

Influence of MDF residues on the physical and mechanical properties of clay ceramic products, after firing

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Abstract:

The aim of this research is based on the social importance of using MDF waste in clay ceramic pastes for building applications, aiming at reducing the environmental impacts caused by the inappropriate disposal of waste and the exploitation of natural resources to obtain clay ceramic products. The goal of the work is to analyze the influence of the MDF residue addition on the physical-mechanical properties of clay ceramic pastes for the manufacture of ceramic blocks by uniaxial pressing forming method. The residues and ceramic pastes were characterized physically, chemically, and mineralogically using a range of methods. Physical characterization included determining particle size distribution laser diffraction, linear shrinkage, apparent porosity, plasticity and flexural strength. Chemical characterization was achieved by determining elemental composition (X-ray fluorescence). Mineralogical characterization was performed using scanning electron microscopy (SEM) for high-resolution imaging and elemental analysis. Subsequently, specimens were prepared with mixtures of 10 and 20% of the residue by mass, which were subjected to heat treatment at 800 and 900°C. The physical-mechanical properties were determined using the following tests: water absorption, apparent porosity, loss on ignition and flexural modulus of rupture. The results showed that, when subjected to high temperatures, the MDF residue is largely eliminated from the composition, leaving the body with high porosity, directly influencing the final strength of the product, making it impossible to immediately indicate the use of the material in civil construction.

Keywords: Clay ceramic; MDF; uniaxial pressing; porosity.

1. INTRODUCTION:

The search for solutions and alternatives related to environmental issues is a critical topic in today's world. One significant issue is waste generation and its effects on the environment. The increase in waste generation, especially non-biodegradable waste, has led to environmental pollution, degradation of natural habitats, and adverse impacts on wildlife. As a result, there is a need to develop sustainable solutions and alternatives to address waste generation and its effects.

Reuse is an essential alternative that can reduce waste generation and mitigate its effects on the environment. This includes strategies such as recycling, upcycling, and repurposing, which can help to conserve resources, reduce landfill waste, and minimize the carbon footprint linked with the production of new materials. [1]. In recent years, the use of MDF (medium density fiberboard) in the furniture sector is growing in Brazil, replacing solid wood furniture [2]. MDF is a sheet produced from the agglutination of wood fibers, with synthetic resins and joint action of temperature and pressure [3].

Like other types of raw material, MDF also generates waste, which causes damage to the environment, as it is disposed of improperly, without reuse or prior treatment. MDF panels were introduced in the 1960s and began to be manufactured in Brazil by Duratex, in 1997, with a factory in Agudos (SP). In 2005 its world consumption reached 40 million m³. In the period between 1995 and 2005, world consumption of MDF grew at an average annual rate of 18.5%. With 61% of world demand in the segment, China (40%), United States (12%), South Korea (5%) and Brazil (4%) are the major centers of world consumption [3].

Due to the inherent characteristics of MDF, it has a wide applicability. From the manufacture of door and drawer fronts and other more elaborate pieces in the furniture industry, with machining on edges or faces, such as table tops, racks and shelves. To the production of floors, baseboards, door cushions, jambs, machined doors, and turned parts as stair balusters, table legs and packaging in the building industry [3].

The Brazilian clay ceramic industry represents about 1% of the national GDP. The State of Paraíba has many companies in this segment. Although their economic and social importance, investments in technology for a more rational and optimized use of these resources are considered insufficient [4]. The challenges of the production processes of these companies are varied, from the attempts to reduce costs, the environmental responsibilities, to stand with quality and market parameters in a very competitive sector. In the present work, the MDF residue was added to a clay ceramic paste aiming to recover or reuse the waste. Using MDF residues from the furniture industry in ceramic pastes, a clay ceramic product can be obtained, keeping the quality standards required by responsible bodies and entities for specific physical-mechanical parameters. At the same time reducing the energy used in the firing of the product, reducing the amount of raw material used, and reducing the environmental impact.

Research on the reuse of solid waste for the development of innovative products, as with the addition of MDF powder in building materials, can save the use of natural, non-renewable clays.

The aim of this research is based on the social importance of using MDF waste in clay ceramic pastes for the manufacture of building materials, aiming at reducing the environmental impacts caused by the inappropriate disposal of waste and the exploitation non-renewable natural resources to obtain clay ceramic products. Also, the goal is to analyze the influence of the addition of MDF waste on the physical-mechanical properties of specimens of clay ceramic pastes formed by uniaxial pressing.

2. MATERIALS AND METHODS:

2.1. Materials

For the development of this work, a clay from a ceramic block factory, located near the city of Soledade, State of Paraíba, Brazil, was used. The factory produces ceramic blocks sold in the regions of Campina Grande, PB, and Maceió, AL, both in Brazil. Pure MDF waste, collected at Premium Gestores company (Campina Grande, PB), was used. MDF is used as the main raw material for designed furniture and derivatives. The waste was taken to the laboratory and was sieved in #80 mesh to be mixed with the clay sample. Subsequent procedures were performed.

2.2. Methods:

The MDF residue was dried and ground until it passed through an 80-mesh sieve. The additions were 0 (AP), 10 (AM10) and 20 wt.% (AM20) of the MDF residue to the total weight of the composition of the clay paste with residue, as detailed in Tab.1.

Table 1. Composition of the samples

Samples	% Clay	% MDF
AP	100	0
AM10	90	10
AM20	80	20

2.2.1. Characterization of the raw materials

Initially, the clay sample was dried at room temperature and subsequently subjected to the following processing steps: crushing, grinding, and sieving in ABNT N 200 (0.074mm) sieves, to carry out the characterization tests and ABNT N 80 (0.018mm) for forming and obtaining the physical-mechanical characteristics.

2.2.1.1. Determination of plasticity

The clay sample was sieved through an ABNT 80 mesh, then water was added until the clay showed workability. The sample was characterized using the Casagrande test to determine the liquidity limit (LL), the maximum amount of water that turns the plastic paste into a fluid suspension, the plasticity limit (LP), the amount of water to soil become deformable, and therefore to determine the plasticity index (IP), which indicates the workability of the material. The plasticity index was calculated according to NBR 7180 standard [5], equation (1).

The plasticity index is a measure of the plasticity of a soil or clay, which indicates its ability to deform without breaking. The crumbling water from the percentage of moisture after 25 blows (liquidity limit):

$$IP=LL-LP \quad (\text{eq.1})$$

The moisture content at 25 strokes represents the amount of water required to achieve a certain consistency of the soil when subjected to 25 strokes in a standard device called the Casagrande apparatus. The crumbling water content is the amount of water required to cause the soil to crumble when rolled into a thread of a certain diameter.

The plasticity index is an important parameter in the classification of soils, as it provides an indication of their engineering properties, such as compressibility, shear strength, and bearing capacity. It is also useful in predicting the behavior of soils under different environmental conditions and for designing structures on or with soils.

2.2.1.2. Particle size distribution by laser diffraction

The particle size of the samples was determined using a Cilas 1064 equipment. Sodium hexametaphosphate (HMFNa) was used as a dispersing agent for the pure clay (PA) sample.

2.2.1.3. Chemical analysis

The clay sample was classified by sieving in a granulometry less than 74 μm and dried in an oven at 110 $^{\circ}\text{C}$ for 24 h. The chemical analysis was carried out in an X-ray fluorescence spectrometer (Shimadzu EDX 720), under vacuum, using the semi-quantitative method to determine the elements present in the sample.

2.2.2. Forming of the test specimens

The clay sample plus an amount of the MDF residue were mixed and moistened, with a water content of 7 wt.%, and remained for 24 hours, in a basin over a damp cloth, aiming to obtain a good homogenization. The compositions were subjected to manual uniaxial pressing process in a rectangular shape in dimensions (6.0 \times 2.0 \times 0.5) cm^3 . The samples were molded under pressure of 2400 kgf/cm^2 . The specimens were measured, weighed, and placed in an oven at 110 $^{\circ}\text{C}$ for 24 hours. After drying, they were again measured and weighed, and subsequently, the firing process was carried out.

2.2.3. Firing of the test specimens

After being formed and dried in an oven, the specimens were fired in an electric oven. The firing was carried out at two temperatures (800 and 900 °C). A heating rate of 5 °C/min was used, with a residence time of 120 minutes at the maximum temperature.

2.2.4. Characterization of the test specimens

Fig.1 shows the specimens made with the compositions described in Tab.1, before and after firing.



Figure 1. Raw and fired specimens

2.2.4.1. Linear drying shrinkage

For the drying shrinkage calculation, the specimens were measured right after forming and after the drying step at 110 °C, following the ABNT NBR 15097-1 (2011) standard. Equation (2) was used for the calculations.

$$RS(\%) = \frac{CM-CS}{CS} \times 100 \quad (\text{eq.2})$$

Were RS being the drying shrinkage (%); CM is the length of wet sample (mm); and CS is the length of dry sample (mm).

2.2.4.2. Flexural bending strength (TRF) after drying

The TRF determines the minimum strength that the sample will present for handling (for drying, before firing, etc.). The test was carried out on dry specimens using a Shimadzu AG-X testing machine, with a loading rate of 0.5 mm/min, and a capacity of 100 N. The specimens were placed on two rectangular supports and a third support was fixed above to exert pressure on the specimen. The number of samples used for the TRF test was 50. The value of the flexural bending strength (dry) for solid bricks must be at least 1.5 MPa and 2.5 MPa for ceramic blocks for masonry. The TRF for fired solid bricks must be at least 2 MPa, and for ceramic blocks for masonry, 5.5 MPa. After firing, the specimens were evaluated for water absorption, firing linear shrinkage, loss on ignition, and flexural bending strength.

2.2.4.3. Water absorption (AA)

The AA determines the amount of water that the material absorbs and indirectly determines the porosity of the ceramic bodies. Water absorption values vary according to the purpose of the material. For ceramic blocks for sealing masonry, the value can vary from 8 to 22%, according to the NBR 15270-1 (2005) standard [6]. For tiles, the maximum permissible limit is up to 20%, according to the NBR 15310 (2005) standard [7].

The calculation of the water absorption was using equation 3, and the test was carried out in accordance with the ABNT NBR 15097-1 (2011) standard. The specimen was weighed after firing, then immersed in a container with water, left for 24 hours at rest, and then weighed again.

$$AA(\%) = \frac{PU-PS}{PS} \times 100 \quad (\text{eq.3})$$

Were AA being the water absorption (%); PU is the wet weight (g); PS is the dry weight (g).

2.2.4.4. Apparent porosity (AP)

To determine the apparent porosity, the specimens were immersed in water for 24 hours. After this time, the specimens were weighed on a hydrostatic scale, thus getting the immersed weight. With a damp cloth, the excess water was removed from the surface of the specimens, which were weighed again on an analytical balance, thus getting the wet weight saturated with water. With these values, it was possible to calculate the apparent porosity using equation 4.

$$PA(\%) = \frac{PU-PS}{PU-PI} \times 100 \quad (\text{eq.4})$$

Were PA being the apparent porosity (%); PU is the wet weight (g); PS is the dry weight (g); and PI is the immersed weight (g).

2.2.4.5. Fired linear shrinkage (RQ)

After the firing step, the specimens were measured again, and using equation 5, the shrinkage after firing was determined, following the ABNT NBR 15097-1 (2011) standard.

$$RQ(\%) = \frac{CS-CQ}{CS} \times 100 \quad (\text{eq.5})$$

Were RQ being the fired linear shrinkage (%); CS is the length of dry piece (mm) (g); CQ is the length of fired piece (mm)

2.2.4.6. Loss on ignition (PF)

After the firing stage, the specimens were weighed again, and with the weights of the fired and dried specimens, the loss in mass after drying and firing were analyzed.

2.2.4.7. Scanning electron microscopy (SEM)

Among the dried and fired specimens submitted to the bending strength test, four samples were selected to be submitted to SEM analysis, one dried sample and the other fired at 900 °C, one specimen of each composition (AP, AM10 and AM20). Initially, the samples were covered with gold (Sanyu Electron SC-701metallizer) for a time of 4 minutes at 15 mA, then the SEM analysis was performed (Shimadzu S5X-550).

3. RESULTS AND DISCUSSION:

3.1. Characterization of clay plasticity

The plasticity indexes, the liquidity limit (LL), plasticity limit (LP), and plasticity index (IP), were determined to indicate the range of consistency of the clay sample, if appropriate for processing.

The clay sample (AP) presented LL = 46.76%, LP = 29.52% and IP = 17.24%, which are appropriate for molding. When the plasticity index is greater than 15% the sample is classified as highly plastic, according to the consulted literature [8].

Therefore, the clay sample presented a PI of 17.24%, and for clay ceramics, plasticity indexes of 10 to 20% are suggested. Therefore, the sample shows a plasticity index within the recommended limit range, according to [9]. Plasticity indexes below 10% can be problematical, as a small variation in the water content for forming can lead to a change in the consistency of the ceramic paste. The referred indexes depend on the handling during the test using the Casagrande method, which changes the chained structure of the clay minerals.

3.2. Particle size distribution by laser diffraction

Fig.2 shows the particle size distribution referring to the granulometric analysis of the clay sample (AP).

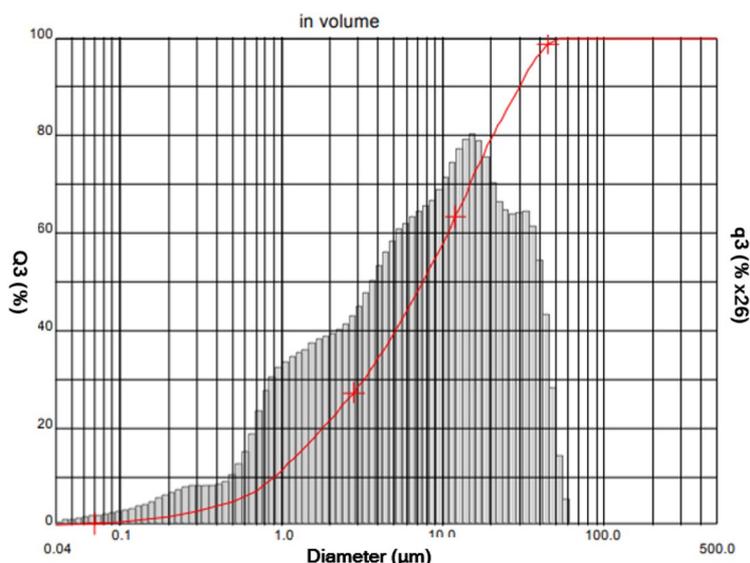


Figure 2. Histogram of the granulometric analysis of the AP sample

It is common to consider the clay fraction of a natural ceramic raw material, with a granulometric fraction smaller than 2 µm. Therefore, the clay fraction would have particles below 2 µm. The silt fraction would have particles between 2 and 20 µm, and the sand fraction particles greater than 20 µm [10]. The clay fraction is mainly related to clay minerals, which are responsible for the development of the plasticity of the clay + water system, according to [11].

Observing Fig.2, the sample presented 21.60% of clay fraction, 57.74% for the silt fraction, and 20.66% for the sand fraction, with an average diameter (Dm) of 11.65%. Therefore, the clay sample shows a low amount of clay fraction, but a high amount of silt fraction, resulting in a balanced particle size, giving the clay paste a moldable behavior for the forming process.

3.3. Chemical analysis by X-ray fluorescence (XRF)

Tab.2 shows the chemical composition of the clay sample, analyzed in an X-ray fluorescence spectrometer. The clay sample can be classified as aluminous silica with a high silica content (more than 50%), probably originating from clay minerals and free silica in its composition.

Table 2. Chemical composition of the clay sample (AP), in (wt.%)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	TiO ₂	CaO	MgO	Other oxides
53.50	25.00	10.54	3.88	1.22	1.85	2.98	0.99

Note the presence of alumina (Al₂O₃), which is normally related to the proportion of clay mineral and feldspar. This low alumina content is characteristic of the clays used in clay ceramic technology. The iron oxide content is greater than 10%, which is characteristic of clays that fire in red colors and are used in clay ceramics. Therefore, the sample has a typical clay composition for clay ceramics [12].

The alkaline oxides (K_2O and others in lower levels) and alkaline earth oxides (MgO and CaO) are flux oxides and are important for the formation of the glassy phase during firing, which may be an indication that there is a reasonable concentration of feldspar in the raw material. The presence of Na_2O in the sample was not detected. Regarding MgO , the presence of this oxide generally indicates the presence of dolomite or chlorite (a kind of mica) [12].

3.4. Technological parameters evaluated after the stages of forming and firing of the samples

3.4.1. Drying

In Tab.3 the data of the physical-mechanical properties of the specimens dried at $110\text{ }^\circ\text{C}$ are shown.

Table 3. Physical-mechanical properties after drying

Sample	Drying linear shrinkage (%)	Flexural bending strength (MPa)
AP	2.2 ± 0.0	4.17 ± 0.69
AM10	0.37 ± 0.23	2.05 ± 0.10
AM20	0.47 ± 0.0	2.14 ± 0.18
VR	6.00	4.5 (MPa)

Based on the dimensions of the wet and dried specimens at $110\text{ }^\circ\text{C}$, there was a small variation in dimensions due to the loss of forming water. The variation was expected to be greater for samples with the addition of MDF, but remained in the reference range, with a maximum of 6% of linear shrinkage after drying.

There was a decrease in strength with the addition of MDF powder, but even so, the range of reference values for strength after drying is between 4.5 and 6.5 MPa [12]. With the addition of MDF powder, the minimum value of strength after drying was measured at 4.7 MPa. These results indicate that, during the drying process, in the manufacturing plant where the blocks are stockpiled, up to seven blocks can be stacked without suffering deformation in their shape.

3.4.2. Firing

Tab.4 shows the data of the physical-mechanical characteristics of the specimens sintered at 800 and $900\text{ }^\circ\text{C}$. The water absorption is directly related to the strength of the final product, since the greater the water absorption by the clay sample after firing, a decrease in the strength of the block would result. In this work, there was an increase in the absorption of water in the specimens of the AM10 and AM20 samples regarding the AP sample. But the individual specimens of the AM10 sample were within the limit range regarding the reference values for both temperatures. The results can be explained by the presence of pores in the compositions, because during firing the MDF powder suffered combustion, leaving empty pores, and thus increasing the water absorption in the ceramic body.

The apparent porosity had a gradual increase from the AP sample to the AM10 and AM20 samples, due to the combustion of the MDF powder during firing, resulting in a more porous material, and consequently more water was absorbed, implying a decrease in the strength of the final product. This feature can be observed in the images of the SEM analysis.

The linear shrinkage after firing was insignificant. In the AP sample there was a small expansion of the ceramic bodies, caused by pyro-expansion, which can be explained by the composition of the clay. Some oxides are fluxes that can form a liquid phase inside the clay paste, that expands during firing. The compositions AM10 and AM20, as expected, showed a small shrinkage, justified by the pyrolysis of the MDF powder during sintering at high temperatures, thus leaving empty pores in the ceramic bodies. Considering the mass variation as the main parameter, the loss on ignition during firing analyzes how much the ceramic body lost mass during sintering at high temperature. There was a gradual loss of mass in the AM10 and AM20 samples in relation to the AP sample. This mass loss can be attributed to the dihydroxylation of the clay mineral at around $550\text{ }^\circ\text{C}$, a process in which the hydroxyl groups ($-OH$) of the clay minerals are released due to the breakdown of its crystalline structure, resulting in a loss of mass.

The flexural bending strength is the most important characteristic of the whole work, as it determines the strength of the material under working conditions, and consequently the workability of the product. A predetermined range from 6 to 30 MPa is defined in the standards as the reference. Therefore, the AP composition is within the reference standards [11,12]. Compositions AM10 and AM20 presented values well below the minimum specified, due to the elimination of MDF from the mixtures during sintering at high temperatures, increasing the porosity of the material, therefore drastically reducing its strength. This feature results in low quality products, as they do not meet the minimum strength to be marketed for applications as building material.

Table 4. Physical-mechanical characteristics of the fired samples

Samples	T (°C)	Water absorption (%)	Apparent porosity (%)	Linear shrinkage after firing (%)	Loss on ignition (%)	Flexural bending strength (MPa)
AP	800	11.88 ± 0.14	23.94 ± 0.20	-0.57 ± 0.15	8.24 ± 0.10	7.71 ± 0.55
	900	11.62 ± 0.39	23.29 ± 0.61	-0.18 ± 0.23	9.03 ± 0.42	6.21 ± 0.76
AM 10	800	24.62 ± 0.24	37.63 ± 0.14	0.43 ± 0.11	17.53 ± 0.03	0.77 ± 0.33
	900	25.16 ± 0.67	37.86 ± 0.94	1.07 ± 0.11	18.51 ± 0.11	0.53 ± 0.12
AM 20	800	33.69 ± 0.77	42.61 ± 0.23	0.81 ± 0.12	32.26 ± 0.25	0.25 ± 0.04
	900	37.26 ± 0.83	46.43 ± 0.81	1.55 ± 0.12	34.63 ± 1.10	0.65 ± 0.16
VR*	950	2 - 25	5 - 42	NE	NE	6 - 30

3.5. Scanning electron microscopy (SEM)

Fig.3 (a, b, c, d) show the images obtained by SEM analysis. Fig.3(a) show the microstructure of the dried AM20 composition. The presence of very well defined MDF powder fibers mixed with clay can be observed. Fig.3(b) shows the microstructure of the fired AP sample, where its packaging can be observed.

Fig.3(c) shows the AM10 sample fired at 900 °C. There is a decrease in the number of fibers of the MDF powder and appearance of pores during the pyrolysis of the fibers during firing. Finally, Fig.3(d) shows the AM20 sample fired at 900 °C. There is a large presence of pores due to the removal of the MDF dust after firing, justifying the large absorption of water and the sudden decrease in strength of the ceramic bodies.

The pores act as stress concentrators and are the main cause of defects in ceramic products, since they ease the onset of cracks and, therefore, a possible fracture. The pore size distribution and total porosity change the properties of ceramics. The voids act negatively on the fracture strength.

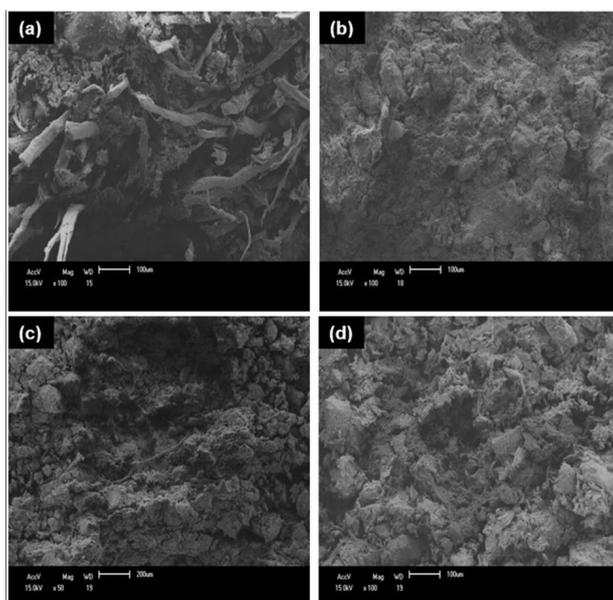


Figure 3. a) AM 20 dried sample; b) AP sample fired at 900 °C; c) AM10 sample fired at 900 °C; d) AM20 sample fired at 900°C

4. CONCLUSIONS

The purpose of this study was to analyze the influence of MDF waste added to clay ceramic production on post-firing physical-mechanical parameters. A pure clay composition was prepared as a reference and two compositions with the presence of MDF residues were prepared for comparison.

Analyzing the laboratory results, the chemical, physical and granulometric characteristics of the pure AP clay sample, used as a standard, are in the range recommended for the manufacture of clay ceramic products, except for small numerical differences. The AP sample has a granulometry and plasticity limits within the ranges recommended by the literature. According to the chemical analysis, the AP sample has a typical clay composition for clay ceramics, with a predominance of SiO_2 , Al_2O_3 and Fe_2O_3 , which is responsible for the natural red color of the clay ceramic products. Regarding the technological characteristics of the samples processed in a manual press, when subjected to high temperatures, the MDF residue is largely eliminated from the composition, leaving the test specimen with high porosity, directly influencing the final strength of the ceramic body, therefore not being possible to immediately use the MDF waste in building materials. The images produced by the scanning electron microscope prove the great porosity that remains in the ceramic bodies after firing, which shows their fragility, resulting in products of low strength, not suitable for the manufacture of clay ceramic products for building materials.

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