Characterization of rice husk ash as a filler in ceramics through contact angle and flexural strength experiments

Caracterização da cinza da casca de arroz como carga em cerâmica por meio de experimentos de ângulo de contato e resistência à flexão

Caracterización de la ceniza de cáscara de arroz como carga em cerámica mediante experimentos de ángulo de contacto y resistencia a la flexión

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Resumo: O Brasil produz uma quantidade significativa de resíduos de casca de arroz que são considerados de baixo ou nenhum valor econômico, podendo gerar impactos negativos ao meio ambiente. Este estudo apresenta uma experimentação da mistura das cinzas da casca de arroz com a argila comercial, visando aproveitar as propriedades presentes nas cinzas no material. Para isso, as cinzas foram misturadas à argila, em quantidades pré-definidas, para a fabricação de amostras de teste. As amostras foram submetidas a ensaios de ângulo de contato e resistência à flexão, para caracterizar preliminarmente o efeito das cinzas na argila, em diferentes concentrações. Os dados coletados mostraram que este compósito tem potencial para ser utilizado comercialmente até uma faixa de 10% de cinzas, sendo que os resultados obtidos mostraram-se relevantes, pois servirão como base para o aprofundamento da pesquisa e possível desenvolvimento de produtos cerâmicos que necessitem de características levantadas neste estudo.

Palavras-chave: materiais cerâmicos; resíduos; sustentabilidade.

Abstract: Brazil generates a significant amount of rice husk waste, considered to have little to no economic value, which can result in negative environmental impacts. This study presents an experiment involving the mixing of rice husk ash with commercial clay, aiming to leverage the properties found in the ash to enhance the material quality. For this purpose, rice husk ash was mixed with clay in predefined quantities to create test samples. These samples were subjected to contact angle and flexural strength tests to preliminarily characterize the effect of the ash on the clay at different concentrations. The collected data demonstrated that this composite material has commercial potential with up to 10% ash content, as the results were relevant and will serve as the foundation for further research and potential development of ceramic products requiring the characteristics identified in this study.

Contribuição dos autores segundo a Taxonomia CRediT

COA: investigation, validation, visualization, writing - original draft MKC: conceptualization formal analysis, writing – review, supervision JTP: visualization, writing – review editing RMG: methodology, validation, supervision FLP: project administration, methodology, resources, writing – review, and supervision

Conflito de interesses

Nothing to declare.

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Keywords: ceramic materials; waste; sustainability.

Resumen: Brasil genera una cantidad significativa de residuos de cáscara de arroz, considerados de bajo o nulo valor económico, lo que puede ocasionar impactos negativos en el medio ambiente. Este estudio presenta una experimentación de la mezcla de cenizas de cáscara de arroz con arcilla comercial, con el objetivo de aprovechar las propiedades presentes en las cenizas para mejorar la calidad del material. Para ello, las cenizas de cáscara de arroz se mezclaron con arcilla en cantidades predefinidas para fabricar muestras de ensayo. Estas muestras fueron sometidas a pruebas de ángulo de contacto y resistencia a la flexión, con el fin de caracterizar preliminarmente el efecto de las cenizas en la arcilla en diferentes concentraciones. Los datos recogidos demostraron que este material compuesto tiene potencial comercial hasta un contenido del 10% de cenizas, ya que los resultados obtenidos son relevantes y servirán de base para profundizar en la investigación y el posible desarrollo de productos cerámicos que requieran las características identificadas en este estudio.

Palabras clave: materiales cerámicos; residuos; sostenibilidad.

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1 INTRODUCTION

Rice is the second most cultivated cereal and the main food staple for more than half of the world's population, occupying nearly 163 million hectares and capable of being grown in various systems and ecosystems (Coêlho, 2021). Data from the United States Department of Agriculture (USDA) for 2020, as reported by Coêlho (2021), indicate that the largest global producers of rice are: China, India, Bangladesh, Indonesia, Vietnam, Thailand, Myanmar, the Philippines, Japan, and Brazil, with Brazil being the only non-Asian country among the top ten producers. In Brazil, almost 95% of the population consumes rice, and more than half do so at least once per day (CONAB, 2015). Rice cultivation is present in all regions of Brazil; however, the Southern Region stands out for its production. The state of Rio Grande do Sul alone accounts for approximately 68% of the country's total rice production (Santos; Tavares, 2018). According to CONAB data from 2016 and 2017, as cited by Santos e Tavares (2018), the gross revenue from rice cultivation in Brazil exceeded R$8.8 million in 2015.

Given that rice is a staple food for the global population and is produced in massive quantities, it is important to consider that both industrial and agricultural activities generate a significant amount of waste that is not inherent to the production objectives. According to Mayer, Hoffman e Ruppenthal (2006), various materials, such as crop residues and agricultural byproducts, or even leftovers from processed products in rural environments, are often disposed of improperly, leading to adverse effects on the environment, such as soil and water pollution and degradation. Furthermore, according to Della, Kühn e Hotza (2005), rice, wheat, and corn represent the largest cereal crops, and their non-utilizable parts—such as leaves, stems, and husks—are generally considered waste. Among these, rice is the largest generator of waste. Additionally, Della, Kühn e Hotza (2005) report that for every ton of rice produced, 23% corresponds to husk and 4% to ash. For comparison, in 2004, global rice production reached 608 million tons, with Brazil producing 13.3 million tons (Della; Kühn; Hotza, 2005).

The primary alternative considered by producers for disposing of this waste is rice husk composting. According to Mayer, Hoffman e Ruppenthal (2006), this method is indirectly employed by most rice producers, as a substantial portion of the generated husk is applied to soil. The challenges of this method include the lengthy decomposition time, which takes approximately five years, the significant amount of methane produced during the process, and, due to the husk's low density, the large volume of material that must be managed. Another common destination for rice husk, according to Mayer, Hoffman e Ruppenthal (2006), is uncontrolled open-air burning, which generates considerable quantities of carbon monoxide and carbon dioxide. Therefore, it is essential to seek solutions that mitigate or resolve the environmental problems caused by inefficient waste disposal, with the aim of promoting sustainable development. The complete reuse of this resource, in addition to addressing environmental concerns, can also serve as a source of income through its direct use as fuel for thermoelectric energy generation or as a raw material for other processes, such as silica production from ash resulting from husk combustion (Mayer; Hoffman; Ruppenthal, 2006).

As a subject for study and experimentation, ceramics are among the most suitable materials for the application of silica in their composition due to their versatility. According to Ashby e Johnson (2011), ceramics possess high durability, are generally hard and brittle, have high melting points, and low thermal expansion coefficients. Moreover, most ceramics are excellent electrical insulators. The size of commercial ceramic components ranges from small parts, such as spark plug insulators, to large nose cones for reentry vehicles. Traditional ceramics are well known, including bricks, tiles, bathroom fixtures, and domestic ware, while technical ceramics are used for bearings, turbine blades, cams, cutting tools, extrusion blocks, nozzles, seals, filters, crucibles and trays, electrical insulators, substrates, and heat sinks for electronic circuits (Ashby; Johnson, 2011).

Among the many discarded materials, it is important to highlight the often-overlooked impact that certain types of waste can have (Palombini; Cidade, 2022), raising sustainability concerns that encompass environmental, economic, and social attributes (Palombini; Cidade; Jacques, 2017). In this context, the role of design is essential—not only as a transformer of form and function but also for its ability to influence economic, ecological, and social aspects (Cidade; Palombini, 2023). According to Pazmino (2007), sustainable design is an increasingly employed alternative to minimize environmental impacts, maximize economic objectives, and enhance social well-being. It also proposes a responsibility to avoid harming current environmental balance and to ensure its preservation for future generations. Additionally, as Papanek (2005) states, design should be an innovative, highly creative, and interdisciplinary tool that responds to human needs. Based on this, the present study aims to explore the incorporation of ash derived from controlled rice husk combustion as a modifying filler in traditional ceramics. Thus, preliminary experimental tests, such as contact angle and flexural strength, were conducted to assess the influence of ash on the material at different concentrations.

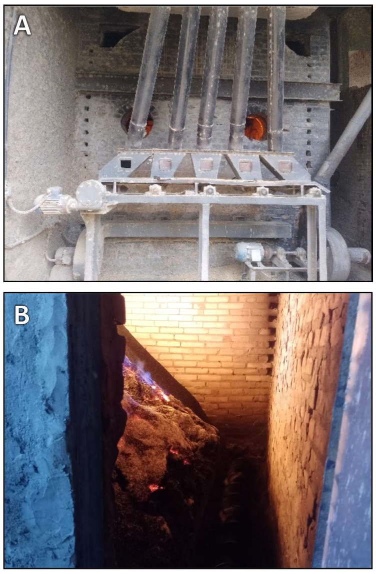
2 MATERIALS AND METHODS

The methodology described below was applied experimentally with the initial goal of exploring the physical properties resulting from the addition of rice husk ash to clay. In order to use ash derived from an actual production process, a sample was collected at a rice mill in Santa Maria, RS (Figure 1). At this mill, approximately 70 cubic meters of rice husk are produced per day (about 12 tons) during the milling process. Of this amount, about 2 tons of husk are burned daily in a dedicated furnace (Figure 1B) to generate heat for rice drying (Figure 1A). This combustion results in approximately 400 kg of ash produced each day, representing an additional cost for the mill due to its large volume and the need for proper disposal. After collecting the ash, the next step was to prepare the materials and define the criteria for constructing the test specimens.

2.1 Materials preparation

To construct the test specimens, a mixture of rice husk ash and powdered clay was prepared, and an amount of water deemed ideal for the mixture was added, as this was found to be the most efficient manual process.

Figure 1 – Processing of rice husk at the mill in Santa Maria – RS: (A) drying furnace and (B) husk burning chamber.

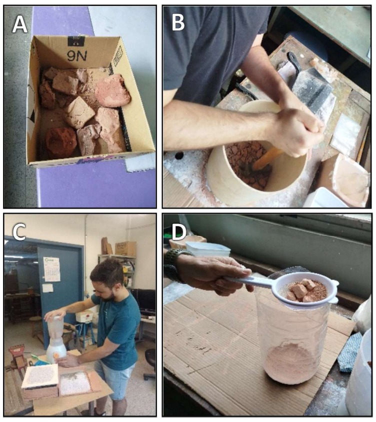


Source: Authors (2025).

2.2 Clay Grinding

The first step in material preparation was grinding the dry clay into powder, as shown in Figure 2. In this process, the dry clay blocks (Figure 2A) were placed in a container and broken into coarse pieces with a hammer (Figure 2B). These pieces were then placed in a blender to be ground into fine pieces (Figure 2C). After grinding, the powder was sieved (Figure 2D) and stored in a plastic container.

Figure 2 – Clay grinding process: (A) raw dry clay; (B) coarse grinding; (C) fine grinding; (D) sieving.



Source: Authors (2025).

2.3 Definition of test specimens

Subsequently, the criteria for constructing the test specimens were established. For this purpose, the ABNT NBR ISO 10545-4:2020 standard was used as a reference, as it outlines the procedures for determining breaking load and flexural strength for ceramic tiles. The guidelines provided by this standard were decisive in defining the dimensions of the test specimens. Based on these criteria, it was determined that the specimens should be approximately 150 mm in length, 50 mm in width, and 10 mm in thickness. These measurements were initially employed to develop a template for producing the test samples (Figure 3), which involved the use of guides. The template was constructed using wooden boards with a thickness of 10 mm, spaced 120 mm apart.

Figure 3 – Clay template construction process: (A) Aligning the guides; (B) Fixing the glides; (C) Ready template with plastic insulation.

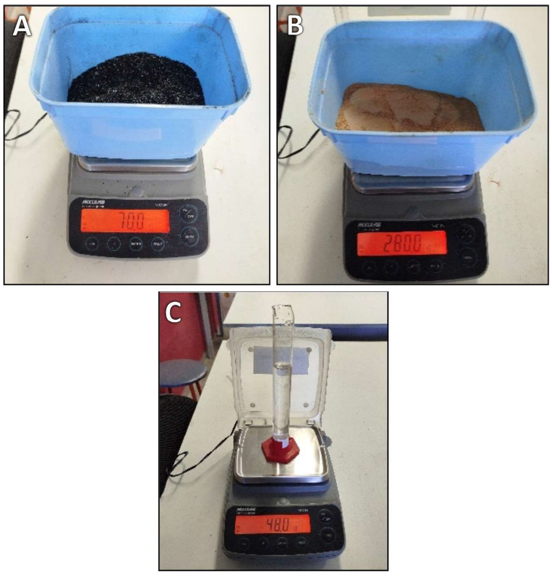
Source: Authors (2025).

With the template ready, the next step was to define the different percentages of each material for the test specimens. Taking into account the sampling required for the tests, it was decided that the test specimens would be constructed in batches of five, starting with 5% ash by mass, and adding 5% of the ash content to the mixture to each batch, until reaching 30%, for a total of six batches.

2.4 Materials mixing and test specimens manufacturing

Once the percentages were defined, the process of mixing the materials and constructing the test specimens began. The first step in the process of constructing a batch of samples was weighing the materials (Figure 4), where sufficient quantities were separated to construct five test specimens.

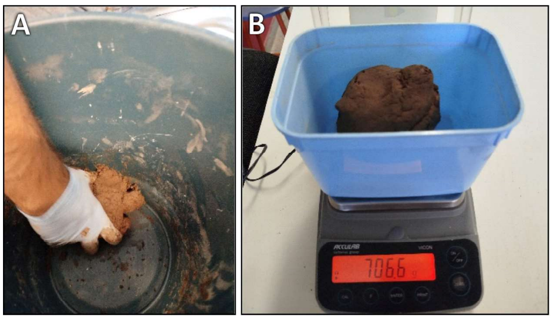
Figure 4 – Weighing: (A) rice husk ash; (B) powdered clay; and (C) water.



Source: Authors (2025).

Next, the mixing process took place manually (Figure 5), where the clay and ash were combined and, subsequently, small amounts of water were added, until the mixture became homogeneous and had the ideal plasticity for molding the samples (Figure 5A).

Figure 5 – Mixing for test specimens: (A) homogenization and (B) weighing.



Source: Authors (2025).

After mixing, the material was weighed again (Figure 5B) and then molded in the template (Figure 6). A PVC roll was used for forming (Figure 6A). After being molded, the material was cut into the five test specimens of the batch, with the aid of a blade (Figure 6B), and each specimen was weighed, as shown in Figure 6C.

Figure 6 – Test specimen manufacturing: (A) shaping the mixture in the template; (B) cutting the mixture into a 5x15 sample; (C) weighing the wet sample.

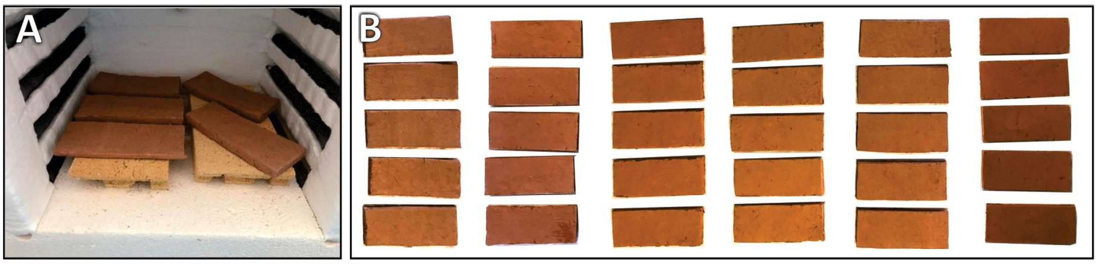
Source: Authors (2025).

After the molding process, the specimens underwent a week-long drying process, after which they were weighed again. After the molding and drying process, they were fired.

2.5 Specimen firing

Finally, the samples were subjected to a firing process (Figure 7) for a period of 10 hours in a muffle furnace (Figure 7A), with a digital controller (Jung brand, model 0213), where the initial temperature was 25°C and was gradually increased to a temperature of 900°C. After firing, the samples were weighed again and with the finished specimens (Figure 7B), the subsequent stage of the research was to conduct the contact angle tests and the flexural strength test.

Figure 7 – Firing process: (A) samples in the muffle furnace; (B) finished specimens.

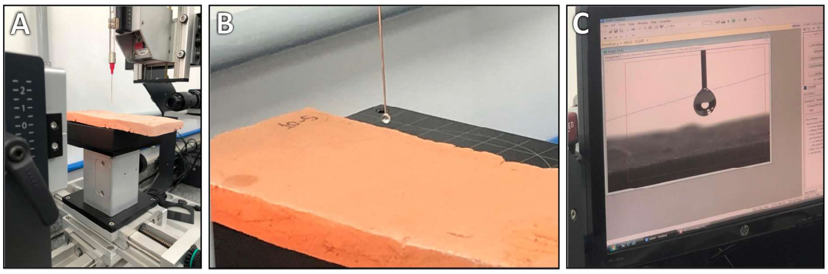


Source: Authors (2025).

2.6 Contact-angle tests

The contact angle test (Figure 8), or hydrophobicity/repellency test, was performed at the Laboratory of Biomaterials in Dentistry (BIOMAT/UFSM). According to Altafim, Gelfuso e Thomazini (2008), hydrophobicity is defined as the repulsion of a water film on a surface. Furthermore, the authors state that when a hydrophobic surface is wet, water tends to form discrete droplets that do not spread across the surface. On hydrophilic surfaces, water is not repelled by the surface, forming a film that covers it (Altafim; Gelfuso; Thomazini, 2008). Based on these concepts, the contact angle test was performed, which sought to demonstrate whether different percentages of each material present in the mixture would make the samples more hydrophobic (more water-repellent) or more hydrophilic (more water-absorbing). The contact angle test consists of dropping a 10 mm droplet onto the material being analyzed with a pipette and then analyzing the angle formed by the droplet in relation to the material's surface. If the angle formed by the droplet is greater than 90 degrees, the surface is hydrophilic, and if it is smaller, it is hydrophobic. For this test, sample number 5 from each batch was selected, and different drops were placed on each.

Figure 8 – Contact-angle test: (A) sample positioning; (B) dripping; (C) viewing the experiment on the camera.



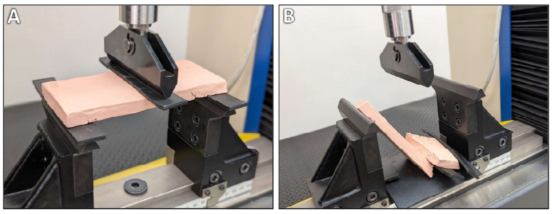
Source: Authors (2025).

2.7 Contact-angle tests

The three-point flexural strength test was performed after the hydrophobicity test, using the same specimens (Figure 9). The test sought to determine whether different percentages of each material make the samples more or less flexurally resistant. The tests were conducted in accordance with ABNT NBR ISO 10545-4:2020 standard and on an EMIC DL2000 device located at the Laboratory for Supporting Product and Process Development and Innovation (LADIPP/UFSM).

The preparation consists of positioning the samples at a distance of 130 mm from each fixed lower support, with a 3 mm rubber sheet between each of the 3 points (Figure 9A). The use of a rubber sheet allows for greater distribution of the applied load until rupture (Figure 9B), without concentrating it, which could lead to a false result.

Figure 9 – Three-point bending test: (A) sample positioning; (B) broken sample after the test.



Source: Authors (2025).

3 RESULTS AND DISCUSSION

In general, the main characteristics analyzed in the experiment were mass loss, material hydrophobicity, and structural strength. Regarding specimen mass, the values were generally inversely proportional to the increase in ash percentage, as shown in Figure 10, with the averages for each composition, for the post-molding, post-drying, and post-firing stages.

Figure 10 – Mass loss of test specimens during the manufacturing process.

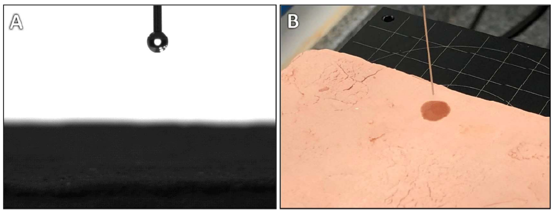
Source: Authors (2025).

Considering the post-molding samples (still wet), the specimens with 30% ash content had approximately 82% of the mass of the specimens with 5% ash. After the drying and firing processes, this difference increased to 66%. In relative terms between each composition, the specimens with 5% ash content lost approximately 20% during drying and 10% during firing, resulting in a total of 28%.

The specimens of the other compositions also lost approximately 10% of their mass during firing. However, this varied significantly throughout the drying process, with the mass loss increasing proportionally to the addition of ash. Consequently, across all experimental processes, the specimens with 10%, 15%, 20%, 25%, and 30% ash content exhibited average mass losses of approximately 29%, 31%, 33%, 37%, and 42%, respectively. This consistent trend clearly demonstrates how the incorporation of ash enhances the material's water absorption capacity, leading to a greater loss of mass throughout the entire experimental process.

Regarding the contact angle test (Figure 11), it was not possible to extract a conclusive result from the initial experiments. After the initial dripping of the liquid (Figure 11A), the samples of all tested concentrations quickly and uniformly absorbed the liquid (Figure 11B), making it impossible to measure a stable contact angle. Subsequently, an attempt was made to repeat the dripping using a pipette and oleic acid, which was extracted from rice. However, these modified trials yielded the same rapid absorption results, further preventing the determination of a clear and reproducible contact angle.

Figure 11 – Contact angle test results: (A) camera capture at the moment of dripping; (B) sample surface after dripping.

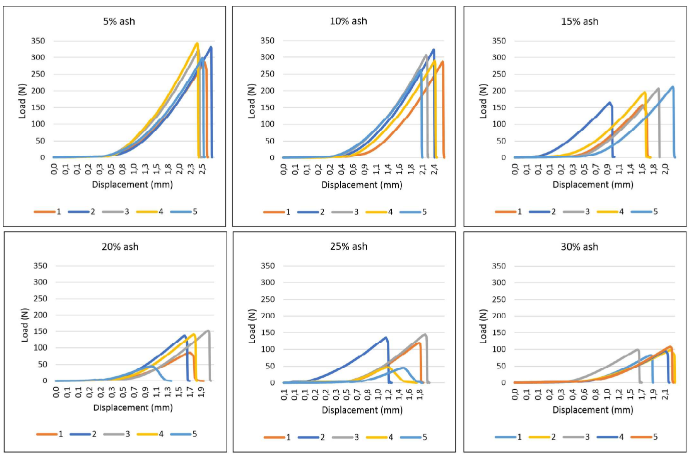


Source: Authors (2025).

The results demonstrate that the material's large pores facilitate the absorption of both polar liquids (such as water) and nonpolar liquids (like oleic acid). Due to its highly porous nature, the ceramic rapidly and completely absorbed the tested liquids, which prevented the accurate measurement of the contact angle formed by the droplet on the surface. This observation strongly suggests that the material is extremely hydrophilic, exhibiting a high affinity for liquids regardless of the proportion of silica (derived from rice husks) incorporated into its composition.

Subsequently, the samples were categorized based on their distinct compositions for the flexural tests. Detailed load-deformation graphs for each individual test, grouped by concentration, are presented in Figure 12. Generally, the obtained curves were consistent with typical 3-point flexural graphs observed in brittle materials. These materials exhibited minimal deformation (approximately 2 mm) prior to failure, with some of this deformation potentially attributable to the compression of the underlying rubber sheet. A small elastic phase, representing recoverable deformation, was discernible within the material. However, beyond the yield stress, the material underwent immediate collapse, indicating a complete absence of a plastic phase (permanent deformation).

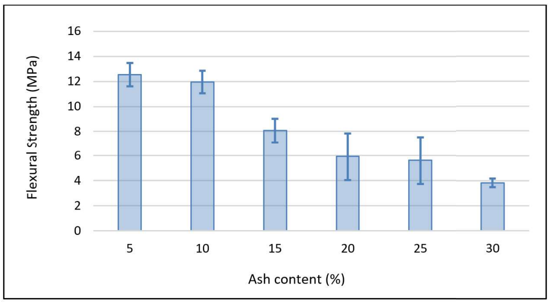
Figure 12 – Results of the bending test, with displacement as a function of the applied load, for each ash composition.



Source: Authors (2025).

It should be noted that, at all concentrations, the load-strain curves were very similar in terms of characteristics (low deformation and absence of a plastic phase). However, the maximum loads obtained varied according to the concentrations. Therefore, Figure 13 presents the average flexural modulus values obtained for each group of samples, divided by concentration, along with the values marked for standard deviation.

Figure 13 – Average flexural strength for each group of samples.



Source: Authors (2025).

According to ABNT NBR ISO 10545-4:2020 Standard, the calculation of the Flexural Strength (R) is calculated based on the relationships between the applied load and the dimensions of the test specimens, according to Equation (1):

$$R = \frac{3 \cdot F \cdot I_2}{2 \cdot B \cdot h^2} \quad (1)$$

where F is the breaking load, expressed in Newtons (N); I_2 is the distance between the support bars, expressed in millimeters (mm), being 130 mm for the experiment; B is the shorter side of the test specimen, expressed in millimeters (mm), or 50 mm in the experiment; and h is the minimum thickness of the tested specimen, along the fracture edge, expressed in millimeters (mm), being 10 mm in this experiment.

In general, as the ash concentration in the samples increases, their flexural strength is reduced. Compared to samples containing 5% ash, samples containing 10%, 15%, 20%, 25%, and 30% ash showed a relative strength of 93%, 59%, 36%, 31%, and 31%, respectively. Furthermore, samples with higher ash content exhibited greater structural variation, indicated by a larger standard deviation of the results. This demonstrates the difficulty in homogenizing ceramic mixtures with higher quantities of ash fillers. Conversely, it can be observed that the use of up to 10% ash does not present a significant change in flexural performance or homogeneity, with the greatest differences occurring in the 10% to 20% ash range, where its inferior performance tends to stabilize.

4 CONCLUSIONS

The reutilization of rice husk, through its ashes, in ceramic products represents a significant approach to add value to a raw material typically considered to have low or even no aggregate value, thus generating environmental, economic, and social impacts. This study presented a preliminary experiment involving the mixture of commercial clay with rice husk ashes, primarily through manual steps. The main difficulties encountered during the specimen preparation process were related to the manual mixing of the materials, a stage where a homogeneous composition of the mixture is essential. A feasible alternative to accelerate the process would be the use of mechanized equipment for mixing.

In terms of physical properties, the material exhibited a mass loss proportional to the increase in ash percentage throughout the process, indicating that a higher ash addition resulted in a lighter final material. Overall, the tested material can be considered highly porous, largely due to the presence of ashes, which led to a lower relative density. Consequently, the material proved to be extremely hydrophilic, absorbing liquids with great ease regardless of the ash concentration and the type of liquid used (water or oleic acid) in the droplet test.

Regarding mechanical properties, it was observed that as the addition of ashes to the mixture increased its porosity, its flexural strength modulus also decreased. A greater influence of the manufacturing process employed was also noted, with mixture homogenization becoming a more critical factor as a higher percentage of ashes was added, as evidenced by the standard deviations of samples with higher concentrations. However, preliminarily, it can be stated that the use of a ceramic mixture with up to 10% rice husk ash content can impart mass reduction properties to the specimens while maintaining similar flexural strength, with little influence from the manufacturing process on its homogeneity.

Despite the time and labor required for the raw material refining process, the potential to transform an input considered of low or no value into a material with higher added value is something that can be considered and appreciated, particularly by leveraging its main properties obtained. Finally, this study aims to be an initial step for new research and experiments, focusing on the reutilization of such materials and the development of new characterization tests, with an emphasis on their possible incorporation into the creation and manufacturing of new products with applied design.

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