ceramic industry, therefore mechanical properties must be known. Several 10x10 mm cylindrical pellets containing residues amounts from zero up to 20% of the mixture were manufactured by forming pressure around 25 MPa and fired at 950 °C. Due to its geometry the only test that can be performed in order to determine strength and fracture limits is Brazilian Disk, which yields tensile strength by means of diametrical compression. A total of 25 pellets are tested and ultimate compressive strength and linear shrinkage for the different compositions presented. The effects of different contamination levels over fracture properties are discussed. A brief discussion on the uncertainty measurement is also presented.

KEYWORDS

Contact stresses, compressive strength, oily wastes, diametrical compression, ceramics properties

INTRODUCTION

Campos dos Goytacazes County, located at State of Rio de Janeiro (Brazil), holds extensive clay quarries presenting kaolinite as its main clay mineral [1]. Local ceramic plants exploit these clays as raw material for producing mostly hollow construction bricks and roofing tiles. On the other hand, Campos County has the largest Brazilian oil basin, which is responsible for more than 75% of the national oil production. However, during the process of oil extraction, transportation and storage, great amounts of toxic wastes are generated, among them an oily sludge residue. This waste in particular is sealed in tanks and brought to land to be mixed with an encapsulation substance, organophillic bentonite. Yet less harmful, the resulting material cannot be simply disposed in landfills or just piled up. As a consequence, efforts have been made to find a permanent solution for the management of the referred waste, without causing major risks to the environment.

The Advanced Materials Lab, from Northern Fluminense State University (UENF) has tried to make the oil sludge waste inert by adding it in clayey ceramic pastes and firing it. Some of the most environmentally aggressive components are expelled during fire process. It is expected that the vitreous phase formed during the firing process completely inactivates the residue, and permits the use of this mixture as raw material for

the local ceramic industry, which will be paid by local oil companies to handle oily wastes. One possible use for this bentonite-oily wastes-clay mixture is as structural clay products. For this purpose, material is submitted to compressive pressures to take shape and density and fired afterwards. This work simulates manufacturing conditions by producing pellets under identical conditions. Important physical properties as ultimate compressive strength and linear shrinkage are determined to verify if the presented mixture is useful and the variations brought by means of different percentages of oily slurry addition.

Testing for mechanical properties present a challenge due to the pellet geometry. The height to diameter ratio is very close to one, eliminating the possibility of compressive testing use. The use of the Brazilian Disk [2,3] test solved the problem of consistent geometrical dimensions [4], but brought another one, namely localized fracture. The solution was to add an elastomeric foundation to the contact region and verify if the new stress field and uncertainty analysis correctly reproduces the test.

EXPERIMENTAL PROCEDURE

The clay sample employed in this work belongs to a quarry from Campos dos Goytacazes-RJ (Brazil). Previews characterization showed that the ceramic mass is kaolinitic, with illite, quartz and gibbsite as main impurities [5]. Chemical analysis revealed that the clay consists basically of SiO₂, Al₂O₃, and Fe₂O₃, the latter conferring a reddish color after firing, and TiO₂, MnO, MgO, CaO, K₂O and Na₂O in smaller amounts. The oil sludge waste chemical composition before bentonite encapsulation in average consists of 21% water, 62% solid material, 16% oil and 1% sulphur [6]. Waste granulometric analysis reveals that this material is essentially sandy silt.

Raw materials were crushed and sieved until the 60 mesh screen fraction passed. Ceramic masses containing clay and 0, 5, 10, 15 and 20wt% waste were prepared (5 each). The masses were mixed and homogenized, and 7wt% water was added in order to provide plasticity. Afterwards, the pastes were formed in a 10mm diameter steel die. The resulting 10 x 10mm cylindrical pellets were dried (110°C for 24h) and fired at 950°C for one hour. Heating and cooling rates have been controlled. Linear shrinkage (ABNT MB-305 [7]) and diametrical compressing strength [2] of the ceramic bodies were determined. Microstructure of the fracture surface was investigated using a DSM 962 Zeiss scanning electron microscope coupled with an Energy Dispersive Spectroscopy device.

A model 5582 INSTRON universal testing machine was used and both contact plates were covered with latex sheet. Crossbar speed was kept 0.5 mm/min for all tests. Specimens were placed between contact plates and a PC displayed a real time load x displacement graphic. At fracture load dropped very visibly, making quite easy to determine the ultimate compressive load. All pellets dimensions, after been fired, were recorded and used to determine individual ultimate compressive strength.

BRAZILIAN DISK TEST (DIAMETRICAL COMPRESSION)

Diametrical Compression Testing

Fired pellets presents an average 10 mm diameter and height. To correctly evaluate compressive strength limit, a compressive test is necessary but the pellets geometry do not allow the traditional compressive testing use, for it requires a height of 2 to 3 times the diameter size [8]. The solution is to use the Brazilian Disk Test, exploring diametrical compression as means to cause specimen fracture failure. In this test load is applied in two diametrically opposing points [9].

Stress Field

It is assumed that fracture initiates at the central point. Stresses acting over the horizontal diameter have the following form [10]:

$$\sigma_x = \sigma_1 = \frac{2P}{\pi Dt} \left(\frac{D^2 - 4x^2}{D^2 + 4x^2} \right)^2 \quad (1)$$

$$\sigma_y = \sigma_2 = -\frac{2P}{\pi Dt} \left(\frac{4D^4}{(D^2 + 4x^2)^2} - 1 \right) \quad (2)$$

$$\tau_{xy} = 0 \quad (3)$$

Where P is the applied load, D is the disk diameter, t is thickness and x is the horizontal position along disk diameter. For crack opening, only σ_x matters, because σ_y is a compressive type of stress. σ_1 component acting over the x direction and under the load line (x = 0) expression (1) is reduced to:

$$\sigma_x = \sigma_1 = \frac{2P}{\pi Dt} \quad (4)$$

Above expression ignores the existence of contact stresses acting near the points where loads are applied. Expression (4) is used in this work to determine Ultimate Compressive Strength (S_{uc}).

LOCALIZED FRACTURE

In the preliminary tests was observed that due to existence of contact stresses plus the fact that the contact area in this case is very small considered the body shape, localized stresses will fracture contact regions, thus altering stress distribution, crack initiation region and rendering useless expression (4) [11,12].

Controlling Localized Fracture

To avoid fracture initiation at the contact areas it was used an elastomeric rubber layer, applied to the contact region, to reduce magnitude of localized acting stresses. In this study latex was used and eliminated the problem. The used layer thickness measured 0.2 mm.

New Stress Distribution At Contact Area

The presence of that rubber layer is modeled as an elastic foundation and its presence alters stress distribution at contact region by increasing contact surface thus loading distribution area. Johnson shows that the new contact pressure distribution is paraboloidal rather than ellipsoidal as given by Hertz theory. Although localized stress field changes, at the center of the disk, where fracture is supposed to have started, no influence is felt, once contact stresses, regardless its shape, are expected to act at no more than up to 0.15D from the contact surfaces [13].

UNCERTAINTY MEASUREMENT

Uncertainty measurements, related to existing errors associated with the measurement system and material properties dispersion, for the proposed tests, are determined as described by ISO standards [14]. For the measurement system, these errors are originated from the load cell (ultimate compressive load) and the caliper (pellets dimensions). Both uncertainty sources combined and expanded to yield a 95% confidence level are called U_{95} and are represented by error bars on the following figures.

RESULTS AND DISCUSSION

Waste addition to the clayey ceramic mass clearly influences the ceramic pellets properties. Figure 1 shows the ceramic pellets Ultimate Compressive Strength (S_{uc}) variation as a result of the oil sludge waste addition. As can be noticed from this figure, the addition of oil sludge waste reduced the strength of the ceramic pellets. As shown in Figure 2, the pellets linear shrinkage is decreased with waste addition. Error bars size is

determined by U_{95} , as described before. According to the waste composition, the non-plastic materials present such as quartz may be contributing to these phenomena. Figure 3 (a) shows the fractured surface of a waste-free fired pellet. Comparing with the microstructure of waste containing pellets, Figure 3 (b) and (c), the present phases are relatively well distributed and inserted in a continuous matrix. A quartz particle found in a waste-containing pellet is outlined in Figure 3 (d), confirming the presence of non-plastic components. Moreover, quartz particles are likely to induce flaws to the sintered microstructure, acting like stress concentrators.

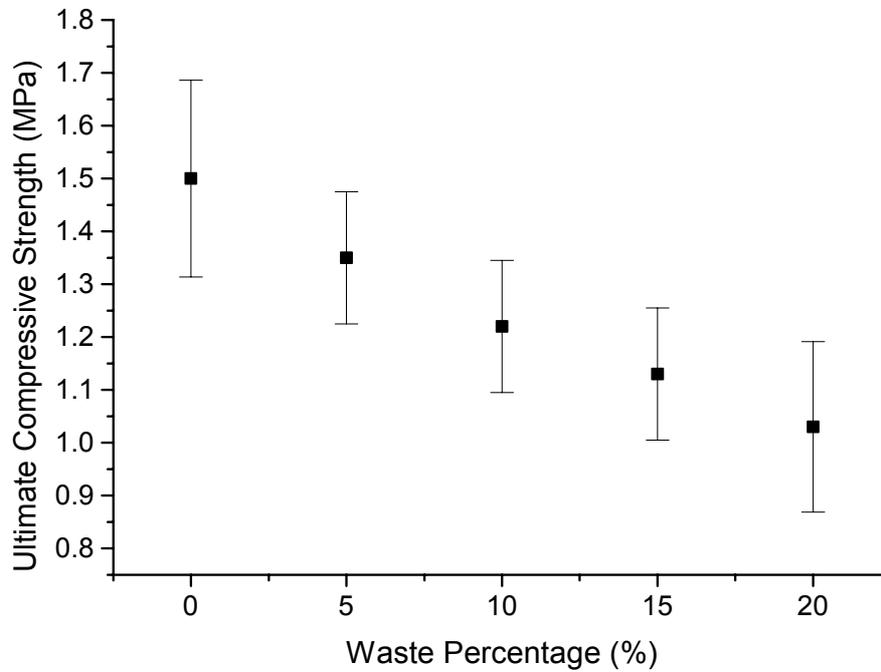


Figure 1: Compressive strength as a function of waste addition for the ceramic pellets.

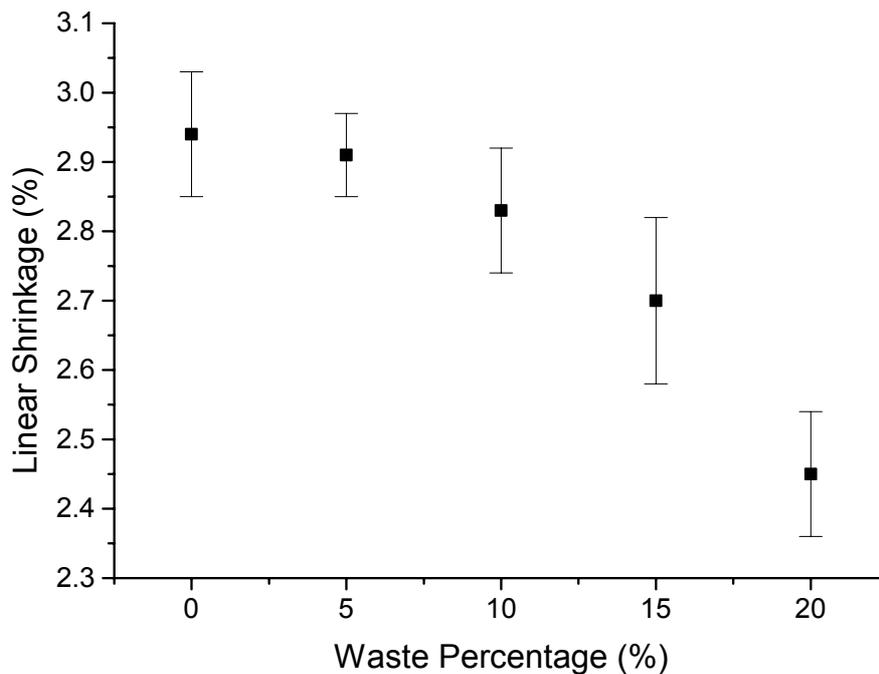
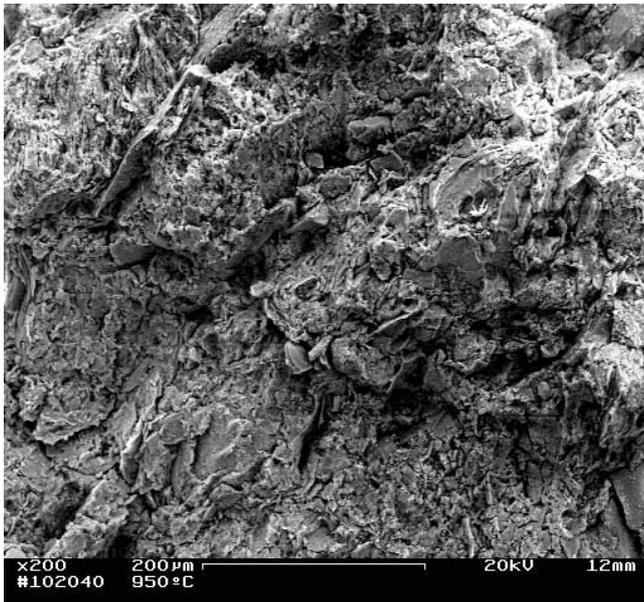
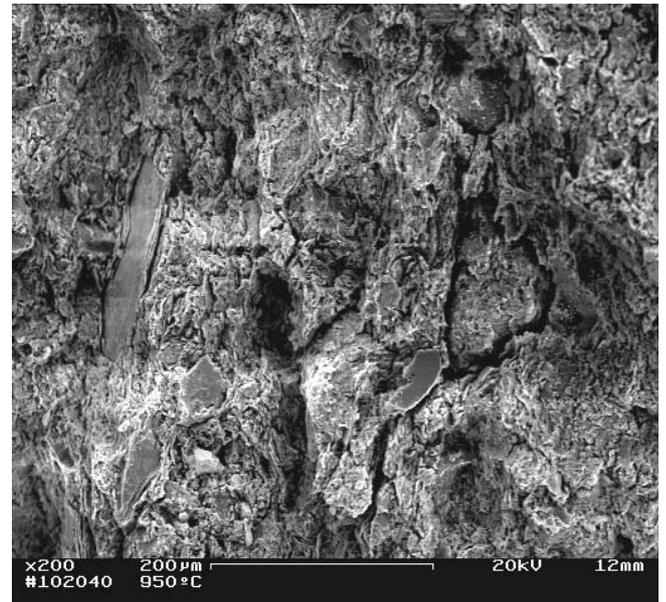


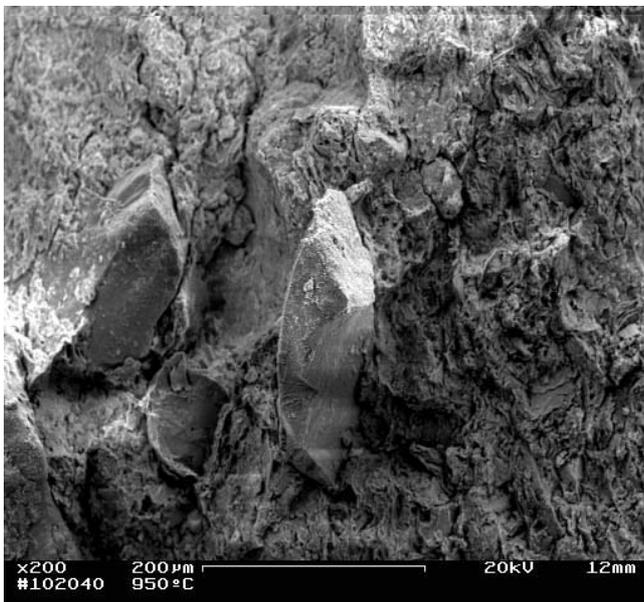
Figure 2: Linear Shrinkage as a function of waste addition for the ceramic pellets.



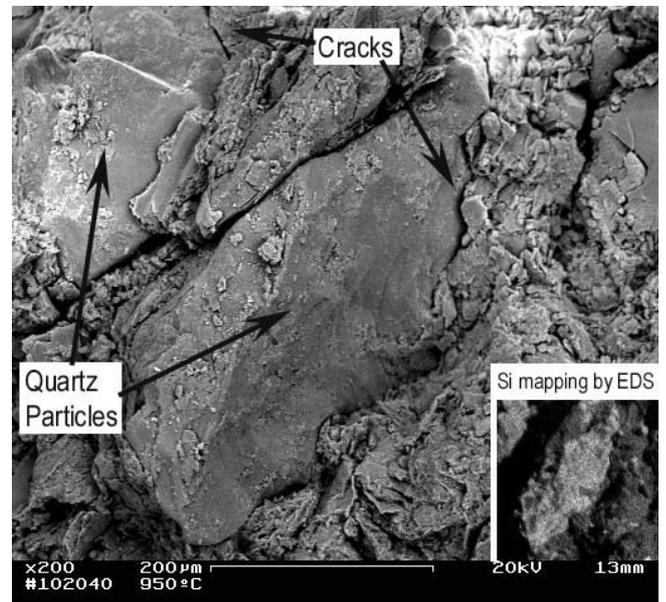
(a)



(b)



(c)



(d)

Figure 3: MEV micrographies showing (a) a waste-free pellet, (b) a 15% waste pellet, (c) a 20% pellet and (d) a detailed quartz particle bearing cracks. (a) through (d) magnification is 200x.

CONCLUSIONS

The chosen thickness for the elastomeric layer did not change significantly the stress field far from the contact region and solved the problem of fracture initiation in the contact area.

As the amount of waste increased, S_{uc} decreased. SEM pictures reveal an increasing amount of quartz crystals as waste amounts also increase and cracks initiating from their edges area are also observed.

Linear shrinkage reduces as waste amount increases, a desired effect for the tile industry. Although a limit for waste addition must be set, as to avoid a sharp decrease in S_{uc} .

The determined expanded uncertainty (U_{95}) shows that result dispersion for S_{uc} tends to be smaller for contaminated clay.

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