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Influence of Paper Industry Effluent Sludge in Ceramic Formulation for Red Wall Tiles (BIII Group)

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At present, the friendly recycling of paper industry effluent sludge has gained great prominence due to the ecological and economic benefits. The aim of this work is to evaluate the influence of the incorporation of paper industry effluent sludge into a red wall tile formulation (BIII group), replacing natural limestone material by up to 10 wt.%. For this purpose, five red wall tile formulations were developed by the dry process, pressed at 47 MPa, and fired at 1170 ºC by using a fast-firing cycle. The wall tile formulations were characterized in terms of chemical analysis, thermal analysis (DTA-TG), and dilatometric analysis. The influence of paper industry effluent sludge on the technical properties (apparent density, water absorption, and flexural strength) and sintered microstructure was investigated. The results showed that red wall tiles containing up to 10 wt.% of paper industry effluent sludge have very good usable final properties, indicating their suitability for the wall tile industrial production (BIII group - ABNT NBR ISO 13006). Such results emphasize the feasibility of ecological and economic recycling of paper industry effluent sludge for the production of red wall tiles.

Keywords: *Paper industry effluent sludge, Wall tile, Properties, Microstructure.*

1. Introduction

It is well known that industrial solid wastes with wide compositional variability are generated in huge amounts worldwide. In most cases, they are usually destined for landfills or illegally discarded in the environment without any treatment. Such a situation causes strong environmental, social, economic and public health impacts. Therefore, one of the major concerns of industries and society around the world is how to find viable solutions for the sustainable final destination of industrial solid wastes with significant repercussions for the circular economy[1-4].

Brazil has an important paper industry. In 2021, Brazilian paper production totaled 10.7 million tons[5]. However, the paper production activities also generate vast amounts of a polluting waste known as paper industry sludge, which comes from the effluent treatment system[6,7]. The paper industry effluent sludge is mainly composed of cellulose fibers, kaolin, calcite, and chemical substances used in effluent treatment[8-10]. It presents elements with water solubility characteristics and, as such, is classified as a non-inert solid waste[11,12]. In Brazil, the main destination of this polluting waste has been landfill disposal, which is considered expensive and problematic. Therefore, it is of crucial importance for the paper production industry to find a sustainable technical solution for the final destination of this abundant polluting waste generated every year. In this scenario, several promising recycling attempts for the use of paper industry effluent sludge has already been explored, including applications in clay bricks, agriculture, concrete, cement-based materials, geopolymers, particleboard, composite panels, energy, ethanol fermentation, etc[9-11,13-21].

The wall tiles belonging to the BIII group of ABNT NBR ISO 13006[22] is widely applied in the coating of residential interiors and protection of building facades[23,24]. The ceramic formulations used in the manufacture of dry-pressed wall tiles are basically composed of natural raw materials, including kaolins, plastic clays, calcareous, feldspars, and quartz in varying proportions[24-26]. Such ceramic formulations are by nature multi-component systems with wide chemical and mineral variability. For this reason, wall tile formulations have the potential to tolerate variable amounts of solid wastes in their composition, as a partial or total replacement for non-renewable natural raw materials. A literature review[26-36] showed that various types of solid wastes have been tested to produce white or red wall tiles, with promising results. It turns out that the paper industry effluent sludge, which contains appreciable amounts of kaolin and calcite in its composition, has rarely been tested in the manufacture of ceramic tile materials[37]. Thus, more researches are needed to generate more knowledge about the use of paper industry effluent sludge in wall tile formulations, with particular interest in its effects on thermal behavior, sintering evolution, and technological properties.

This research was carried out with the aim of increasing knowledge about the use of paper industry effluent sludge to replace natural limestone for the production of red wall tiles (BIII group). Special emphasis is given to the influence of the paper industry effluent sludge on the thermal behavior, sintering evolution, and technical properties.

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2. Materials and Methods

In this work, the raw materials selected for the formulation of red wall tiles were plastic red clay, quartz, dolomitic limestone, and paper industry effluent sludge. The plastic red clay was provided by a ceramic company located in Campos dos Goytacazes-RJ (Brazil). Quartz and dolomitic limestone are commercial raw materials. The paper industry effluent sludge was supplied by a paper manufacturing industry (Companhia Paduana de Papéis - COPAPA) located in Santo Antônio de Pádua-RJ (Brazil), which manufactures tissue paper for sanitary purposes. The starting raw materials were dried in an oven at 110 ºC for 24 h, comminuted, and then sieved to a fraction < 200 mesh (< 75 μm ASTM). The chemical compositions of the starting raw materials have already been reported elsewhere[37]. In addition, the paper industry effluent sludge is essentially composed of a mixture of calcite, kaolin and cellulose fibers[37].

The red wall tile formulations containing paper industry effluent sludge are described in Table 1. In particular, the paper industry effluent sludge was used to replace up to 10 wt.% of dolomitic limestone in the reference formulation (FC1 formulation; sludge-free)[38]. The wall tile formulations were mixed, homogenized, moistened with 7 wt.% of water, and granulated by the dry process.

The mineralogical analysis of the starting raw materials was performed by X-ray powder diffraction using Cu-Ka radiation and 1.5º(2θ)/min scanning speed in a conventional diffractometer (XRD 7000, Shimadzu). The chemical analysis of the wall tile formulations was determined by using an energy-dispersive X-ray spectrometer (Shimadzu, EDX 700). The differential thermal analysis (DTA) and thermogravimetric analysis (TG) of the wall tile formulations were carried out in a simultaneous thermal analyzer ATG-ATD, Netzsch, model STA 409E, from room temperature to 1100 ºC with a heating rate of 10 ºC/min. The dilatometric analysis was performed on compacted pieces using a Netzsch dilatometer, model DIL 402 C, from room temperature to 1150 ºC with a heating rate of 10 ºC/min.

The red wall tiles were produced into rectangular pieces (11.50 cm x 2.54 cm) by uniaxial pressing at 47 MPa, dried at 110 ºC for 24 h, and fast-fired in air at 1170 ºC for 5 min. The heating rate was 30 °C/min. In this work were produced five test specimens for each formulation described in Table 1.

The following technical properties of the fired red tile pieces were determined: apparent density, water absorption, and flexural strength. The values of apparent density and water absorption were determined according to the ASTM C 373- 14a/2014 standard. The flexural strength was determined by means of a three-point loading test using a universal mechanical testing machine (Instron, model 5582), according to ABNT NBR ISO 10545-4.

The microstructure of the fractured surface of the fired wall tile pieces was performed using confocal laser microscopy (3D Measuring Laser Microscope, Lext OLS4000).

3. Results and Discussion

The mineral phases of the starting raw materials used in the red wall tile formulations are given in Table 2. The plastic clay is composed of kaolinite, gibbsite, goethite, and quartz, with predominance of kaolinite. The presence of an iron compound (goethite) in the clay is responsible for the reddish color of the wall tiles after firing. The dolomitic limestone used is essentially composed of calcite and dolomite. High purity commercial quartz has been used. The paper industry effluent sludge is composed of calcite and kaolinite. It also contains an appreciable amount of cellulose fibers. Therefore, the composition of the paper industry effluent sludge used in this work is in accordance with the literature[8-10]. Hence, the red wall tile formulations proposed in this work are complex mixtures of inorganic (kaolinite, gibbsite, goethite, quartz, calcite, and dolomite) and organic (cellulose fibers) components.

Table 3 gives the chemical compositions of the red wall tile formulations. The chemical composition of the studied formulations reflects with good agreement the mineral composition of the starting raw materials (Table 2). However, the partial replacement of up to 10 wt.% of limestone by paper industry effluent sludge resulted in small, but important changes in the chemical composition of the reference formulation (FC1 formulation). The progressive incorporation of paper industry effluent sludge tends to decrease the SiO_2 and MgO contents, but increases the Al_2O_3 and CaO contents. Loss on ignition (LoI) has also increased. This finding is interesting

Table 1. Compositions of the red wall tile formulations (wt.%).

Formulation	Red clay	Quartz	Limestone	Paper sludge
FC1	70.0	15.0	15.0	0.0
FC2	70.0	15.0	12.5	2.5
FC3	70.0	15.0	10.0	5.0
FC4	70.0	15.0	7.5	7.5
FC5	70.0	15.0	5.0	10.0

Table 2. Mineral phases identified in the starting raw materials.

Raw Material	Mineral Phases
Red clay	Kaolinite, gibbsite, goethite, mica, and quartz
Quartz	Quartz
Limestone	Dolomite and calcite
Paper sludge	Kaolinite and calcite

Table 3. Chemical composition of the red wall tile formulations (wt.%).

	SiO2	Al2O3	Fe2O3	CaO	MgO	K2O	Na2O	MnO	TiO2	P2O5	+LoI
FC1	60.67	15.89	2.29	3.75	3.27	0.97	0.38	0.07	0.79	0.07	11.85
FC2	60.31	15.98	2.28	3.81	2.79	0.97	0.38	0.07	0.80	0.08	12.53
FC3	60.02	16.06	2.28	3.86	2.30	0.96	0.37	0.07	0.80	0.07	13.21
FC4	59.70	16.15	2.26	3.92	1.82	0.96	0.36	0.07	0.78	0.07	13.91
FC5	59.37	16.24	2.26	3.97	1.34	0.95	0.36	0.06	0.79	0.06	14.60

+loss on ignition

and may influence the processing of high quality wall tiles using a fast firing cycle.

DTA-TG curves of the FC1 formulation (sludge-free wall tile formulation) are shown in Figure 1. The thermal behavior is well correlated with the chemical and mineral compositions of the FC1 formulation and can be described as follows. Four endothermic events accompanied by a mass loss were detected. A small endothermic event observed at ~ 100 ºC and accompanied by a mass loss of 1.021% is related to surface water desorption. The endothermic event observed at 275.5 ºC with a mass loss of 1.142% is due to the dehydration of hydroxides (gibbsite and goethite). The endothermic event seen at 515.4 ºC and accompanied by a mass loss of 4.280% is associated with the dehydroxylation of kaolinite. At 769.1 ºC, an endothermic event related to the decomposition of carbonates (calcite and dolomite) occurred, with a mass loss of 5.470%. The total mass loss of the FC1 formulation was 11.913%. The ATD curve also indicated that at 950.7 ºC an exothermic event occurred. This event is probably related to the formation of new crystalline phases[24-26,31], particularly calcium and magnesium aluminosilicates and mullite.

Figure 2 shows the thermal behavior (DTA-TG curves) of the FC3 formulation (with 5 wt.% of sludge). It may be noted that the partial replacement of limestone by paper industry effluent sludge tends to influence the thermal behavior of the reference wall tile formulation. More specifically, the ATD curve showed an additional exothermic event at 341.7 ºC accompanied by a loss of mass. Such an exothermic event is associated with the thermal decomposition of the cellulose fibers present in the paper industry effluent sludge. The FC3 formulation showed a total mass loss of 12.112%, which is higher than that of the reference formulation. This is in agreement with the loss on ignition of the red wall tile formulations, as shown in Table 3.

The dilatometric curves of the FC1 formulation and FC3 formulation are shown in Figures 3 and 4, respectively. As it can be observed, the partial replacement of limestone with paper industry effluent sludge caused small differences in the sintering behavior of the reference red wall tile formulation. It was found that the sintering curves presented little expansion from room temperature (~ 25 ºC) up to ~900 °C. Both formulations showed a knee with increased expansion between ~500 ºC and 600 ºC, which is related to simultaneous thermal events of α-β quartz inversion and kaolinite dehydroxylation. Between 600 e 900 ºC, the expansion rate gradually decreased due to the decomposition of carbonates (dolomite and calcite). Both formulations also showed the beginning of shrinkage around 900 ºC, but it became more pronounced from 1035 ºC for the FC1 formulation and 1065 ºC for the FC3 formulation. In this temperature region, sintering and reactions that lead to the formation of new crystalline phases take place simultaneously. The temperature of maximum sintering rate was 1131.3 ºC for the FC1 formulation and 1197.8 ºC for the FC3 formulation.

The microstructural characterization of the fractured surface of the red wall tiles fired at 1170 ºC was performed by laser confocal microscopy, as shown in Figures 5 and 6. The 3D images remarkably show complex topographies and highly porous microstructures. According to the literature[34-36], this type of microstructure is typical of porous wall tiles. However, a trend towards a more porous sintered microstructure can be observed for the wall tile formulations incorporated with paper industry effluent sludge. This finding is related to the paper industry effluent sludge that contains cellulose fibers, which decomposes during firing and generates more open pores. This result is in line with the highest loss on ignition (Table 3) and highest mass loss (Figure 2) presented by the formulations containing paper industry effluent sludge.

The apparent density of the red wall tile pieces fired at 1170 ºC is shown in Figure 7. The apparent density of the wall tiles produced ranged between 1.73 and 1.82 g/cm^3. In particular, the effect of the incorporation of paper industry effluent sludge was to decrease the apparent density. This finding is mainly associated with the composition of the paper industry effluent sludge, which is rich in cellulose fibers, resulting in higher loss on ignition in the red wall tile formulations, as shown in Table 3. The TG curve also showed a higher mass loss, as seen in Figure 2. Therefore, porous

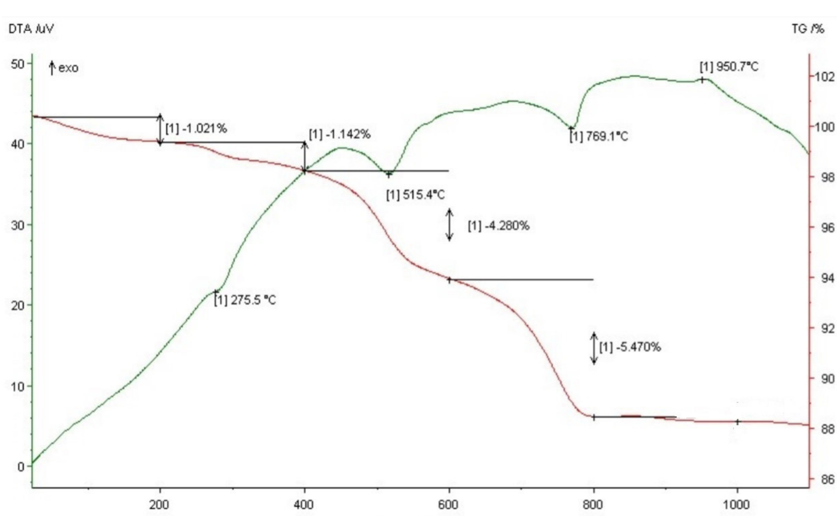

Figure 1. DTA-TG curves for the FC1 formulation.

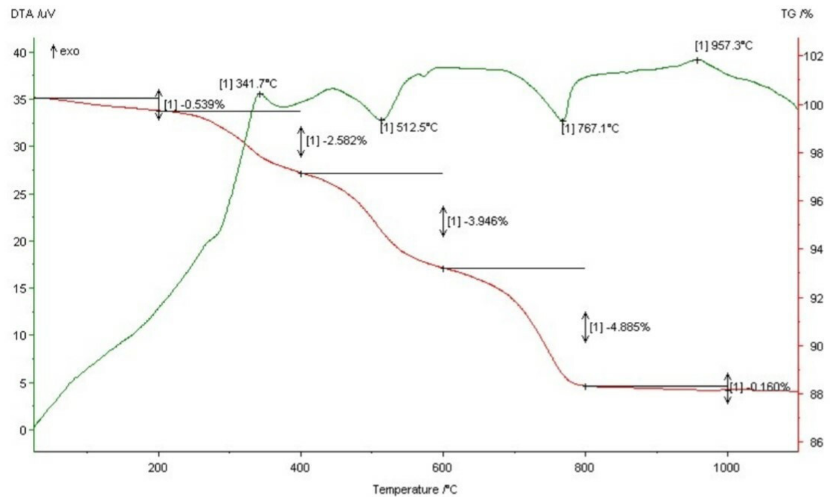

Figure 2. DTA-TG curves for the FC3 formulation.

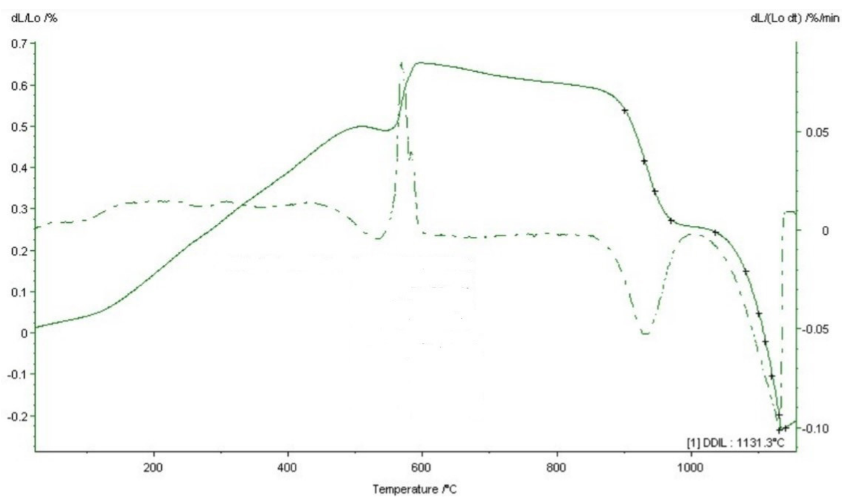

Figure 3. Dilatometric analysis for the FC1 formulation.

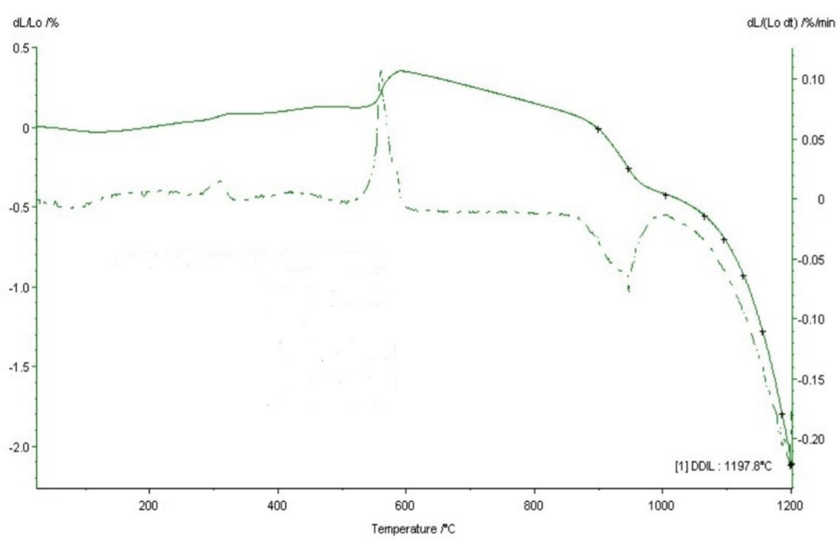

Figure 4. Dilatometric analysis for the FC3 formulation.

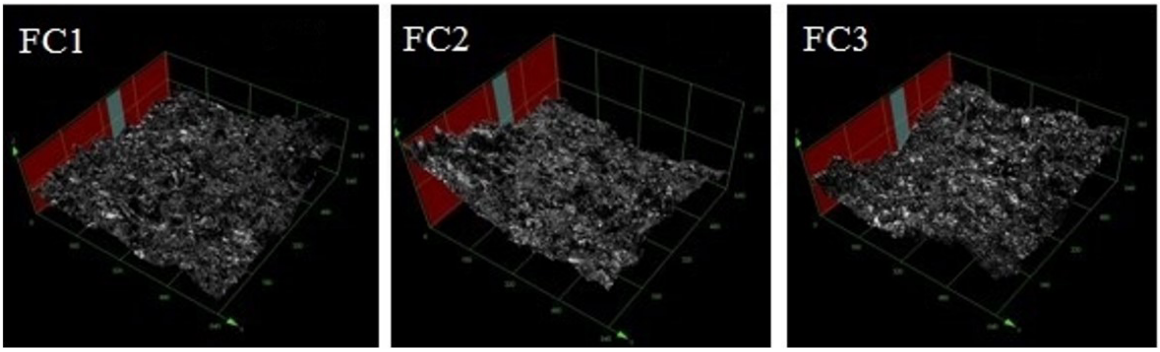

Figure 5. 3D confocal images of the fired red wall tile pieces (Magnification: 430x).

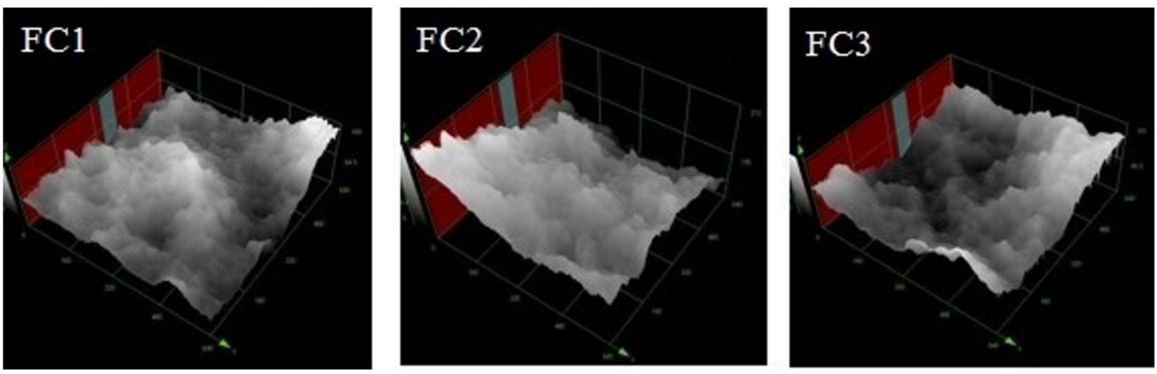

Figure 6. 3D confocal images with noise filter of the fired red wall tile pieces (Magnification: 430x).

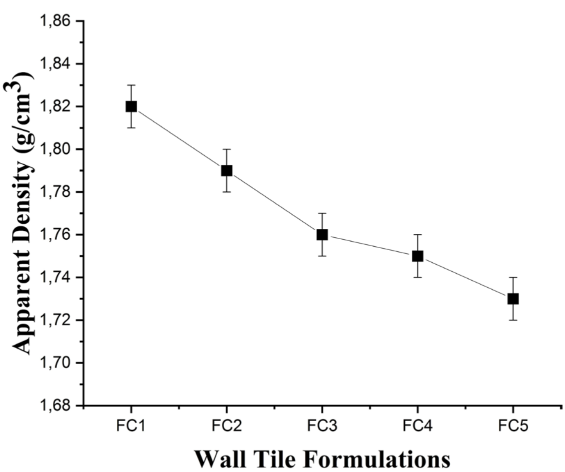

Figure 7. Apparent density of the red wall tile pieces.

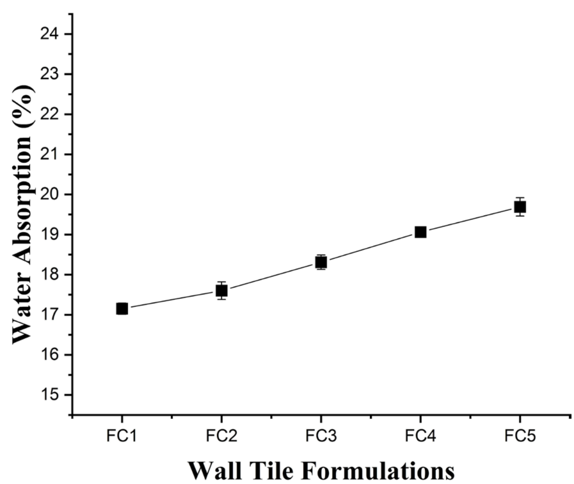

Figure 8. Water absorption of the red wall tile pieces.

wall tile specimens can be prepared with paper industry effluent sludge with only a small sacrifice of densification.

The water absorption (open porosity) of the red wall tile pieces is shown in Figure 8. The high values of water absorption (17.15% – 19.69%) obtained are mainly associated with the dehydration of hydroxides, kaolinite dehydroxylation, and decomposition of carbonates (calcite and dolomite), which generates open pores during the firing step. The results also showed that as the amount of paper industry effluent sludge increased, the water absorption also increased. This behavior can be explained by the decomposition of cellulose fibers from the paper industry effluent sludge, which influence the densification behavior of the wall tile pieces. Thus, a correlation between water absorption, sintered microstructure and apparent density has been well established.

The flexural strength of the floor tile specimens is shown in Figure 9. The red wall tile pieces showed flexural strength values between 14.42 MPa and 16.17 MPa. However, for additions of paper industry effluent sludge above 5 wt.% (FC4 and FC5 formulations), a tendency towards a gradual decrease of the mechanical strength was observed. This behavior reflects the lowest degree of densification (lower apparent density) and higher open porosity (higher water absorption) of the red wall tiles produced with paper industry effluent sludge.

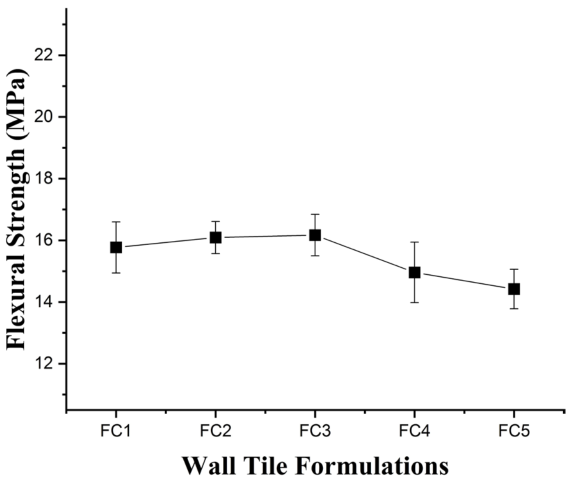

Figure 9. Flexural strength of the red wall tile pieces.

In terms of potential for practical application, the water absorption values (WA) of the red wall tile pieces produced in this work (WA = 17.15% – 19.69%) were compared with the prescribed value (WA > 10% - BIII group) in the ABNT NBR ISO 13006 standard[22]. All red wall tile pieces produced can be classified as belonging to BIII group, regardless of the amount of paper industry effluent sludge added. The ABNT NBR ISO 13006 standard also recommends that fired wall tiles have a flexural strength (FS) value > 15 MPa. As can be seen in Figure 9, the FC1, FC2 and FC3 formulations (up to 5 wt.% of sludge) reached the FS value (> 15 MPa) recommended by ABNT NBR ISO 13006. For the FC4 and FC5 formulations (above 5 wt.% of mud), however, the FS value was only reached at the threshold of the recommended value, within the limits of dispersion. This result is important, as it points to a maximum limit for replacing dolomitic limestone by up to 10 wt.% of paper industry effluent sludge. In this context, it is recommended to avoid the addition of paper industry effluent sludge above 10 wt.% in the red wall tile formulation, as it strongly impacts the mechanical strength.

4. Conclusions

The results indicated that paper industry effluent sludge used in this work could be recycled as a partial replacement for dolomitic limestone in red wall tile formulations (BIII group). It was found that the incorporation of paper industry effluent sludge tends to influence the chemical composition, thermal behavior, densification, and technical properties. The results also suggest that red wall tiles (BIII group; ABNT NBR ISO 13006 standard) containing up to 10 wt.% of paper industry effluent sludge can be produced at 1170 ºC by using a fast-firing cycle. Based on this, the use of paper industry effluent sludge in red wall tile formulations can be a sustainable technical solution that leads to many economic and environmental benefits.

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