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Journal Civil Engineering

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Presentation of Content

As first article we present, *Thermal coating with rigid recycled polyurethane foam as a partial substitute of limestone aggregate* by CERVANTES-RAMÍREZ, Elmer Marcial, TREJO-ARROYO, Danna Lizeth, CRUZ ARGÜELLO, Julio César and GURROLA, Mayra Polett, with adscription at the Instituto Tecnológico de Chetumal, as the next article we present, *Suitability of biochar as supplementary cementitious material (SCM) or filler: waste revalorization, a critical review* by NAHUAT-SANSORES, Javier Rodrigo, CRUZ-ARGÜELLO, Julio César, GURROLA, Mayra Polett, TREJO-ARROYO, Danna Lizeth, with adscription at the Instituto Tecnológico de Chetumalas, as the next article we present, *Hybrid nanocomposite of vanadium dioxide and carbon nanotubes embedded in a gypsum binder for thermal energy storage* by VILLEGAS-MENDEZ, Jesús Roberto, FIGUEROA-TORRES, Mayra Zyzlila, GUERRA-COSSÍO, Miguel Ángel and RUVALCABA-AYALA, Fabián René, with adscription at the Universidad Autónoma de Nuevo León, as the last article we present, *Proposal for a fiber cement panel with the addition of sugarcane bagasse* by MORENO-MARTÍNEZ, Tonatiuh, HERNÁNDEZ-ZARAGOZA, Juan Bosco, MARTÍNEZ-MOLINA, Wilfrido and LÓPEZ-LARA, Teresa, with adscription at the Universidad Autónoma de Querétaro and the Universidad Michoacana de San Nicolás de Hidalgo.

Content

Article

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Thermal coating with rigid recycled polyurethane foam as a partial substitute of limestone aggregate

Recubrimiento térmico con espuma rígida de poliuretano reciclado como sustituto parcial de agregado calizo

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Abstract

This research presents the results of an experimental study about the effect of the rigid recycled polyurethane foam used as a partial substitute of limestone aggregates in the elaboration of cement-based coating, with the objective of reducing the consumption of natural aggregate by replacing it with a recycled material and reducing the thermal conductivity of the coating. The rigid recycled polyurethane foam was crushed to be used as a partial substitute of the fine limestone aggregate in proportions of 15, 20 and 25% in volume, maintaining a cement:sand ratio of 1:3., and its mechanical, physical and thermal properties were evaluated. The mortar of coating with 20% of substitution of recycled polyurethane foam by limestone aggregate, presented a better physical-mechanical and thermal behavior to a laboratory level; therefore, so it was used in real conditions as an exterior coating in a construction prototype elaborated with a wall of blocks and exposed to environmental conditions for a year. The results demonstrated that the modified coating improved its thermal performance by decreasing the interior temperature of the prototype by around 15% compared to the traditional coating, with a difference of 0 to 1.5 °C low, thus maintaining it for most of the year and with relative humidity without significant changes.

Mortar coating, Thermal properties, Limestone aggregate, Rigid polyurethane foam

Resumen

Esta investigación presenta los resultados de un estudio experimental sobre el efecto de espuma rígida de poliuretano reciclada utilizada como sustituto parcial de agregado calizo en la elaboración de recubrimiento base cemento, con el objetivo de reducir el consumo de agregado natural al sustituirlo por un material reciclado y disminuir la conductividad térmica del recubrimiento. La espuma rígida de poliuretano reciclada fue triturada para ser utilizada como sustituto parcial de agregado fino calizo en proporciones de 15, 20 y 25% en volumen, manteniendo una relación cemento:arena de 1:3, y sus propiedades mecánicas, físicas y térmicas fueron evaluadas. El recubrimiento con 20% de sustitución de espuma de poliuretano reciclada por agregado calizo, presento mejor comportamiento físico-mecánico y térmico a nivel laboratorio, por lo que fue utilizado en condiciones reales como recubrimiento exterior en un prototipo de edificación elaborado con muros de block y expuesto a condiciones ambientales durante un año. Los resultados demostraron que el recubrimiento modificado mejoró su desempeño térmico disminuyendo la temperatura al interior del prototipo alrededor de un 15% comparado con el recubrimiento tradicional, con una diferencia de temperatura de 0 a 1.5°C menor, manteniéndola así durante casi todo el año y con humedad relativa sin cambios significativos.

Mortero de recubrimiento, Propiedades térmicas, Agregado calizo, Espuma rígida de poliuretano

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Introduction

Due to the constant climatic changes, new problems arise that require as a solution to reduce the consumption of natural resources, as well as to improve the thermal performance of the materials commonly used in construction, these possible solutions range from the addition of a new material to the replacement of the materials commonly used, in the elaboration of coating mortars (Ponton-Giraldo, 2022; Quiro trujillo, 2022). The term mortar refers to the paste mixture composed of cement, water and fine aggregate, sometimes with additives, which is used in the bonding of bricks for the elaboration of masonry walls or as a covering for the latter. Polyurethane (PU) rigid foam, on the other hand, is a highly spatially cross-linked, thermosetting, hard thermoplastic synthetic material.

At the usual densities for thermal insulation, the foam contains only a small part of the volume of solid matter (with a density of 35 kg/m³), only 3% of the volume is solid matter (Oushabi *et al*., 2017). PU is a material widely used in different sectors, mostly in the construction, refrigeration, automotive and textile industries. PU products in the form of high or low density foam have a wide range of applications. However, due to the high worldwide production and disposal of polyurethane foam from various origins, there is a need for post-use waste disposal processes.

Yang *et al*., 2012, suggests in their research that the most effective means of environmentally friendly disposal is physical recycling, which basically consists of shredding the waste by changing only the physical form as the particles have no reactive activity, leading to a simpler method of operation and a more active application (Yang *et al*., 2012). In the new trends of replacement and addition of building materials, the use of recycling to reincorporate disused materials into the construction process takes a very important role and new recycling methods emerge (Buitrago-Bonilla & Sarmiento-Rodríguez, 2022; Vargas, 2022). For polyurethane foam it is important to employ an effective recycling method due to its properties such as durability, high density, non-flammable, hydrophobic and it is a potential option to be used as a partial substitute for fine limestone aggregate in coating mortars.

For example in Thailand, Tantisattayakul *et al*., 2018, reported that for the use of polyurethane rigid foam waste from dismantled refrigerators and its implementation in lightweight concretes, the best method used is physical recycling, with less environmental and energy impact, however, in terms of insulating properties, they suggest that recycling needs to be improved for its implementation to further decrease the environmental impact in terms of energy consumption during the use stage (Tantisattayakul *et al*., 2018).

On the other hand, Gómez-Rojas *et al*., 2019 evaluated the feasibility of using recycled polyurethane foam (PUR) waste from different industries such as refrigeration and automotive, in building materials, specifically incorporated in base-plaster mixtures, where it was reported that, in the case of waste from the refrigeration industry, they presented good thermal behaviour related to the microstructure of semi-closed hexagonal cells. Such behaviour can be exploited for the improvement of building materials if incorporated as thermal insulation. These polymers degraded around 200ºC without chemical and physical changes, they are inherent materials and did not show leaching. Additionally, only these wastes met the standards for combustion and heating value test which is ideal for interior building cladding (Gómez-Rojo *et al*., 2019).

Other studies reported on lightweight mortars have shown that it is possible to reincorporate waste materials such as expanded polystyrene, thermoplastic waste and paper sludge ash, which contribute greatly to the non-overexploitation of stone aggregates if they are used as a partial substitute for aggregate (Corinaldesi *et al*., 2011; Oushabi *et al*., 2017; Parada Rocha, 2022; Saikia & De Brito, 2012). There are also several studies on the incorporation of PUR as a partial replacement of sand for lightweight mortars, determining important mortar characteristics such as workability, density, air content and the evolution of compressive strength as a function of mortar age. Gadea *et al*., 2010, reported that it is possible to replace the fine aggregate by polystyrene particles for the production of lightweight mortars maintaining a particle size between 0 and 4 mm PU, it is important to mention that in most of the studies carried out, the type of aggregate used is river or natural sand (Gadea *et al*., 2010; Harith, 2018).

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The partial substitution of polymeric waste in mortars and concretes decreases their physical and mechanical properties. Density decreases with increasing PUR substitution (Gutiérrez-González *et al*., 2012). Calderón *et al*, 2018, used PUR as a partial substitute for silica sand in cement mortars, in substitution proportions of 50%, 60% and 75% by volume, where long-term durability was determined by elastic behaviour through fracture analysis subjected to repeated cycles of compressive loading and unloading, where they reported that the fatigue capacity and structural properties of mortars with partial PUR substitution were similar to those of mortars with partial PUR substitution, was similar to that of the reference mortars used in most masonry works, which guarantees long-term durability when placed directly into building elements, even suggesting that these mortars with partial substitution of PU rigid foam waste can be an alternative product to lightweight mortars based on the addition of expanded clay (Calderoń *et al*. , 2018).

In most of the research reported in the literature about the partial substitution of PUR by aggregates for mortars, they perform their tests at experimental level in laboratory to evaluate their physical, mechanical and thermal behaviour, however, very few report the behaviour of mortars with partial substitution of PUR, under real circumstances of application, subject to climatic conditions in the outdoors.

In this study, the substitution of fine aggregate by recycled polyurethane foam from refrigerators up to 25% was established as a maximum parameter, parameters suggested in order not to compromise the compressive strength.

The objective is to evaluate the thermal performance of the mortars under real environmental conditions used as exterior wall cladding and to compare the physical-mechanical properties between a traditionally manufactured mortar and a mortar with partial substitution of the limestone aggregate by PUR, and to reduce the thermal conductivity of the cladding mortar.

2. Materials and methods

2.1 Materials

Portland Cement Composite, type I classified as CPC according to the standard (ASTM C 150, 2012), density of 3.05 g/cm3 and volumetric weight of 1216.22 kg/m3 was used. Locally obtained crushed limestone aggregate, particle size 0 to 4.75 mm and density 1648.31349 kg/m3. Recycled rigid polyurethane foam from waste refrigerators (PUR) with a density of 37.20208 kg/m3. The PUR was collected, crushed and sieved, establishing the maximum particle size of 4.75 mm, based on other research on lightweight mortars.

2.2 Characterisation of limestone fine aggregate and PUR

The fine limestone aggregate was characterised by means of particle size, specific gravity and absorption percentage, according to the standards (ASTM C 128, 2001; ASTM C 136, 2020), respectively, where the volumetric weight of the aggregates and PUR was determined. Due to the light weight of the PUR and to facilitate the application process under real conditions, the substitution with fine limestone aggregate was by volume.

2.3 Specimen dosage

The mixing ratios of the coating mortars with a cement:fine aggregate ratio of 1:3 are shown in Table 1. The mortar mixtures were prepared and classified according to their percentage of fine aggregate substitution as: reference mortar with 0% PUR substitution (CPC), mortar with 15% substitution (C15), mortar with 20% substitution (C20) and mortar with 25% substitution (C25).

Cement: Aggregate				
Properties	**CPC**	**C15**	**C20**	**C25**
Water / Cement	1.07	1.03	1.05	1.04
Fluidity (%)	106	110.9	99.5	103.1
Cement (Kg)	0.42	0.42	0.42	0.42
Fine aggregate (Kg)	1.48	1.26	1.18	1.11
PUR (ml)	0	135	180	225
Water (ml)	321	310	314	311

Table 1 Specimen dosage

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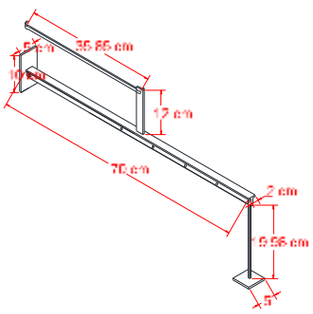
2.4 Compressive strength

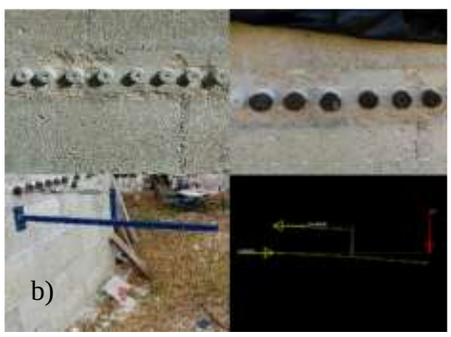
The compressive strength of the samples made with the different compositions was determined according to the standard (ASTM C 109/C109M, 2002). For the test, seven 5 cm cubes of each composition were prepared. The reference fluidity was taken as 110±5 %, which is recommended for coating mortars. The samples were cured in water under ambient conditions for 28 days. An ELVEC E 659-4 press was used for the test.

2.5 Adhesion between mortar and block Wall

Among the most important characteristics for the coating mortar is the bond strength between the coating and the block wall. Therefore, test tests were carried out based on the standard (ASTM C 1857, 2020), for which it was necessary to adjust to a coating mortar. For the purpose of this research, the objective was to find the value of the pullout strength of the mortar adhered to the surface of the block wall, with the support of a mechanical device to produce the pullout failure shown in the image in Figure 1a. The mortar mixtures in the different substitution compositions were applied directly to the block walls where they were cured for a period of 28 days. Subsequently, they were cut into cylinders attached to the wall with a diameter of 2.82 cm, with a length of 1.5 m. Ten tests were carried out for each mortar composition as shown in the image in Figure 1b.



a)



b)

Figure 1 a) (b) Adhesion testing device, (b) Process of testing adhesion of mortar samples on block wall

2.6 Thermal Conductivity and Specific Heat (Cp)

The thermal conductivity study was carried out using a hot plate conductivity meter with guard, based on the standard (ASTM C 177, 2019). Samples of 152 x 127 ± 2 mm cross-section and 25 ± 2 mm thickness were prepared. For each test, a pair of mortar specimens was used, which were traced from vertex to vertex to determine the length of the groove and the thermocouple to be installed in the plate. The resulting dimensions were 76 mm long and 5 mm deep in the centre of each face. Four thermocouples were connected to a 16-channel monitor (Stanford Research System model SR630).

The entire thermal conductivity test process included four runs for each pair of mortar slabs. The first run was performed by supplying the copper resistor with a current of 20 volts for 12 consecutive hours. For the subsequent runs, the voltage was increased to 25, 30 and 35 volts every 24 hours, respectively. The specific heat was determined based on the standard (ASTM C 351, 1999). Initially, the mass and dimensions of each sample to be tested were obtained, which were used in the thermal conductivity coefficient test.

2.7 Prototype construction and monitoring of temperature and humidity under real environmental conditions

Based on the analysis of the results obtained in the laboratory of compressive strength, adhesion and thermal performance of the mortar samples of different compositions CPC, C20, C25 and C30, it was determined that the mortar mixture with PUR substitution classified as C20, presented the best performance, so it was used for the elaboration of the prototypes for in-situ exposure, under real climatic conditions. Two prototypes of blocks for direct placement of the mortar mixtures were made, the reference CPC and C20. Both were applied directly with the specifications of the plan shown in the image in Figure 2, to determine the temperature and relative humidity by means of sensors (HOBO UX100-011 temperature / relative humidity data logger). The construction of the prototypes was carried out with a foundation based on masonry with limestone and dados at the castle foundation points of both prototypes.

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The body of the prototypes was made of block walls and the slab was made of joist and vault. Both prototypes had the same dimensions and were placed facing each other in a mirror image, with a separation of 3 m between them. On top of the slab, a 1:3:7 cement:lime:fine aggregate mix was placed with a thickness of 0.05 m and a 1% slope every metre to prevent water stagnation and filtration inside the prototypes. A 0.07 m thick concrete slab with a rustic finish was made inside each prototype.

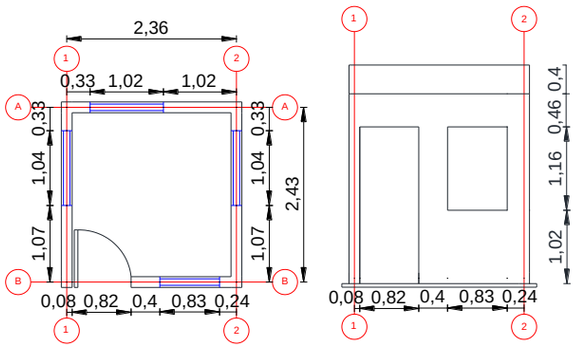

Figure 2 Architectural drawing of prototypes for mortar mix placement and temperature and humidity monitoring

The first prototype was covered with CPC mortar, with a thickness of 0.15 m in a single layer. The walls were previously dampened with abundant water, to prevent part of the moisture in the mixture from being absorbed. The application was carried out on a single day to ensure the same environmental conditions. Alternatively, the coating was applied on the second prototype with C20 mortar at the same thickness. Once the application was completed on both prototypes, the coating was cured in water for 28 days.

According to the standard (ASTM C 1046, 2021), both prototypes must be completely closed, so the windows and doors were sealed with 0.015 m thick wood and the joints as well as the electrical outlets were sealed with polyurethane expanding foam, both inside and outside the prototypes, as shown in Figure 3a and b, exterior and interior, respectively. The monitoring equipment was installed at the centre of each prototype, then closed and sealed to maintain stable conditions inside. The outdoor temperature and humidity sensor (EXT) was installed at a distance of 15 m from the prototypes, using a dht22 sensor measuring temperature between -40 to 125, with an accuracy of 0.5 °C and using a program developed in Arduino software.

Monitoring was carried out under real conditions. Temperature and humidity were recorded every 30 minutes for 24 hours for a period of 12 months both inside and outside the prototypes, thus covering the warm and cold months, to verify the performance of the thermal envelope, also considering that the characteristics of the area have a warm sub-humid climate, where most of the year there are high temperatures and high relative humidity.

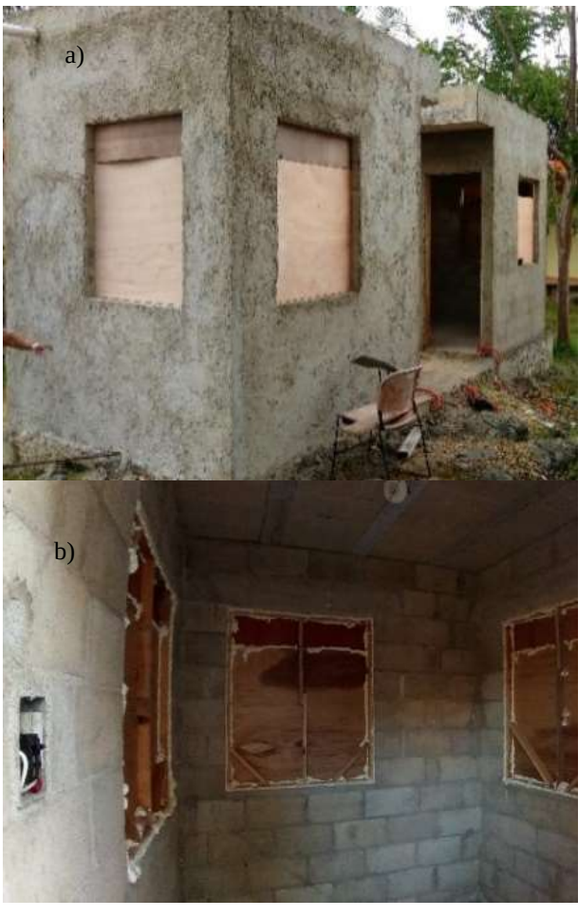

Figure 3 Building prototypes sealed with polyurethane expandable foam. a) Exterior, b) Interior

3. Results and discussion

A coating mortar that is applied outdoors under real climatic conditions must maintain its consistency and workability during the entire application time. To achieve the required flowability, the amount of water had to be higher due to the high degree of absorption of limestone material (Trejo-Arroyo *et al*., 2019) and due to the type of use of the mortar, so the water-cement ratio reached values greater than one.

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As for the volumetric weight values of both fine aggregate and polyurethane foam were determined to ensure that each sample is as accurate as possible to the next, with the same substitution ratio, which were 37.20 kg/m3 and 1648.31 kg/m3, respectively.

The results of the compressive strength of the mortars are presented in the graph in Figure 5. The reference mortar, CPC, achieved an average value of 30.86 MPa. The trend showed that the compressive strength of the mortars decreases with increasing PUR substitution. The strength of mortar C15 decreased by 10.5% with respect to the reference mortar, reaching an average value of 27.62 MPa. In the case of mortar C20, the strength decreased by 21.55%, with an average value of 24.21 MPa and finally in the case of mortar C25 the strength decreased by 24.97%.

Despite the 21.55% decrease in compressive strength in mortar sample C20, it was the mortar considered with the best response to be exposed under real conditions, as it does not significantly compromise its mechanical properties. An overlay mortar must withstand a compressive strength greater than 20 MPa, thus benefiting the lowest aggregate consumption natural limestone.

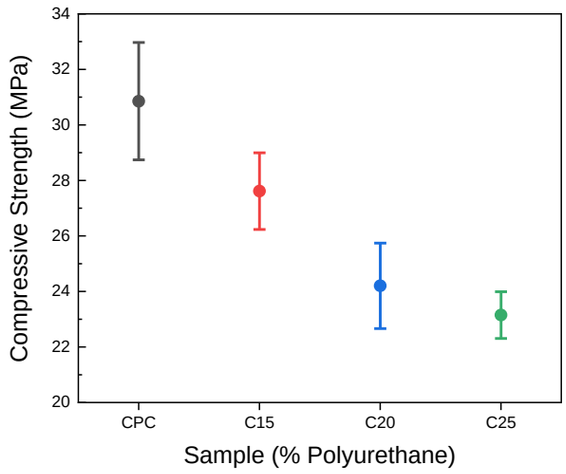

Figure 4 Compressive strength of mortar samples with partial replacement of fine aggregate by PUR

As for the bond strength results, the reference mortar CPC achieved an average value of 11.0461 kg/cm2, as shown in Table 2. These values were obtained after dividing the applied force by the effective contact area of the mortar cylinders with the failure device.

Based on the bond strength obtained from the CPC sample, a decrease in strength of the mortar with 15% PUR substitution of 25.24% was observed. For the case of the C20 and C25 mortars the strength improved considerably compared to the C15 mortar and remained in a stable range with respect to the CPC reference mortar. It is important to mention that, with the addition of the polyurethane foam in the mortar mix, the friction during the placement of the coating in real conditions was considerably reduced and the final finish presented better visual and textural characteristics.

It is also important to highlight that the compressive strength and the adhesion strength between the block and the covering mortar are the most important characteristics of any building envelope, which provides the durability of the material in place and allows it to perform its function correctly, both related, because if one decreases the other is affected, a covering with very high resistance but that does not stay in place due to lack of adhesion, does not work correctly, in the same way a covering that remains adhered, but when hitting it detaches is not considered functional either.

Mortar	σ (kg/cm^2)	Compressive strength (MPa)
CPC	11.046	30.86
C15	8.257	27.62
C20	11.371	24.21
C25	10.694	23.15

Table 2 Results mechanical properties

In the SEM micrograph of Figure 5a, the interaction zone between the cementitious matrix and a PUR particle can be observed. The microstructure of the PUR particles shows a homogeneous interconnected network of pores inside walls with sizes of approximately 50 to 150 µm. According to (Gómez-Rojo *et al*., 2019) it corresponds to a closed cell type structure in which gas is occluded inside the cells, characteristic of a rigid polyurethane foam type. The mechanical properties can be correlated by the presence and amount of PUR.

The surface of the polyurethane particles has a smooth texture as seen in the image in Fig. 5b, which is inferred to reduce the adhesion with the cementitious paste, in addition to its hydrophobic nature.

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The contact surface between the PUR particles and the cementitious mix was analysed and the analyses suggest that the mortar paste coats the polyurethane particles with little adhesion, so that as the percentage of PUR substituted by the limestone aggregate increases, the mechanical resistance tends to decrease.

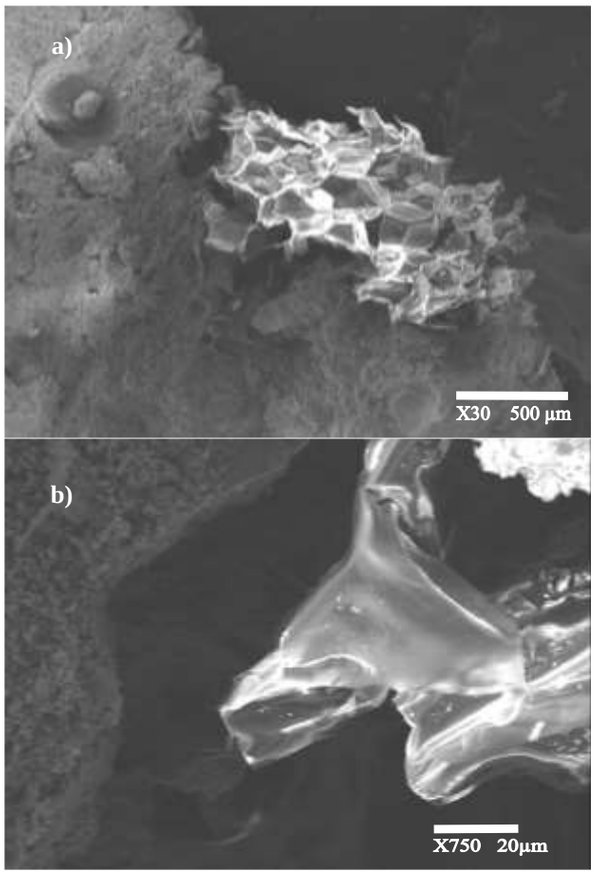

Figure 5 Micrographs obtained by SEM. a) PUR particle surface, b) contact surface between the polyurethane particle and cementitious paste

The results of the thermal conductivity and specific heat tests are presented in Table 3. The general trend indicated that, the higher the amount of fine aggregate substitution by polyurethane foam, the thermal conductivity coefficient tends to decrease, with this trend becoming narrower from 20% PUR substitution onwards. It is important to mention that the thermal conductivity is low but not within the range of the best commercial insulating materials, however, it was decreased compared to traditional coating mortars. In the case of specific heat, it increased with increasing PUR substitution, indicating that more heat is required to raise its temperature; the C15 and C20 mortar samples presented very similar values.

Type of mortar	Thermal conductivity (W/m °C)	Specific heat (kJ/kg °C)
CPC	1.733	0.721
C15	1.711	0.754
C20	1.460	0.752

Table 4 Thermal conductivity results

Figure 6 shows the graph corresponding to the temperature data recorded throughout the year, both for the outside temperature (EXT) and inside the prototypes with the CPC and C20 mortar coating envelope. The months recorded with the highest temperatures were from May to August and it was observed that the EXT temperature was higher than inside both the CPC and C20 prototypes. In September, the EXT temperature started to decrease due to weather conditions, increased rainfall and the arrival of autumn-winter. In addition, the heat flow inside both prototypes also started to decrease, however, as the prototypes were closed and sealed, the temperature remained higher compared to the outside.

If the EXT temperature is compared with the inside temperature of the CPC prototype, the difference is recorded in the order of 0 to 1.8ºC. Comparing the outdoor EXT temperature with the indoor temperature of the C20 prototype, the difference was recorded in the order of 0 to 2.5ºC, which, according to the conditions of the area, of warm sub-humid climate, high temperatures most of the year and high RH, it is noted that a difference of more than 2ºC is very significant which will considerably improve the conditions inside a building.

On the other hand, the results suggest that, in the lowest temperature months recorded, the thermal envelope works in the opposite direction, retaining the temperature inside in both prototypes CPC and C20, however, comparatively the prototype with mortar C20 recorded the lowest temperature in the order of 0 to 1.5ºC during almost the whole year.

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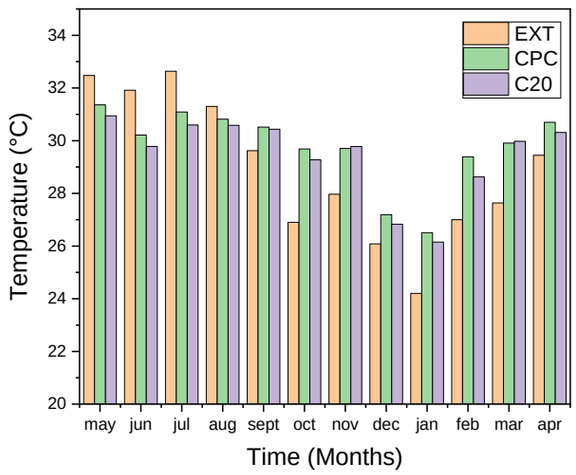

Figure 6 Average temperature record per month, exterior (EXT) and interior of the prototypes with the envelope of CPC and C20 mortar

Regarding humidity, the results of the outdoor (EXT) and indoor data recording of both prototypes with CPC and C20 mortar coating are shown in Figure 7. It shows the monthly average of the three different sensors for the same period of 12 months, it is remarkable how the humidity increases in the months where, in general, rains are more common, it is important to mention that the exterior EXT humidity was lower in the sunniest and warmest months, however, as the months become more rainy, the exterior humidity increased more than the interior of both prototypes, the recording of the humidity inside the prototypes was very similar, only differences between the two were of the order of 0. 5 %, highlighting that unlike the commercial thermal insulators installed in slabs or formwork, this C20 mortar coating did not increase the interior humidity concentration significantly.

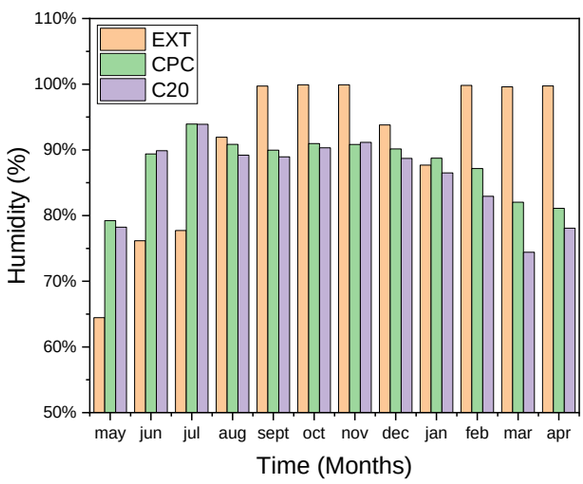

Figure 7 Average humidity record per month, exterior (EXT) and interior of the prototypes with the CPC and C20 mortar envelope

Figure 8a and b shows the average values per month of the minimum and maximum temperature recorded, respectively, for both prototypes CPC and C20. As shown in the graphs, the year-round behaviour of the temperature inside the C20 prototype remained below the minimum temperature recorded in the CPC prototype, both in the minimum and maximum temperature record throughout the year, maintaining a difference between 0 to 1.5°C below. These results suggest that, under real exposure conditions, the modification of the coating mortar mix with the substitution of PUR for limestone aggregate effectively lowers the temperature inside a building.

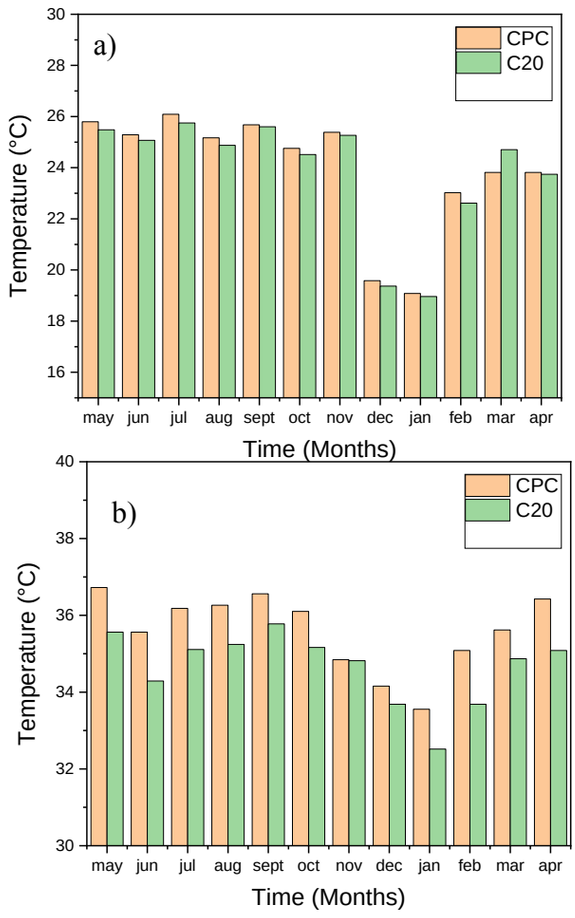

Figure 8 Annual average temperature record. a) Minimum temperature, b) Maximum temperatura

As for relative humidity, the results are presented in the graphs in Figure 9. The average values obtained per month of humidity in both prototypes CPC and C20 are shown, both for the minimum and maximum values, humidity remained constant in both prototypes with differences of less than 0.5 %.

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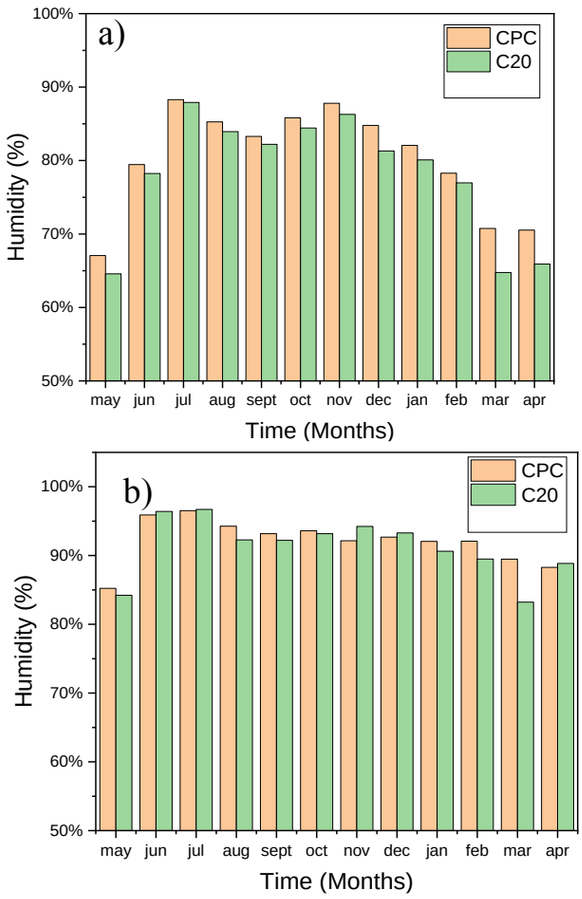

Figure 9 Annual average humidity record, a) Minimum humidity and b) Maximum humidity

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Conclusions

Covering mortars with different percentages of substitution of recycled polyurethane rigid foam (PUR) for fine limestone aggregate were characterised by means of different techniques, in order to evaluate their thermal performance when exposed to weathering under real environmental conditions with high temperatures and high relative humidity most of the year. The results indicated that with increasing PUR substitution content, the compressive strength decreased.

However, from the experimental laboratory tests, it was determined that the coating mortar with 20% partial substitution was the one that obtained the best conditions to be applied in-situ as the envelope of a prototype building and to be exposed to weathering under real climatic conditions. The compressive strength of the C20 mortar was 24.21 MPa and with a bond strength between the facing mortar and the block wall of 11.3710 kg/cm2, very similar to the traditional mortar, which does not compromise the mechanical properties and integrity of the structure.

Regarding the thermal conductivity of the mortar slabs, the results indicated that by increasing the substitution content of PUR for limestone aggregate, the thermal conductivity decreased by 15% compared to that of the traditional mortar, and the specific heat increased, indicating that more energy is required to increase the temperature. The mortar topping envelope with PUR substitution by 20% fine limestone aggregate exposed to real environmental conditions reduced the temperature inside the prototype building by about 1.5°C during most of the year while keeping the humidity constant. Future work could focus on the thermal performance and durability of the mortar with partial replacement of fine aggregate by recycled polyurethane rigid foam applied on the interior walls or roofs as thermal insulation for energy savings in buildings.

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Suitability of biochar as supplementary cementitious material (SCM) or filler: waste revalorization, a critical review

Viabilidad del biochar como material cementante de reemplazo (MCR) o filler: revalorización de residuos, review crítica

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Abstract

For decades, researchers on materials science have highlighted the potential of biochar as a CO_2 adsorption medium and the possibility of its incorporation into other materials to reduce the overall carbon footprint. This present study is a critical review of a selection of articles about biochar potential as a material on the construction industry. Biochar is a promising material in order to mitigate GHG emissions when added to cementitious materials, reducing its carbon footprint through a dual effect: CO_2 sorption and replacement of cement or aggregates. Literature evidenced that replacement ratios of around 2-8 of cement wt% improved or leveled with conventional cementitious composites. However, some recent studies have shown that the incorporation of biochar up to >10% replacement ratios have the potential to improve the composites. Based on this premise, the present review emphasizes on the durability and long-term properties of biochar cementitious composites by providing up-to-date discussions of the studies on the matter and the future perspectives of the research in order to develop more eco-efficient concretes or mortars.

Biochar, Eco-efficient cement, Cementitious composites

Resumen

Durante décadas, investigadores en ciencia de materiales han destacado el potencial del biochar como un medio de absorción de CO_2 y la posibilidad de incorporación en otros materiales compuestos con el fin de reducir su huella de carbono. El presente trabajo es una review crítica compuesta de una selección de artículos enfocados en el potencial del biochar como un material en la industria de la construcción. El biochar es un material prometedor para reducir los gases de efecto invernadero cuando es incorporado a materiales cementantes, al reducir su huella de carbono a través de un efecto dual: absorción de CO_2 y el reemplazo de cemento o agregados. La literatura indica que tasas de reemplazo entre 2 a 8% en peso de cemento desarrollan cementantes con mejores o iguales propiedades a las de un cementante convencional. No obstante, estudios recientes destacan la posibilidad de reemplazar >10% de cemento y obtener compósitos con mejores propiedades. Con base en esta premisa, la presente investigación enfatiza las propiedades relacionadas a la durabilidad y largo plazo de compósitos base cemento con biochar, proporcionando discusiones actualizadas y perspectivas futuras de investigación con el objetivo de desarrollar concretos y morteros con mayor eco-eficiencia.

Biochar, Cementantes eco-eficientes, Compósitos cementantes

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1. Introduction

Concrete is the most abundant composite material available nowadays. Its constituents are mainly cement, water and aggregates -as fillers-, the former has the most important role overall: binding the mixture and providing the characteristic properties of hardened concrete -versatility, strength and durability-. Due to its crucial role in concrete mixtures, the carbon footprint associated with cement production has been extensively analyzed in several studies (Andrew, 2019; Busch *et al*., 2022; IPCC, 2018; Liao *et al*., 2022; Mahasenan *et al*., 2003; Nidheesh & Kumar, 2019; Scrivener *et al*., 2016), ranging from around 0.81kgCO_2-eq to 0.90kgCO_2-eq per kg of cement produced (Bellona Foundation, 2018; Plaza *et al*., 2020; Samad & Shah, 2017; Shen *et al*., 2016). Concerning anthropogenic greenhouse gas (GHG) emissions, cement production contributes to around 5-8% of global emissions owing to its energy intensive production process, mostly related to acquisition of raw materials, calcium carbonate calcination, fuel burning and logistics (Barcelo *et al*., 2014; Miller *et al*., 2021).

One of the foremost solutions that have been proposed in literature to mitigate the above-mentioned environmental impact is the use of supplementary cementitious materials (SCMs) (Siddique & Khan, 2011; Snellings, 2016). Conventional SCMs include blast furnace slag(Amran *et al*., 2021; Lee *et al*., 2019), fly ash (Y. Han *et al.*, 2022; Park *et al*., 2021) and silica fume (Ibrahim, 2021; Tavares *et al*., 2020), however, in recent years other candidates have been explored, such as: agro-industrial waste (Aprianti *et al*., 2015; Chand, 2021; Manan *et al*., 2021; Ramos *et al*., 2022; Siddika *et al*., 2021), livestock manure (Leng *et al*., 2019; Rehman *et al*., 2020), municipal solid waste (Caprai *et al*., 2019; L. Chen, Wang, *et al*., 2022; Ryłko-Polak *et al*., 2022; Tome *et al*., 2020), glass waste (Bueno *et al*., 2020; Ibrahim, 2021), forest and vegetable crops and residue (Martirena & Monzó, 2018; Puga *et al*., 2022; Restuccia *et al*., 2020; Sirico *et al*., 2020) among other organic wastes also known as biomass. The disposing of these wastes traditionally includes their revalorization as fertilizers, open-air landfilling or open-air incineration.

The aforementioned methods of disposal have revealed to significantly disrupt the subsoil and groundwater integrity through phenomena such as eutrophication and liberation of GHG emissions into the atmosphere (Abiriga et al., 2020; Speight, 2020). Parallel to this, biomass is a suitable material for thermal conversion processes such as pyrolysis for energy production, fuel production or element recovery (Fahimi *et al*., 2020; Fiameni *et al*., 2021; Leng *et al*., 2019; Pandey *et al*., 2022; Shashvatt *et al*., 2018).

The residual ashes obtained after thermal conversion of biomasses are commonly outlined under the term biochar. Biochar is the pyrogenic residual waste obtained after the thermochemical conversion of a given biomass (Arif *et al*., 2020; Bergman *et al*., 2022; Vieira *et al*., 2022). Biochar has four main production processes: pyrolysis, hydrothermal carbonization, gasification and torrefaction (Bartoli *et al*., 2020; Roychand *et al*., 2021). Moreover, literature indicates the characteristic calcination temperatures at which biochar is conventionally obtained ranging from 300° to 1000°C (Yaashikaa *et al*., 2020). These temperatures and methods of thermal conversion heavily influence the properties and reactivity of the resultant biochar (Cosentino *et al*., 2018; Tan *et al*., 2020). Generally, these ashes are known to be carbon neutral or have negative impacts through energy production and, in some cases, even function as CO_2 sequestrating agents which result in further GHG emission abatements (Gupta *et al*., 2018; Ibarrola *et al*., 2012).

In view of the favorable environmental performance of biochar it has been proposed in a wide number of studies as an SCM (Jittin *et al*., 2020), as an admixture (Akhtar & Sarmah, 2018; Gupta *et al*., 2018), or as filler *(Cuthbertson et al.*, 2019) in cementitious mixtures in order to produce carbon-neutral composites by reducing their carbon footprint. Considered as an SCM, its abatement potential positions biochar at an advantage point amidst the rest of SCMs which, although possessing a smaller carbon footprint than cement, rarely exhibit abatements.

Furthermore, the advent of clean energies (Buonocore *et al*., 2021) has shifted attention away from coal-burning production industry into more eco-efficient ways of obtaining energy and materials.

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In this context, given its high availability, its prevalent chemical composition and environmentally troublesome disposal, pyrogenic residual ash -biochar- becomes a suitable construction material. Notwithstanding the prospective benefits of biochar as a replacement material in the construction industry, the major challenge for its adoption as a suitable replacement material for cementitious composites lies in its vast mutability and variability. Not only the way various biochars interact and react within a cementitious matrix is widely variable, but also the chemical composition and intrinsic properties differ greatly from biochar to biochar (Suarez-Riera *et al*., 2020). In consequence, in-depth analysis of the reactions and interactions that ensue from the addition or the replacement of cement with biochar is needed, in order to diminish the negative influence on performance and durability of cementitious binders.

Literature indicates that low percentages of substitution have been predominantly explored, whereas relatively high percentages of substitution are generally dismissed due to poor strength-related performance and durability concerns (Danish *et al*., 2021). However, in accordance with standards such as ASTM C618 and ASTM C311, the test procedures to determine pozzolanic activity require a 20% substitution (ASTM C311-18, 2018; ASTM C618-19, 2019). In addition, there is a paucity of data concerning the properties influencing long-term interactions between biochar properties and cementitious materials, indicating an important research gap.

In the present review paper, a critical review of the biochar merits and demerits on cementitious matrixes is outlined, state-of-the-art research from January 2017 to June 2022 concerning properties of biochar cementitious composites is examined from environmental, durability, and long-term performance viewpoints, key indicators of suitability of biochar as SCM are reviewed and future opportunities of research to develop appropriate biochar cementitious composites are discussed throughout the review; authors find it timely to conduct a systematic evaluation of recent advances, challenges, future outlooks and applications regarding biochar as a potential SCM or filler.

Thence, this is a unique review, providing up-to-date discussions on biochar as a functional material in the construction industry, its properties, characteristics, environmental performance, durability perception and long-term performance.

1.1 Selection criteria

There are plenty research works regarding biochar cementitious blends, however, not all of them provide the same level of in-depth discussion and their contributions on long-term performance, environmental performance and durability are limited. In addition, not all of them provide the same level of detail discussing residence time, temperature and thermochemical conversion method. The purpose of this review is to serve as a guide for researchers to assess the feasibility and potential applications of different biochar as supplementary cementitious material by exploring some challenges such as (1) which properties are affected by the replacement of cement with biochar; (2) the potential of biochar cementitious composites to improve ecoefficiency in contrast with regular blends; and (3) the long-term expected performance of composites containing biochar.

The systematic literature search was conducted in two electronic databases: ScienceDirect and Dimensions.ai. The search was limited to publications from January 2017 to June 2022. A combination of the following keywords in conjunction with the Boolean operators AND and OR were used to narrow the scope of the search: biochar, cementitious, carbon footprint, abatement, construction, material, building, SCM, replacement. A supplementary search was conducted by revising the references lists on the articles obtained.

The eligibility criteria was determined by: (i) published in full-text and in English, (ii) high priority was given to articles that discuss the properties of biochar blends in conjunction with their expected long-term and environmental performance, and (iii) articles that discuss the hydration kinetics of biochar blends; meanwhile, the exclusion criteria was determined as follows: (i) review or conference articles, (ii) articles merely focusing on biochar as is and not in cementitious composites, (iii) articles that provide no details on the thermochemical conversion process and the biomass feedstock.

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(iv) articles which include properties or applications of biochar blends which are not suitable for the construction industry -like heavy metal removal or waste water treatment-. A total of 379 items were retrieved from the electronic databases ScienceDirect and Dimensions.ai, 329 and 50, respectively. Thereupon, 41 items were excluded during a preliminary screening due to duplicity, leaving 338 items for the next stage of screening which is based on the eligibility criteria described above, from which 293 items were excluded due to the insufficient information provided regarding thermochemical conversion process or properties covered and shallow discussion about environmental and durability relation or the lack thereof; a total of 45 items were selected for in-depth full-text revision. Full-text revision refined the total of items that adhered to the criteria, resulting in a total of 20 items fit for the present review. The selected items are presented in Table 1.

Article	Proposed use of biochar	Biomass feedstock
(Gupta & Kua, 2018)	Admixture	Mixed wood sawdust
(Dixit *et al*., 2019)	Cement replacement	Mixed wood sawdust
(Gupta, Krishnan, *et al*., 2020)	Cement replacement	Wood wastes and coconut shells
(Gupta, Palansooriya, *et al*., 2020)	Admixture	Sorghum, dairy manure, cotton stalk, mixed wood waste, Vermont biochar, Wakefield biochar, Hoffman biochar
(Tang *et al*., 2020)	Cement replacement	Municipal solid waste
(X. Chen *et al*., 2020)	Cement replacement	Municipal sludge
(Gupta, Muthukrishnan, *et al*., 2021)	Cement replacement	Rice husk and mixed wood sawdust
(Gupta, Kashani, *et al*., 2021)	Cement replacement	Peanut shell
(X. Yang & Wang, 2021a)	Cement replacement	Rice husk
(X. Yang & Wang, 2021b)	Cement replacement	Commercial biochar (Korean)
(Praneeth *et al*., 2021)	Sand replacement	Enhanced poultry litter
(Maljaee *et al*., 2021)	Cement replacement	Olive stone, rice husk and forest residues
(Dixit *et al*., 2021)	e (UHPC)	Marine clay and wood waste
(Sikora *et al*., 2022)	Cement replacement	Wood chip (commercial/Polish)
(X. Han *et al*., 2022)	Binder (AAS)	Residue of pine and cedar
(Castillo *et al*., 2022)	Cement replacement	Poultry litter
(F. Wu *et al*., 2022)	Admixture	Miscanthus (commercial/Dutch)
(Kanwal, Khushnood, Khaliq, *et al*., 2022)	Admixture	Bagasse sugarcane
(Kanwal, Khushnood, Shahid, *et al*., 2022)	Admixture	Bagasse sugarcane
(L. Chen, Zhang, *et al*., 2022)	Admixture	Waste wood

Table 1 Proposed use of biochar in cementitious composites, and biomass feedstock

2. Biochar as a functional material

Biochar is the pyrogenic residue of biomass thermochemical conversion processes where oxygen conditions are limited, during which water evaporation occurs first, followed by the release of volatiles and finally the setting of porous-carbonaceous ashes remains. In the broadest sense, biochar is a charcoal-like solid material typically composed of carbon (C), of porous-carbonaceous nature and low bulk density. From a structural point of view, biochar and activated carbon are similar materials given their porous nature and amorphous carbon content, however, their surface functional groups differ. There are several key factors of the thermochemical process governing biochar properties: temperature range, heat rate, biomass-feedstock, pretreatment or refinement, residence time and pressure (W. J. Liu *et al.*, 2015).

Considering its origin, biochar is a highly available material worldwide thereby reducing stress on the supply chain and exploitation of raw materials. Once obtained biochar needs no special processing and can be utilized directly, nonetheless, pretreatments and refinements might prove useful for targeted applications (Shanmugam *et al*., 2022). Even when considering a pretreatment or refinement process biochar is still considered a low-cost byproduct.

Biochar has long been considered a suitable material for carbon sequestration aimed at reducing GHG emissions, as a matter of fact, the Intergovernmental Panel on Climate Change (IPCC) considers biochar as one of the six methods proposed to permanently mitigate carbon emissions on account of its carbon fixing capabilities -acting as a carbon sink- and negative carbon footprint (IPCC, 2018; Neogi *et al*., 2022).

A noteworthy benefit of replacing cement with biochar in cementitious composites could be defined as ecoefficiency-strengthening; namely, a dual-purpose effect: on one side, the potential to strengthen the composite while decreasing its carbon footprint, on the other, fixing carbon on its microstructure for decades-long storage (Yin *et al*., 2021).

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On the same note, certain biochar properties are of interest for alternative cementitious composites development, such as density, surface area, electrical resistivity, porosity, particle shape, hydrophilicity, and surface functional groups, among others. These properties have a direct influence on mechanical behavior, workability, durability and adsorptive efficiency of composites containing biochar.

In regards to characterization techniques for biochar composites, the present paper focuses mainly on those relevant to cementitious composites development and application; several comprehensive reviews and research titles on biochar extensive characterization -which falls beyond the aims of the present review- have been already published. For the full-frame characterization -including mechanical behavior- of biochar as is, the reader is referred to the comprehensive research papers of Yaashikaa *et al*. (Yaashikaa *et al*., 2020), Shanmugam *et al*.(Shanmugam *et al*., 2022), and the references contained therein.

3. Physical and chemical properties related to durability and long-term performance of biochar composites

3.1 Physical and morphological properties

The replacement of Portland cement with biochar is expected to yield composites with lower densities and diminished compressive strength, mainly due to the porous nature of biochar. These reductions in density and strength impact heavily on the expected durability of the composites altogether. Due to the high temperatures set during thermochemical conversion process of biomass into biochar many physical and morphological properties are defined during this stage of production.

Physical and chemical properties tend to be proxies of durability of the overall composite and provide information about its expected long-term performance. The present section reviews some relevant physical and morphological properties related to cementitious materials and their relation with durability and long-term performance of cement-based composites.

3.1.1 Porosity, particle shape, surface area and density

Porosity, surface area and density are some of the most important physical properties of any material with cementitious applications -SCMs, admixtures or fillers-, as these define their contribution to the potential features and performance of cementitious composites.

Given its porous nature, biochar usually presents a low bulk density and high absorption capabilities, these properties carry over to cementitious composites containing biochar resulting in blends with higher porosity, higher absorption rates and a complex pore network. This transfer of properties has been the major concern for researchers seeking to develop more durable cementitious composites containing biochar, as these properties affect durability and resistance directly; workability and setting times are also affected.

Some authors (Gupta & Kua, 2018; Mrad & Chehab, 2019) consider the high porosity of biochar as beneficial and conceive it as an internal curing agent, consistently supplying water to the composite through further hydration. It differs from conventional water curing methods where water is spread through the surfaces, limiting its effectiveness due to its shallow penetration, whereas biochar acts as a water retention agent inside the matrix. In biochar, porosity owes its variability to the escaping of volatile matter during thermochemical conversion and, like with other properties, the rising of temperature promotes an increase in porosity and surface area.

Dixit *et al*. (Dixit *et al*., 2019) found that the increase in size of biochar -fine, medium and coarse- also influences the size and overall availability of pores. Greater pore sizes serve as bridging points between micropores and mesopores, increasing interconnectivity and allowing water absorption and retention to increase as well.

Other carbon-based materials like graphene and nano-sheets have shown shape-dependent properties and features (Tatrari *et al*., 2021; Yoo *et al*., 2019).

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Notwithstanding, in the case of biochar, a study by Suárez-Riera *et al*. (Suarez-Riera *et al*., 2022) investigated the influence of biochar shape on the mechanical performance of cement-based composites and based on their results concluded that sphere-shaped, rod-shaped or sheet-shaped biochar have no significant influence on the mechanical behavior or even workability, unlike graphene, nanotubes or nanosheets. Nonetheless, the addition of biochar -regardless of the particle shape- improved compressive strength, flexural strength and fracture energy more than 20% as compared to reference.

Density is interrelated with porosity, as porosity values increase density values decrease; likewise, when particle size decrease surface area experiences an increase. Yang & Wang (X. Yang & Wang, 2021a) and Praneeth *et al*. (Praneeth *et al*., 2021) reported that an increase in biochar proportion is strongly correlated with a decrease in the density of the resultant composite. By itself, a reduction in bulk or skeletal density is not a deleterious effect, however, it suggests the potential applications of biochar composites as lightweight energy-efficient composites.

Achieving denser biochar composites is possible up to a certain proportion of biochar addition/replacement -which is highly dependent on biomass feedstock and the calcination process-, according to Akhtar & Sarmah (Akhtar & Sarmah, 2018), if this proportion is exceeded then the hydration products are insufficient to fill the pores of the biochar and eventually lead to a more porous and brittle composite instead. Low density composites tend to exhibit low strength, affecting its durability and long-term performance.

Surface area is an important property to evaluate as it influences hydration products growth, workability, and is positively correlated with density and porosity. Surface area has direct incidence on adsorptive efficiency -carbon fixing-. High calcination temperatures and biomass feedstock influence porosity, surface area and contaminant fixing capacity.

The results obtained by the research of Castillo *et al*. (Castillo *et al.*, 2022) indicates that higher biochar substitution proportions can yield positive results in mechanical performance: at 90 days testing every proportion of substitution -10, 15 and 20 wt%- achieved higher strength, from 10% to over 32% increases; in contrast with what several other investigations have reported suggesting lower substitution proportions (Qin *et al*., 2021; Rodier *et al*., 2017); this is mainly attributed to the high calcination temperatures -600°C to 800°C- related to other investigations, since this increase in temperature is positively correlated to an increase in porosity and surface area, thus improving properties like water absorption, reactivity and bonding/bridging in the cementitious matrix.

3.1.2 Transport properties

In cementitious composites several transport properties are important, namely: water absorption, permeability, thermal conductivity, electrical resistivity, sulfate resistance and chloride diffusion.

Biochar presents a complicated pore network -regardless of biomass feedstock-, which contributes to diffusion effects and entrapment of contaminants. Its low thermal conductivity and high porosity allow it to be considered as an insulating agent. Greater porosity leads to a decrease in thermal conductivity -biochar as is possesses a low thermal conductivity-, turning it into a better thermal insulation agent (Tan *et al*., 2020). This is an important property when developing lightweight composites or concretes.

In the investigation of Cuthbertson *et al*. (Cuthbertson *et al*., 2019), thermal conductivity and acoustic properties were evaluated in concretes with biochar addition. Since thermal conductivity and acoustic properties -sound absorption- are associated with porosity it is expected that biochars have the potential to act as thermal insulators and sound dissipators. The authors reported a linear decrease in density of concretes in relation with higher biochar addition, however, substitutions up to 12% were identified to be optimal for maintaining integrity and exhibit good sound and thermal insulation. These results are directly translated into energy savings due to thermal-efficiency of buildings employing these biochar-amended concretes.

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Hydrophilicity -or water absorption and retention- is another important property worth evaluating in biochar cementitious composites. The findings of Praneeth *et al*. (Praneeth *et al*., 2021) indicate a correlation between biochar content and water absorption and void content on composites. However, some other authors (Maljaee *et al*., 2021) reported a decrease in capillary absorption, this effect can be attributed to the filler effect -which is linked to particle size- filling the pores of composites with hydrated products blocking to some degree the superficial penetration of water.

The work of Gupta & Kua (Gupta & Kua, 2018) evaluated the effect of pre-soaked biochar acting as water reservoirs -internal curing agent- in cement mortar It was found that pre-soaked biochar improved the degree of hydration, proving the hypothesized gradual release of trapped water for internal curing even when the external conditions stopped supplying water.

Free chloride ions are a major threat to reinforced concrete due to the de-passivation effect, leading to corrosion and the formation of solids on the surface of the reinforcement. This volume increase derives in internal stresses and the eventual cracking of the concrete layer. Phases of cement like C_3A and C_4AF are known to interact chemically with free chloride ions.

In the light of it, one of the most concerning deleterious phenomena associated with chloride ingress is the conversion of AFm phases to Friedel's salt and the C-S-H dissolution due to this ion exchange in a NaCl rich environment eventually leading to diminished mechanical and durability performances (Glasser *et al*., 2005). Gupta *et al*. (Gupta, Muthukrishnan, *et al*., 2021) evaluated sulfate resistance and chloride diffusion in biochar cementitious composites, crucial transport properties for durability.

The findings of the study suggest that biochar addition helps to preserve strength in an 8-11% proportion even after 120 days of exposure to a NaCl-medium. In the case of sulfates expansion experiences a reduction in the order of 60-68% in contrast with the references. Both cases correspond to a 1-2 wt% mixed wood and rice husk biochar substitution.

As specimens age the free water content decreases and the hydration products fill some of the available pores leading to an increase on the electrical resistivity property, as evidenced by the study of Yang & Wang (X. Yang & Wang, 2021a) in which the electrical resistivities of all specimens increased with time and the more replacement proportion an even greater electrical resistivity was achieved. In contrast with the reference specimen, 2 and 5 wt% replacement derived in a 4.4% and 13.8% increase at 28 days. In the same note, a study carried out by Ram *et al*. (Ram *et al*., 2022) identified the effect of interconnectivity of capillary pores on chloride transportation and identified that due to the formation of secondary hydration products the pores are clogged and the interconnectivity reduced, thus a decrease in chloride diffusion is expected.

3.1.3 Workability

The works of Cuthbertson *et al*. (Cuthbertson *et al.*, 2019) and Maljaee *et al*. (Maljaee *et al*., 2021) indicate that manual mixing could derive in poor homogenization due to the agglomeration tendency present in biochars; coupled with its hydrophilicity, the setting times and flow test are affected in a deleterious manner since biochar tends to have a high absorption capacity depriving the paste of water during mixing. The studies carried out by Tan *et al*. (Tan *et al*., 2020) and Chen *et al*. (X. Chen *et al*., 2020) also reported a reduction in fluidity of around 3% for 1% addition to 36% for 10% addition and an inverse relation between water absorption and fluidity of fresh paste, respectively.

In both studies a common trait is identified: the finer biochar particles present a greater reduction in workability as compared to coarse biochar particles. In all the previously mentioned studies biochar addition or substitution has demonstrated to have a greater influence on workability, setting times and flow of fresh pastes which ultimately leads to a higher w/b ratio, pre-soaking of biochar or the use of superplasticizer to compensate this loss of fluidity.

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3.1.4 Filler effect

While pozzolanic activity refers to chemical reactivity through $Ca(OH)_2$ consumption (M. Wu *et al*., 2021), latent hydraulic activity refers to materials with high contents of CaO and possibly SiO_2 self-reacting with water to form C-S-H gel or C-A-S-H compounds *(Sivakumar et al*., 2021). Both types of reaction develop more hydration products -even at older ages- which is beneficial to the cement paste.

On its part, filler effect is characterized by being inert and non-reactive on its own; nevertheless, filler effect favors the development of a more densified microstructure through two mechanisms: firstly, the filling of interstitial spaces between cement grains, thus promoting densification; and secondly, an increase in the surface area available for the nucleation of hydration products and C-S-H gel. Normally, filler effect is more evident during the early stages of hydration, but its effects have been confirmed in extended periods of study as long as 120 days (Gupta, Palansooriya, *et al*., 2020). Filler effect can coexist with either pozzolanic or latent hydraulic behavior, in fact, most SCMs present filler effect at some point regardless of the nature of the material reaction -either pozzolanic or latent hydraulic-.

Several studies have considered biochar as inert fillers or in conjunction with other SCM's, as well as evaluated its pozzolanic potential by $Ca(OH)_2$ consumption (Asadi Zeidabadi *et al*., 2018; L. Chen, Wang, *et al*., 2022; L. Chen, Zhang, *et al*., 2022; Gupta, Kashani, *et al*., 2021; Gupta, Muthukrishnan, *et al*., 2021). Biochar is able to present pozzolanic potential or latent hydraulic activity depending upon its original biomass feedstock, chemical composition, amorphous phases and surface functional groups, whereas, its filler potential is determined merely by its fineness and the agglomeration of the biochar particles in the fresh paste.

3.2 Chemical properties

Evaluation of chemical properties of biochar are of vital importance to determine the overall performance of composites, due to these properties directly influencing the reactivity, functionality, and ultimately, potential application of biochar in cementitious composites.

3.2.1 Elemental and chemical composition

The main determinant factors of biochar chemical composition are the biomass feedstock composition itself and the pyrolysis conditions (Vassilev *et al*., 2013). The basis of chemical properties are stability and reactivity. Furthermore, the pozzolanic reaction or hydraulic reaction are dominated by the content of SiO_2+Al_2O_3+Fe_2O_3 and CaO oxides, respectively. In materials, stability is conceived as the condition of materials to not react with the environment during conventional use and preserve its properties during its expected service time. Spokas (Spokas, 2010) identified a relation of O/C molar ratio to reliably predict stability in biochar, based on their results a ratio of < 0.2 are the most stable, with an estimated service time of 1000 years; whereas, a ratio between 0.2 and 0.6 offers more variability, oscillating between 100 and 1000 years of service life; lastly, ratios > 0.6 can preserve properties for over 100 years. O/C ratio also denotes the polarity and hydrophilicity of biochar (Jiang *et al*., 2019). H/C ratio has been linked with aromaticity of carbon-based materials, is subject to variability due to thermochemical temperature and is inversely related with carbon content -to a low H/C ratio corresponds a high carbon content- (Sumaraj *et al*., 2020). These two ratios area closely related.

Thereby, the determination of biochar elemental composition is a top priority when considering its application on the construction industry, given the importance of stability of any component of cementitious matrixes in order to prevent deleterious reactions like alkali-silica reaction (ASRs) or alkali-carbonate reactions (ACRs), which can lead to cracking and fracture over the span of several years of service (Adams & Ideker, 2020; Leemann *et al*., 2022). For elemental composition characterization the most used techniques are ICP-OES, ICP-MS and CHNS analysis, extremely useful to determine the ratio of heavy metals in biochar composition. On the other hand, XRF is the most used characterization technique to determine the oxides in a material as a ratio of mass.

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In the selected studies several biomass parent feedstocks were identified: Mixed wood wastes (MWW), coconut shells (CS), dairy manure (DM), sorghum (SOR), cotton stalk (CST), algae (AL), municipal solid waste (MWS), municipal sludge (MS), rice husk (RH), peanut shell (PS), commercial biochar (B), poultry litter (PL), olive stone (OS), marine clay (MC) and bagasse sugarcane (BS), among others; certainly, the variability of chemical compositions is enormous.

Biochar	O/C	H/C
MWW300 (Gupta & Kua, 2018)	0.41	--
MWW500 (Gupta & Kua, 2018)	0.08	--
MWW500 (Dixit *et al*., 2019)	--	0.03
CS500 (Gupta, Krishnan, *et al*., 2020)	0.27	0.05
MWW500 (Gupta, Krishnan, *et al*., 2020)	1.27	0.05
SOR500 (Gupta, Palansooriya, *et al*., 2020)	0.28	0.06
SOR600 (Gupta, Palansooriya, *et al*., 2020)	0.21	0.05
AL500 (Gupta, Palansooriya, *et al*., 2020)	0.46	0.05
CST210 (Gupta, Palansooriya, *et al*., 2020)	1.23	0.12
CST250 (Gupta, Palansooriya, *et al*., 2020)	1.04	0.11
CST290 (Gupta, Palansooriya, *et al*., 2020)	1.00	0.12
DM600 (Gupta, Palansooriya, *et al*., 2020)	0.88	0.08
MS300 (X. Chen *et al*., 2020)	1.22	0.13
MS500 (X. Chen *et al*., 2020)	1.96	0.05
MS600 (X. Chen *et al*., 2020)	1.92	0.01
RH (Gupta, Muthukrishnan, *et al*., 2021)	1.37	0.05
MWW (Gupta, Muthukrishnan, *et al*., 2021)	0.21	0.03
PS (Gupta, Kashani, *et al*., 2021)	0.64	0.05
PL (Praneeth *et al*., 2021)	1.70	0.05
OS (Maljaee *et al*., 2021)	0.27	0.03
MWW (Maljaee *et al*., 2021)	0.25	0.04
RH (Maljaee *et al*., 2021)	0.82	0.05
MWW (Dixit *et al*., 2021)	--	0.04

Table 2 O/C and H/C ratios for different biochar in selected studies

Evidently, as can be seen on Table 2, even sharing the same parent biomass feedstock no two biochars are the same, this is attributed to differences in pyrolysis process, pretreatments or even geographic availability, Certain biochars like RH and PL share the same trend -regardless of parent biomass-, in relation with SiO_2 and CaO ratios, respectively.

Literature has evidenced the higher the thermochemical conversion temperature the more carbon content, and thus a lower O/C ratio, indicating a more reliable biochar. Moreover, the highest H/C ratios correspond to the lowest temperatures, indicating a less aromatic biochar, in other words, less stable carbon in biochar.

Increasing thermochemical conversion temperature has been proven to decrease O/C and this is also evidenced by an adsorption mechanism shift from cation exchange (CEC) to physisorption, which affects its adsorption capabilities; however, this effect is moderately ameliorated due to the increase in surface area associated with elevated temperatures (Enders *et al*., 2012; Rafiq *et al*., 2016). Biochar yield is also associated with O/C and H/C ratios; the higher the temperature of thermochemical conversion the less biochar yield is obtained, thence, while O/C and H/C ratios decrease so does yield proportion.

3.2.2 Pozzolanic reaction

Pozzolanic reactivity is evaluated by $Ca(OH)_2$ -calcium hydroxide- consumption, the most widespread tests to measure this reactivity are the Frattiini test (BS EN 196(5)), the modified Chapelle test and the pozzolanic activity index (ASTM C311); one noteworthy addition is the R3 method to determine reactivity of SCMs (Al-Shmaisani *et al*., 2022; Avet *et al*., 2016, 2022; Blotevogel *et al*., 2020). When the hydration product $Ca(OH)_2$ reacts with the amorphous silica of the SCM in the matrix of cementitious materials to produce C-S-H or C-A-S-H, it can be established that a pozzolanic reaction is taking place. The amorphous phase of silica is of utmost importance since non-soluble silica would not react with calcium hydroxide, which is the case of quartz -high content of crystallized SiO_2-, considered an inert filler.

Tang *et al*. (Tang *et al*., 2020) carried out a study in which the pozzolanic potential of municipal solid waste biochar was evaluated through the calcium hydroxide consumption test -the modified Chapelle test-, in comparison with other commonly used SCMs -Fly ash and GGBFS-. Their results suggest that MSWI biochar is less reactive than FA and GGBFS and confirmed the late age filler effect of biochar due to a poor mechanical performance during the early ages but a comparable strength after 90 days.

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On a similar note, Liu *et al*. (W. Liu *et al*., 2022) proposed bamboo biochar as SCM, having an SiO_2 of >44%, an Al_2O_3 of 15% and Fe_2O_3 of >9%, which equal a sum of >68%, shying away from the 70% requirement established by the ASTM C618 standard for a Class N pozzolan. Moreover, semi-quantitative analysis was performed on the XRD spectrum obtaining the ratio of crystalline phases vs. non-crystalline phases, 59.87% and 44.10% respectively, which directly influences the reactivity of bamboo biochar. In this study, replacement ratios of 0.2, 0.4, 1, 2, 3 and 4 wt% were evaluated and provided excellent performance in terms of strength with an increase for all replacement ratios in comparison with reference.

3.2.3 pH

Serviceability of cementitious composites is affected by numerous parameters such as strength, density, corrosion, carbonation, environment, humidity, chemical exposure, among others. When SCMs, fillers and admixtures are considered the hydration process and the internal reactions tend to become very complicated, due to the modified reactions between C-S-H gels, available $Ca(OH)_2$ and the properties of the material added to the mixture. pH is a useful parameter to determine the durability of cementitious composites through its key role in deleterious phenomena such as carbonation and steel corrosion; an acidic environment facilitates ion exchange and decalcification of hydration gels, thus cementitious composites are prone to corrosion, alkali-silica reaction (ASR) and carbonation (H. J. Yang *et al*., 2021; Zhang *et al*., 2021). Therefore, pH of biochar is an important property to evaluate biochar-cementitious matrix compatibility and long-term performance prediction agent. Low pH values tend to develop retarding effects and decalcification -detrimental effects for cement hydration-, on the other hand, high pH values are associated with a proper strength development and reinforcement steel protection in composites (Kochova *et al*., 2017). Table 3 presents a recollection of the biochar pH values from the selected studies and their respective calcination temperature.

Reference	Biomass feedstock	Temperature (°C)	pH
(Gupta, Palansooriya, *et al*., 2020)	Sorghum	500	7.43
	Sorghum	600	9.62
	Dairy manure	600	9.84
	Cotton stalk	210	5.82
	Cotton stalk	250	5.87
	Cotton stalk	290	6.33
	Mixed wood waste	500	10.14
	Algae	500	10.24
	Vermont biochar	--	9.61
	Agricultural biochar	--	9.36
	Horticultural biochar	--	9.61
(X. Chen *et al*., 2020)	Municipal sludge	300, 400, 500, 600	7.38
(Gupta, Kashani, *et al*., 2021)	Peanut shell	500	8.17
(X. Han *et al*., 2022)	Pine and cedar	760	10.3
(F. Wu *et al*., 2022)	Miscanthus	250	5.2

Table 3 Biochar feedstock, thermochemical conversion temperature and pH in selected studies

As is the case with other properties, temperature is a defining factor for pH values in biochar, Table 3 corroborates that higher temperatures of thermochemical conversion are positively correlated with higher pH values; however, most of pH values of biochars in the selected studies are above 7, indicating a mildly alkaline addition or substitution.

Some authors evaluated the pH values of composites instead of biochars. Dixit *et al*. (Dixit *et al*., 2019) evaluated the pH of cement pastes with biochar replacement at 2-8 cement wt% proportions and their results suggest negative significance between biochar substitution and alkalinity of the overall cementitious matrix; reference paste exhibited a pH value of 12.80 while 2, 5 and 8% substitution showed values of 12.75, 12.60 and 12.10, respectively. Moreover, Chen *et al*. (X. Chen *et al*., 2020) also evaluated the correlation between biochar pH and the biochar cementitious blend pH, based on their results no significant correlation was identified, biochar pH (7.38) showed negligible effect on modified cementitious pastes, exhibiting pH values in the order of 8.03-10.47 with replacement ratios of 2-4 cement wt%.

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The study of Gupta *et al*. (Gupta, Kashani, *et al*., 2021) corroborated the negligible effect of biochar addition or replacement in cementitious composites when the replacement ratios are low (< 3 wt%), regardless of the pH value of biochar as is. Wu *et a*l. (F. Wu *et al*., 2022) obtained pH values for Miscanthus biochar which exhibited marginally acid values: 5.1-6.3, whereas the pH values of biochar cement pastes were alkaline: 11.4-12.5 with addition of biochar at ratios of 1, 1.5 and 2.0 vt%. Meng *et al*. (Meng *et al*., 2021) carried out a study to determine the effects of different pretreatments for raw biomass feedstock on the properties of yielded biochar.

3.3 Mechanical properties

Replacement ratio played a determinant role in the development of mechanical properties of biochar cementitious composites. In a study using sludge biochar, low proportions (< 8%) of replacement or addition volumes biochar presented an internal curing effect and improved the compressive strength in magnitudes of 5% up to 29% (X. Chen *et al*., 2020). Some other findings follow a different trend: regardless of the type of parent biomass compressive strength decreased with a corresponding increase in biochar replacement (Maljaee *et al*., 2021). Nevertheless, recent findings with PL used as parent biomass feedstock, indicate a higher substitution potential -as high as 20%- and still develop a composite with equal or improved properties in comparison with reference cementitious blends (Castillo *et al*., 2022). In the aforementioned study, three ages were evaluated for compressive strength: 7, 28 and 90 days; in the first 7 days all substitution ratios showed a higher strength than reference, at 28 days reference blend surpassed the 20% replacement blend by a hefty 40%, while marginally surpassing the 10% replacement blend; lastly, at 90 days every replacement ratio surpassed reference and the 10% replacement blend obtained the best results overall.

4. Discussion and opportunities for future research

Biochar has proven to possess a great number of properties such as low skeletal and bulk density, contaminant adsorption, hydrophilicity, among others; these properties make it an attractive SCM or filler, for different applications in the construction industry.

Although several studies highlight the deleterious effects on strength development and densification of cementitious composites containing biochar, its carbon-sink properties and low density could be approached from a different viewpoint. The findings of Cuthbertson *et al*. (2019) are promising for biochar composites as heat and sound insulators. X. Han *et al*. (2022) proposed biochar as amendment agent for alkali-activated slags to further improve coral sands in soils. Some other findings support high substitution levels with an equal or higher strength development capability (Castillo *et al*., 2022; Ofori-Boadu *et al*., 2021), positioning biochar as a potential SCM or filler, with either pozzolanic or hydraulic potential. Biochar has also been proposed as a key component of pervious concrete (Qin *et al*., 2021; Tan *et al*., 2022; Xie *et al*., 2021), while some authors have proposed pyrolyzed char as bacteria carriers in autogenous-healing concretes or cement-based materials (Kanwal, Khushnood, Khaliq, *et al*., 2022; Kanwal, Khushnood, Shahid, *et al*., 2022).

Another positive outlook of biochar incorporation to cementitious composites is the reduction of the overall carbon footprint, as several studies have shown (Guo *et al*., 2022; Igalavithana *et al*., 2020; Tan *et al*., 2022).

The main findings of the present review were as follows:

- Biochar dosage and parent biomass feedstock played a critical role in the overall properties and strength development of the composite.
- Dosages of over 10-20% demonstrated to increase compressive and flexural strengths.
- Incorporation of biochar reduced the composite density; however, it acted as an internal curing agent.
- Dosage 2-8% cement replacement enhanced water absorption, increased capillary porosity and decreased water penetration due to saturation.
- Biochar incorporation to cementitious blends resulted in an abatement of GHG emisiions for 59 to 65 kg CO_2-eq for each tonne of produced composite.

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The abatement potential of biochar containing composites -carbon footprint reduction- needs to be examined at length in future studies, due to the enormous variability existent between biochar from different parent biomass.

Greater focus should be placed on developing studies based on the durability issues related to biochar-mended composites mentioned in the selected studies in the present review.

To the best of our knowledge there are no studies which statistically correlate the thermochemical conversion parameters such as temperature, heating rate and residence time with certain properties of biochar related to durability in cementitious composites such as elemental/chemical composition, O/C and H/C ratios, pH, density, porosity, surface area and mechanical properties to establish the statistical significance of these properties in relation to long-term expectation of biochar cementitious composites in terms of durability and as carbon sinks.

Notwithstanding the lack of research about this particular subject, it can be inferred that biochar-amended composites pose as promising materials for the development of novel cement-based materials, such as pervious concrete, water purification, heavy metal removal, soil stabilization, bacteria carriers, heat and sound insulator, among others; while simultaneously acting as carbon footprint reduction agents. Assuredly, further investigations are needed to provide more precision and certainty over biochar-containing composites performance.

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Hybrid nanocomposite of vanadium dioxide and carbon nanotubes embedded in a gypsum binder for thermal energy storage

Incorporación de un nanocompuesto híbrido de dióxido de vanadio con nanotubos de carbono en pastas de yeso para almacenamiento térmico

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Abstract

This investigation studied the heat storage capacity of a gypsum blinder with a hybrid nanocomposite (NH) of vanadium dioxide and multiwall carbon nanotubes (VO_2/MWCNT). The influence of the NH in the hydration kinetics and hydrated products was determined. The effect of the incorporated amount of NH in the wettability, mass loss by humectation-drying cycles, thermal conductivity, specific heat (Cp) and gypsum thermal performance at 40 °C was determined. Characterization techniques exhibited that the presence of VO_2/MWCNT did not modify hydration kinetics and phases development, water drop angle or compressive strength. Nevertheless, gypsum blinders mass loss increased with the presence of the nanocomposite after 6 humectation-drying cycles. According to the thermal properties, it was found that the NH addition increased gypsum blinders thermal conductivity and Cp values. Finally, it was concluded that gypsum with VO_2/MWCNT promotes self-thermal regulation properties without affecting its performance. The usage of VO_2/MWCNT embedded in a gypsum as a construction material would provide thermal comfort conditions in buildings.

Heat, Comfort, Energy

Resumen

Esta investigación estudió la capacidad de almacenamiento de calor en una pasta de yeso al incorporar un nanocompuesto híbrido (NH) de dióxido de vanadio con nanotubos de carbono multiparedes (VO_2/MWCNT). Asimismo, se determinó la influencia del NH en la cinética de hidratación del yeso y las fases hidratadas. En función de la cantidad de NH adicionada, se realizó un seguimiento a la mojabilidad del yeso, la pérdida de masa por ciclos de humectación-secado, la resistencia a la compresión, conductividad térmica, calor específico (Cp) y desempeño térmico al exponer las muestras a 40 °C. Los resultados mostraron que la incorporación del VO_2/MWCNT en el yeso no modifica la cinética ni el desarrollo de los productos de hidratación, la mojabilidad o la resistencia a la compresión. Además, se encontró que la presencia del NH propicia la pérdida de masa después de 6 ciclos de humectación-secado. Adicionalmente, se determinó que la adición del VO_2/MWCNT incrementa la conductividad térmica y el Cp. Se concluyó que la integración del VO_2/MWCNT en el yeso favorece el desarrollo de propiedades térmicas de autorregulación de calor sin afectar su integridad por lo que, su uso como material de construcción favorecería el confort térmico al interior de una edificación.

Calor, Confort, Energía

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Introduction

The development of mankind has led to an increase in the energy demand required to meet basic needs. By the end of 2021, global gross energy consumption will increase by 5.8 % (bp report, 2022). This is worrying because of the contribution to the carbon footprint caused by this increase. Despite the different actors responsible for global energy consumption, the building sector alone uses 40 % of the total gross energy produced (Amaral *et al.*, 2017). Furthermore, it is estimated that around 60% of energy consumption in a building is used by thermal regulation systems in order to provide thermal comfort inside (Faraj *et al*., 2020). From this problem arises the need to generate materials or methods to improve the thermal behaviour of buildings that promote a more efficient performance of thermal regulation systems (Faraj *et al*., 2020).

In this regard, the incorporation of phase change materials (PCMs) in conventional building materials (Alzoubi *et al*., 2020) such as gypsum has been studied because it is a commonly used material in construction due to its low energy cost of production (Lushnikova & Dvorkin, 2016) and its fire resistance (Castellón *et al*., 2021) and acoustic insulation (Boccarusso *et al*., 2020) properties.

PCMs are materials with the ability to store heat latently through a change of state (solid, liquid or gas), this modification occurs at a transition temperature (Tc) with a specific enthalpy called latent heat (Frazzica *et al*., 2019). The use of PCMs is intended to provide traditional building materials with the ability to store a fraction of the heat received in the open to decrease the amount of heat transferred to the interior of buildings (Jeong *et al*., 2019).

Despite the variety of PCMs available, studies on building materials have focused on the use of PCMs with a phase change from solid to liquid state because most of these have a Tc between 20 and 40 °C and a latent heat of 60 to 230 J/g, properties established as ideal for the application of these types of systems (Tyagi & Buddhi, 2007).

However, they show low compatibility with cementitious materials due to the low cohesion between their particles, with leakage of the PCM during the change of state, as well as a decrease in mechanical strength. Guardia *et al*. report a 50 % decrease in the compressive strength of white cement pastes incorporated with commercial paraffin at 20 % of the total weight of the mixtures (Guardia *et al*., 2019). Sic., Cunha *et al*. present a study on the decrease of up to 75% by adding commercial paraffin between 5 and 20% of the total weight of ordinary Portland cement pastes (Cunha *et al*., 2020). On the other hand, there are solid-solid PCMs with melting points between 30 and 68°C and latent heat between 10 and 150 J/g (Fallahi *et al*., 2017). Despite having a higher Tc with lower latent heat than liquid-liquid PCMs, solid-solid PCMs could offer better mechanical stability when mixed with cementitious materials, since they maintain their solid state during their transition, which would prevent PCM leakage and allow better cohesion between PCM particles and the cementitious material (Raj *et al*., 2020).

PCMs in general prove to be an excellent alternative for the development of innovative construction materials, although, most PCMs exhibit low thermal conductivity (λ) (Cheng *et al*., 2020). This characteristic is reflected in the heat storage-release dynamics, since, when interacting with ambient heat the PCM has greater difficulty in absorbing and/or releasing it (Zuo *et al*., 2020). This characteristic has been reduced by coupling materials with high thermal conductivity such as carbon-based materials into PCM (Coppola *et al*., 2016). In doing so, improvements in thermal conductivity of up to 200% have been obtained (Guardia *et al*., 2019; Kim *et al*., 2021). Yu *et al*. used a commercial vegetable oil-based PCM with a thermal conductivity of 0.199 W/mK, to which carbon nanotubes were incorporated at 5% of the total weight of the mixture, obtaining a thermal conductivity of 0.536 W/mK (Yu *et al*., 2014).

Based on the above, in this research, a NH of VO2/MWCTN was prepared as a solid-solid PCM and its incorporation in a gypsum paste was studied at 0.5 %, 1 % and 2 % of the weight of the cementitious agent with the aim of developing a material with the capacity to store/release heat through a rapid response to external temperature changes, depending on the percentage of NH addition.

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The influence of NH on hydration kinetics and hydrated products, water resistance and physical-mechanical performance, which are indispensable properties in materials used in construction, was also determined.

Experimental Methodology

Starting materials

This study considers the following materials:

1. MAXIMO brand plaster
2. Vanadium Dioxide (VO2) with 99 % purity Alfa AesarTM brand.
3. Multiwalled carbon nanotubes (MWCNT) functionalised with OH radicals of 20-30 nm in diameter and 10 to 30 micrometres in length with 95 % purity brand US Research Nanomaterials Inc.
4. Isopropyl alcohol with 99 % purity CTR Scientific brand.
5. CTR Scientific brand ethylene glycol RA (C2H6O2) dispersant additive (AD) with 99.91 % purity.
6. Potable wáter

NH VO2/MWCNT Synthesis

The preparation of NH was carried out by wet impregnation of MWCNTs and VO2 particles in a ratio of 5/95 wt%.

For this process, the materials (VO2 and MWCNT) were placed in the above mentioned ratio in a beaker and 100 mL of isopropyl alcohol was added for each gram of powder. Afterwards, ultrasound was applied to the solution for 1 hour. Subsequently, NH was obtained by evaporating the isopropanol at 90 °C with constant magnetic stirring until total evaporation of the alcohol. Finally, the powder obtained was heated at 150 °C for 3 hours in order to remove any organic residue from the alcohol.

Preparation of the gypsum pastes

Three mixtures were prepared with NH VO2/MWCNT incorporation of 0.5 %, 1 % and 2 % by weight of the gypsum and a base test tube without addition. Table 1 shows the weight per cubic metre of material for each case.

Mix	Plaster (kg/m^3)	Water (kg/m^3)	NH (kg/m^3)	AD (kg/m^3)
Y0	943.58	589.74	0	0
Y05	943.58	579.92	4.71	9.81
Y1	943.58	579.92	9.43	9.81
Y2	943.58	579.92	18.87	9.81

Table 1 Dosage of the pastes under study

The elaboration of the pastes started withthe dispersion of NH in the mixing water in order to mitigate the agglomeration of the nanocomposite in the matrix (Silvestro & Jean Paul Gleize, 2020). For this, the corresponding amount of VO2/MWCNT and mixing water, the latter consisting of 1 %v AD and 99 %v drinking water, was placed in a vessel. The solution was then subjected to ultrasound for 1 hour. At the end of this process, the resulting solution was mixed with the gypsum following the procedure indicated in ASTM C 305.

The pastes were cast in cylindrical moulds of 2.5 cm diameter x 5 cm height and in cubic moulds of 5 cm per side. Finally, all specimens were demoulded after 24 hours and used once they reached 7 days of age.

Microstructural characterisation

The quantification of the compounds present in the starting materials was carried out by X-ray fluorescence (XRF) on a PANalytical Epsilon3-XL spectrometer using 40 mm diameter and 4 mm thick pellets. The crystalline phases present were determined by X-ray diffraction (XRD) on a PANalytical Empyrean diffractometer in a 2θ range from 10° to 60° with an increment of 0.05° and a scanning speed of 5°/min using copper Kα radiation; the identification of the compounds was carried out using the International Centre for Diffraction Data (ICDD) database. In addition, the weight percentage of the identified phases was quantified using the X'Pert HighScore Plus software.

The morphology of the samples and the NH distribution in the gypsum matrix was observed by scanning electron microscopy (SEM) on a JEOL JSM-6510LV microscope, using a gold-palladium coating on the samples. The wettability of the samples was determined by the water contact angle technique on a Krüss model DSA25E using cubic specimens dried to constant weight at a temperature of 45 °C ± 5°.

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Compressive strength

The compressive strength test was performed in accordance with ASTM C 472. Three cylindrical specimens of each percentage of NH addition were used 7 days after preparation. They were then subjected to uniaxial compressive loading until fracture in an Instron model 600DX hydraulic press. A loading rate of 40 psi/s was applied. The results obtained and standard deviation are presented as an average for each sample.

Water resistance

The moisture-drying method was used, which was adapted to the type of material under analysis based on UNE 22190-2 1990 and ASTM D 4843. Three cylindrical samples of each mixture were used and dried to constant weight at a temperature of 45 °C ± 5°. This method began with the total immersion of each specimen in 100 ml of distilled water for 6 hours in a cylindrical glass flask measuring 5 cm in diameter by 12 cm in height, making sure that the sample did not touch the bottom. They were then removed from the water and dried in an oven at 45 °C ± 5° for 12 hours.

This process was carried out in a total of 6 cycles in which the dry and water saturated weights of the specimens were recorded. The total mass loss and water absorption per cycle were calculated with equation 1 and 2, respectively.

$$\%P_m = \frac{P_{i\,s} - P_{6\,s}}{P_{i\,s}} * 100 \quad (1)$$

Where:

$\%P_m$= total mass loss (%), $P_{i\,s}$= initial dry weight of the sample (g) and $P_{6\,s}$= weight of the dry specimen after the 6th cycle (g).

$$\%ABS_x = \frac{P_{saturadoxs} - P_{i\,s\,x}}{P_{i\,s\,x}} * 100 \quad (2)$$

Where:
$\%ABS_x$= water absorption (%) for a given cycle, $P_{saturado\ xs}$= weight of the water-saturated and surface-dried specimen from the post-calculation cycle (g) and $P_{i\,s\,x}$= weight of the dry sample of the cycle under calculation.

The results were then averaged and their standard deviation calculated.

At the end of the 6th cycle, the compressive strength of the specimens was determined in order to evaluate the effect of mass loss on this property.

Thermal properties

The thermal storage capacity and transition temperature of NH was measured by differential scanning calorimetry (DSC) with a TA Instruments model SDTQ600 calorimeter in the temperature range of 30 °C to 100 °C with a heating rate of 2 °C/min in nitrogen atmosphere. The thermal conductivity of the gypsum specimens was measured with a METER model TEMPOS conductivity meter using the SH-3 sensor. Isothermal calorimetry was used to determine the heat of hydration and specific heat in a Calmetrix calorimeter model I-Cal 4000 HPC. The measurement of specific heat was carried out based on that reported by V.-P. Lehto *et al*. (V.-P. Lehto, 1998).

For this purpose, pellets of 25 mm diameter by 10 mm high, without moisture and of known weight were used. They were heated in the temperature range of 25 °C to 55 °C for 1 h. The temperature of the pellet was checked at the temperature range of 25 °C to 55 °C. The temperature of the tablet was checked using a FLIR infrared thermometer model TG165 with an emissivity of 0.80. Immediately afterwards, the pellet was placed inside the calorimeter to start measuring the heat flux released until a temperature of 23 °C was reached (approx. 1 h). Then, the area under the curve of the heat flow graph (Watts) versus time (s) was calculated. The Cp was determined using the following equation.

$$Cp = \frac{Ac}{(T_i - T_c)P_{ms}} \quad (3)$$

Where:
Cp=specific heat (J/kg°C), Ac=area under the curve of the calorimeter measurement (Joules), T_i= initial sample temperature (°C), T_c= calorimeter temperature (°C) (for this experiment was 23 °C) and P_{ms}= weight of dry sample (Kg).

Heating was carried out at 25 °C, 30 °C, 35 °C, 40 °C, 45 °C, 50 °C and 55 °C with 3 measurements for each temperature for each specimen. The Cp obtained were plotted against temperature and linear regression was applied to generate an average line.

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Performance of specimens as thermal regulators

This procedure was developed based on the literature related to the study of PCMs (Du *et al*., 2020; Kuai *et al*., 2021; Mohseni *et al*., 2020). For the experiment, a wooden box in the shape of a quadrangular prism with dimensions of 38 cm per side by 53 cm high, with thermal insulation on its inner sides, was used (figure 1). A 300 watt electric heater was placed inside the box at a distance of 34 cm from the lower inner side of the box. In addition, a cubic specimen of 5 cm per side of each mixture was made, trying to introduce a thermocouple in the centre of it, in order to monitor the internal temperature of the specimens with the help of a Perfect Prime TC0309 4-channel thermometer. First, the empty box was heated and once the temperature remained constant at 40 °C ± 5°, the specimens, which were at room temperature (approx. 25 °C), were introduced. Afterwards, the heating temperatures were recorded for 2 hours followed by 3 hours of cooling to room temperatura.

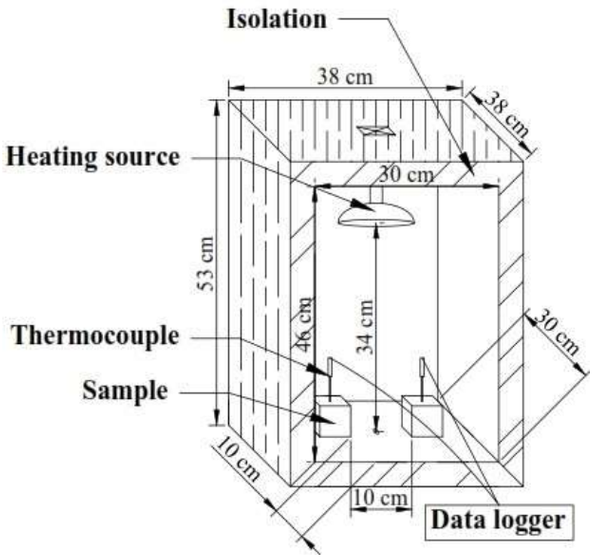


Figure 1 Experimental box arrangement for the evaluation of the thermal performance of pastes

Results

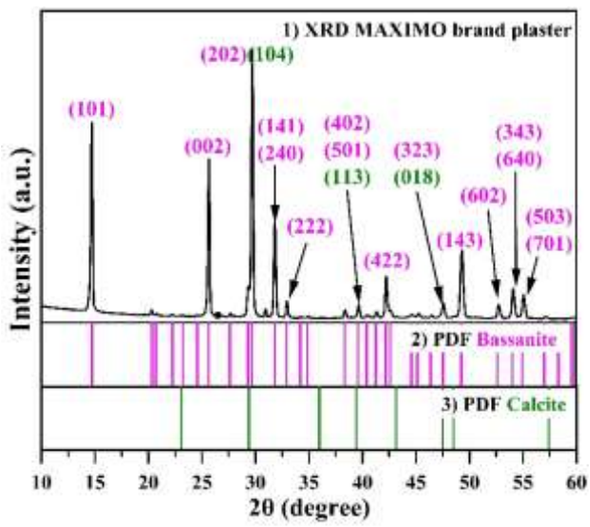
Characterisation of starting materials

The chemical composition of MAXIMO brand gypsum is shown in table 2 below. It details the percentages by weight of the oxides present in this material.

Compound	MAXIMO Gypsum (%p)
SO_3	65.19
CaO	32.34
SiO_2	1.46
Al_2O_3	0.35
K_2O	0.17
MgO	0.16

Table 2 Chemical composition of MAXIMO brand gypsum

Graph 1 illustrates the diffractogram for MAXIMO gypsum, which matched the phase of calcium sulphate hemihydrate or bassanite (CaSO4-½H2O) with Powder Diffraction File (PDF) No.:00-033-0310 and the compound calcium carbonate (CaCO3) or calcite with PDF No.: 01-076-2713. Furthermore, phase quantification indicated 96% bassanite and 4% calcite (table 3). These compounds belong to the expected composition for this type of material (Castellón *et al*., 2021; Rehhoff *et al*., 1990).



Graph 1 Diffractograms of: 1) XRD of MAXIMO gypsum, 2) PDF of bassanite and 3) PDF of calcite

Compound	%p
Bassanita ($CaSO_4$•½H_2O)	96
Calcite ($CaCO_3$)	4

Table 3 Quantification of MAXIMO® brand gypsum phases

On the other hand, the VO2 diffractogram (graph 2) exhibited congruence with the phase of vanadium dioxide with monoclinic crystalline structure (VO2(M)) with PDF No.: 00-009-0142. This ensures the presence of the compound with phase change properties (Li *et al*., 2017).

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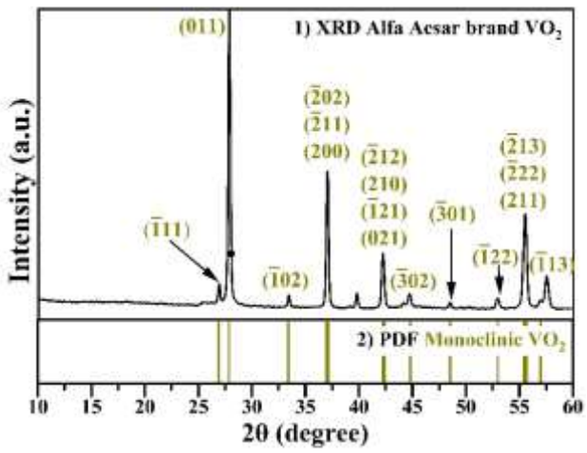
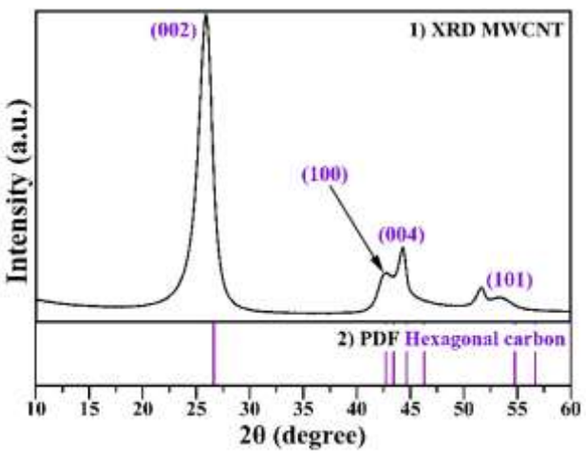


Graph 2 Diffractogram of: 1) XRD of Alpha Aesar brand VO2 and 2) PDF of VO2 with monoclinic crystal structure

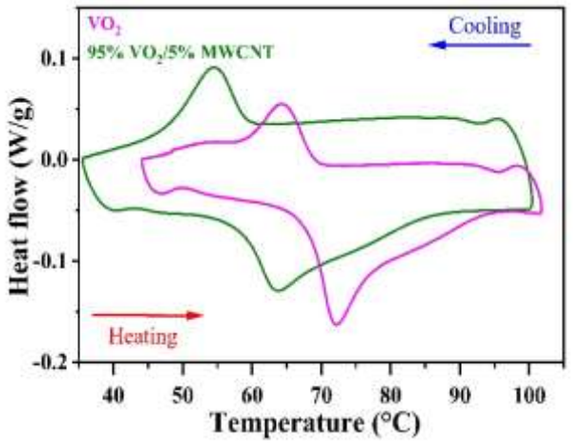
Finally, graph 3 shows the result of the XRD analysis of MWCNTs, where the identified phase corresponds to carbon with hexagonal crystalline structure according to PDF No.:00-001-0640.



Graph 3 Diffractogram of: 1) XRD of MWCNTs and 2) PDF of carbon with hexagonal crystal structure

Characterisation of NH VO2/MWCNT

Once the NH was synthesised, the DSC technique was used on pure VO2 and NH VO2/MWCNT in order to obtain the heat flux graphs shown in graph 4.



Graph 4 Heat flow diagram of pure VO2 and NH 95%VO2/5%MWCNT

The estimates resulted in a heating and cooling Tc of 72.41 ± 0.37 °C and 64.15 ± 0.16 °C for VO2 and 63.73 ± 0.59 °C and 54.53 ± 0.12 °C for VO2/MWCNT. The reduction in Tc is attributed to the presence of MWCNTs in the NH, which increases the thermal conductivity resulting in the improvement of the absorption/release dynamics of the PCM. In addition, a latent heat of heating and cooling of 40.51 ± 0.41 J/g and 22.84 ± 1.14 J/g in VO2 and 32.06 ± 0.53 J/g and 19.35 ± 0.84 J/g for NH were obtained. This reduction in latent heat of NH is caused by the integration of MWCNTs in NH due to the decrease in the amount of VO2 available to store heat.

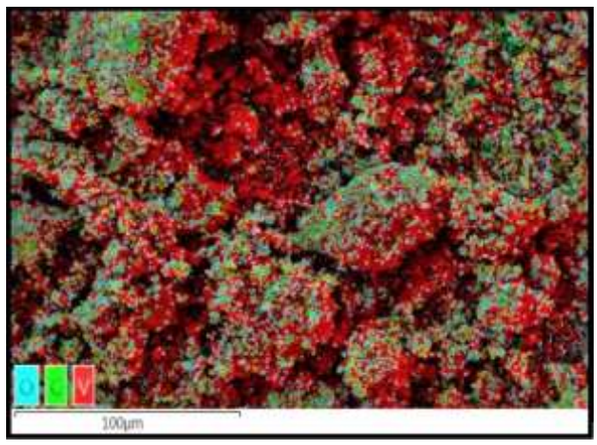


Figure 2 EDS analysis of NH VO2/MWCNT with a focus on the elements oxygen, carbon and vanadium

Sic., figure 2 illustrates the energy dispersive spectroscopy (EDS) analysis of NH. The scattering of MWCNTs on the VO2 surface is observed, which exhibits the saturation of the PCM particles with the thermal conductivity enhancer.

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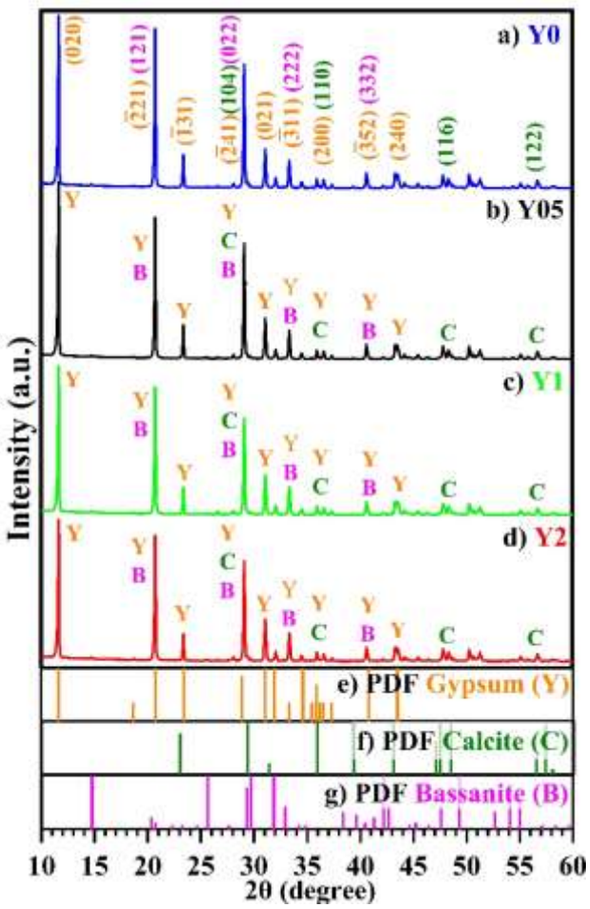
Microstructural characterisation

With the XRD analysis of the hardened pastes, the diffractograms shown in graph 5 were obtained. Firstly, a development of hydration phases is observed with the presence of calcium sulphate dihydrate or gypsum (CaSO4-2H2O) with PDF No.: 00-003-0044, calcite and bassanite (CaSO4-2H2O) with PDF No.: 00-003-0044, calcite and bassanite.



Graph 5 Diffractograms of 7-day-old pastes of: a) Y0, b) Y05, c) Y1, d) Y2, e) gypsum PDF, f) calcite PDF and g) bassanite PDF

In addition to this, phase quantification (table 4) indicates a similar development of the gypsum phase for all samples, however, samples Y1 and Y2 showed 2 % and 4 % of the bassanite phase. Despite this, the diffractograms of the samples show no change in phase development between them.

Compound	Y0 (%p)	Y05 (%p)	Y1 (%p)	Y2 (%p)
Plaster	97	98	98	96
Calcite	3	2	-	-
Bassanita	-	-	2	4

Table 4 Phase quantification of samples Y0, Y05, Y1 and Y2 at 7 days of age

The analysis of the morphology of the matrices (figure 3) showed that the samples consist of particles of similar size in all cases, with the presence of elongated gypsum crystals with lengths of 8-10 microns and widths of 1-5 microns (Vimmrová *et al*., 2020). This is related to the similar phase development illustrated in the XRD analysis of the pastes (graph 5).

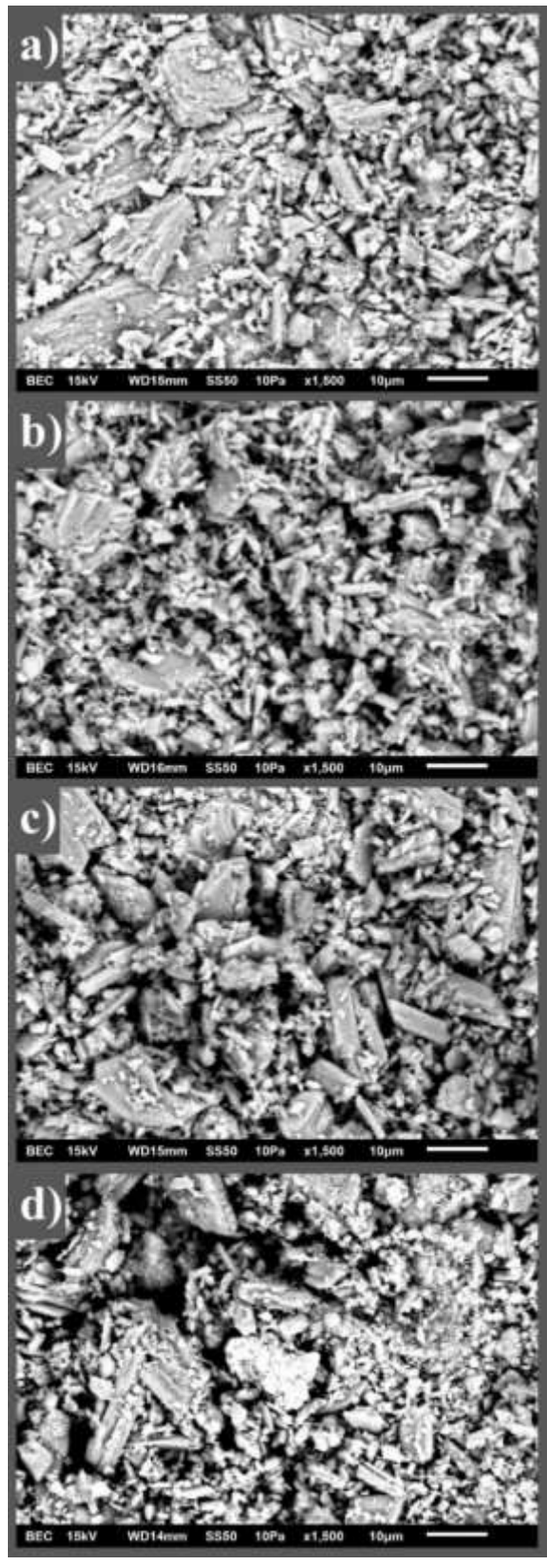


Figure 3 Microscopies at 1500 magnification of: a) Y0, b) Y05, c) Y1 and d) Y2

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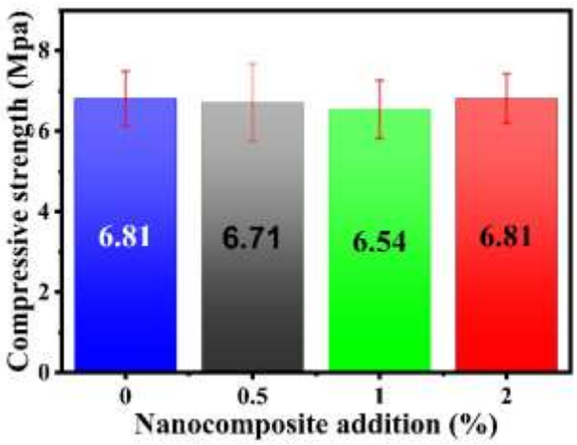
Compressive strength

The compressive strength is presented in graph 6. Here, all the specimens presented a similar strength value of 6.71 Mpa. This indicates that the integration of NH in the gypsum matrix does not generate any modification in the compressive strength development of the pastes with respect to a mix without addition at 7 days of age.

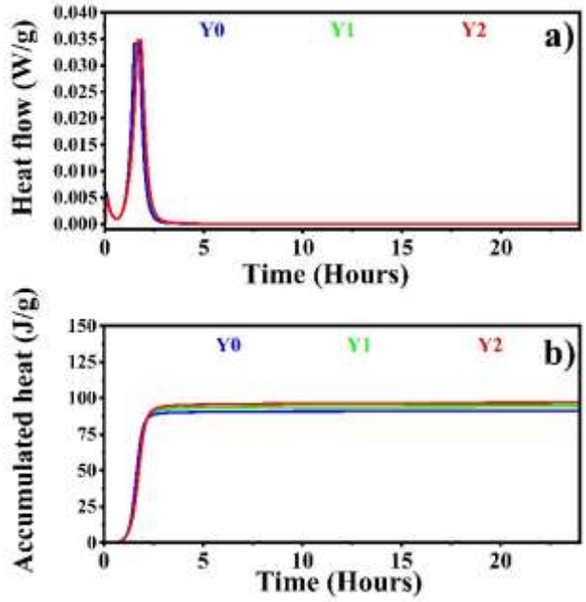


Graph 6 Compressive strength of gypsum pastes

Hydration kinetics

The isothermal calorimetry resulted in the graphs shown in graph 7. The same behaviour of heat release with respect to time due to the exothermic hydration reaction of commercial gypsum is observed for all the samples.

It is interpreted that the presence of NH in the gypsum matrix does not modify the heat flux released during hydration. On the other hand, after 24 hours the total accumulated heat remains similar for all cases. This reveals that NH does not generate alterations in the hydration kinetics of the mixtures or in the development of the hydrated products, which correlates with the diffractograms in graph 5.



Graph 7 Isothermal calorimetry of the pastes. a) graph of the heat flow released and b) graph of the accumulated heat released

Wettability of simples

The water contact angle is an indicator of the surface permeability of materials (Jin *et al.*, 2021). Figure 4 shows the resulting angles measured by this technique. It can be seen that the contact angle value is similar for the four samples presenting a value around 65 ° indicating that the samples are hydrophilic (Gomes *et al.*, 2013) and that the integration of NH in the gypsum matrix did not have a direct effect on the wettability of the material.

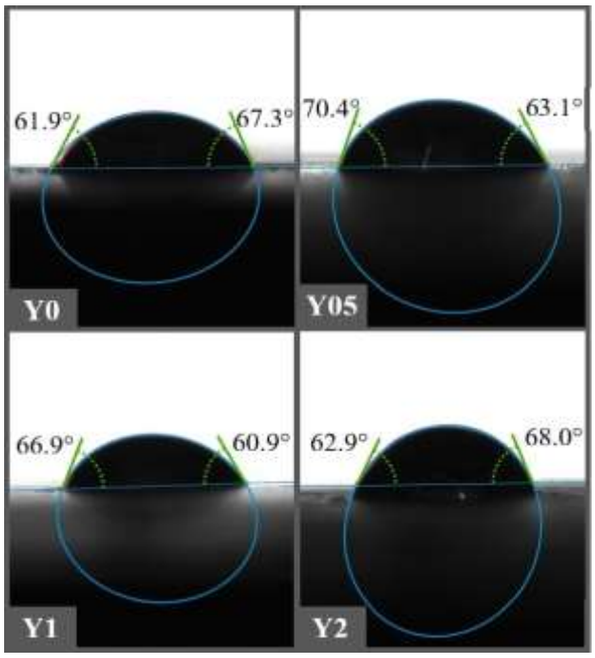


Figure 4 Water contact angle of gypsum pastes

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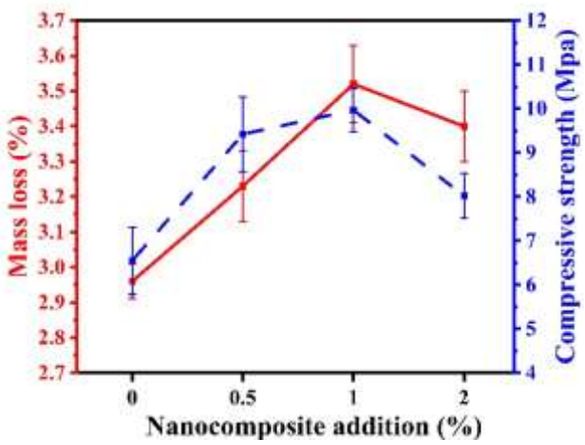
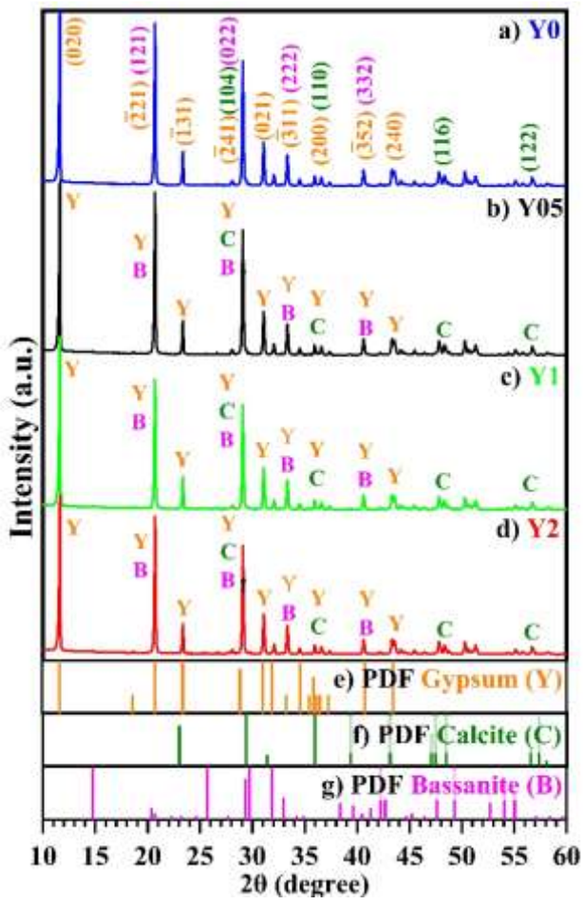
Water resistance

Graph 8 illustrates the percentage mass loss for each specimen after 6 moisture-drying cycles and their compressive strength at the end of the process. Where, the specimens reduced their mass by 2.96 %, 3.23 %, 3.52 % and 3.4 % for Y0, Y05, Y1 and Y2 respectively. Therefore, the incorporation of NH into the gypsum paste increases the dissolution of the material in an aqueous medium, increasing the mass loss by 9.12 %, 18.91 % and 14.86 % when integrating 0.5 %, 1 % and 2 % VO2/MWCNT with respect to the gypsum without NH. In addition, a trend of increasing mass loss is observed between Y0, Y05, Y1, however, Y2 improved this capacity by decreasing the percentage of mass loss by 4.05 % compared to Y1.

Also, graph 8 shows the compressive strength values of the specimens after 6 cycles. Where, specimen Y0 exhibited a strength of 6.55 Mpa which is similar to that obtained at 7 days of age, so its mechanical performance was not affected by the loss of mass. On the other hand, Y05, Y1 and Y2 showed a compressive strength of 9.42 Mpa, 9.97 Mpa and 8.02 Mpa, respectively.



Graph 8 Percentage mass loss and compressive strength after 6 wetting-drying cycles of gypsum pastes



Graph 9 Diffractograms obtained after 6 wetting-drying cycles of: a) Y0, b) Y05, c) Y1, d) Y2, e) gypsum PDF, f) calcite PDF and g) bassanite PDF

Compound	Y0 (%p)	Y05 (%p)	Y1 (%p)	Y2 (%p)
Plaster	96	100	100	97
Calcite	3	-	-	-
Bassanita	1	-	-	3

Table 5 Phase quantification of Y0, Y05, Y1 and Y2 after 6 cycles of the humidity-drying method

Graph 9 shows the diffractograms corresponding to the pastes with and without NH addition after the wetting-drying cycles. It shows similar phases to those exhibited in the samples at 7 days of age, however, the quantification of the phases (table 5) revealed an increase in the amount of gypsum and a decrease in the bassanite phase, which explains the increase in compressive strength of the pastes shown in graph 8.

Graph 10 shows the percentages of water absorption for each sample during the cycles of the moisture-drying method. The reduction in absorption of samples Y05, Y1 and Y2 with respect to sample Y0 is observed. This indicates that NH decreases the absorption of the gypsum pastes.

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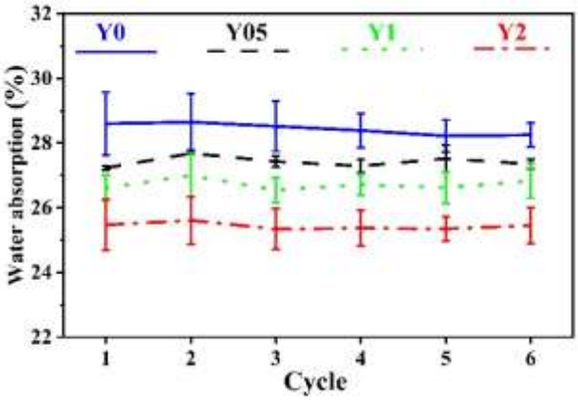
This is attributable to the formation of denser matrices compared to a mixture without addition, since NH occupies voids in the gypsum microstructure.



Gráfica 10 Percentage of water absorption in each wetting-drying cycle of gypsum pastes

Thermal properties

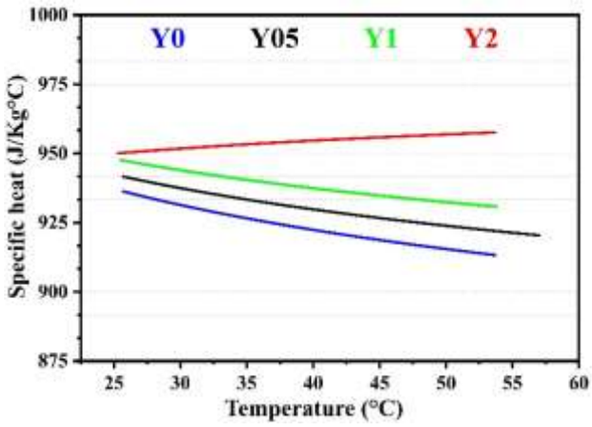
The analysis of the thermal behaviour started with the determination of the thermal conductivity of the specimens. Table 6 reports the values obtained for each sample. The integration of NH in the pastes increases the thermal conductivity value by 0.47 %, 1.07 % and 1.43 % with the addition of 0.5 %, 1 % and 2 % of NH VO2/MWCNT, respectively. The increase of this property is caused by the presence of NH in the cementitious matrix due to 2 reasons: 1. Because of the thermal conductivity of VO2, which is reported to be higher than that of gypsum (Barra *et al*., 2021) and 2.

Sample	Thermal conductivity (W/mK)
Y0	0.4184 ± 0.01
Y05	0.4204 ± 0.009
Y1	0.4229 ± 0.0069
Y2	0.4244 ± 0.01

Table 6 Thermal conductivity of samples

Graph 11 shows the specific heat of the specimens within the range of 25 °C to 55 °C. The interpretation of the diagram shows that at 25.8 °C the Cp of samples Y05, Y1 and Y2 increased by 0.5 %, 1.2 %, and 1.5 %, with respect to the sample without additions. Furthermore, as the temperature increases up to 55°C the Cp of samples Y0, Y05 and Y1 slightly decreased, while Y2 gradually increased its value.

From this it can be stated that at 55°C the Cp of samples Y05, Y1 and Y2 increased their Cp by 0.95 %, 1.91 % and 4.85 % with respect to Y0. This phenomenon is attributed to the heat storage of NH in the pastes. Since, when heat flows through the paste, the PCM particles closest to the heat source are energetically equilibrated before the subsequent ones until all the particles in the system equalise in temperature. Due to this phenomenon, the Cp of NH pastes increases in value as the temperature increases.



Graph 11 Specific heat of pastes with and without addition of NH

Performance of pastes as thermal regulators

The results of the analysis of the thermal behaviour of the specimens when the outside temperature is 40 °C and their cooling to room temperature (25 °C) are shown in graph 12. It compares the temperature increase as a function of time of the specimens with and without NH. It can be observed that, during heating, the increase in the internal temperature of the samples with NH incorporation remained below the Y0 sample, i.e., in the pure gypsum it took 1 h 40 min 17 s to reach 40 °C, whereas, the materials with the addition of 0.5 %, 1 % and 2 % of NH took 1 h 41 min 11 s, 1 h 44 min 24 s and 2 hours 2 min 20 s, respectively. Comparing the temperature of the samples with NH at the same time as the pure gypsum reached 40 °C, the samples with NH showed a temperature differential of 0.1 °C, 1.1 °C and 0.8 °C, respectively.

The higher the amount of NH added, the longer the time for the temperature of the samples to reach 40 °C, indicating that the gypsum with NH is storing heat.

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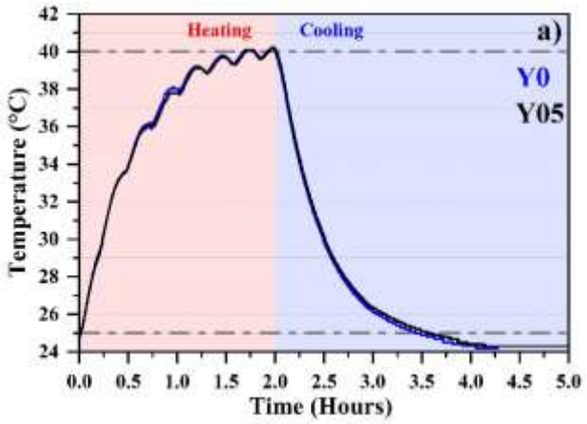
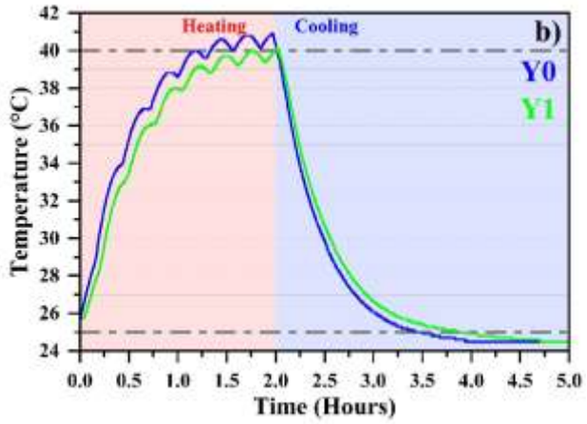
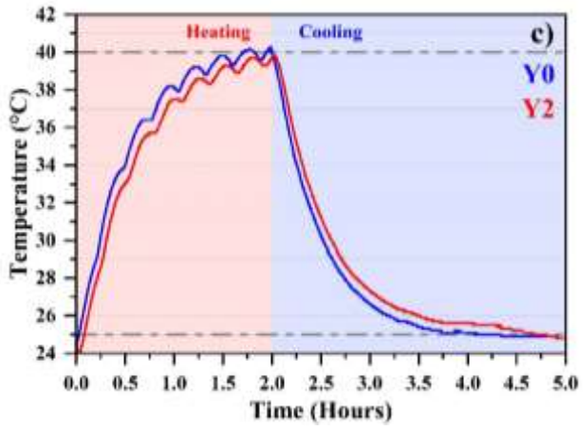
Conversely, during cooling, specimen Y0 decreased its temperature at a faster rate compared to the NH-incorporated samples. Pure gypsum took 1 h 25 min 57 s to reach 25 °C, while the time for Y05, Y1 and Y2 was 1 h 33 min 16 s, 1 h 50 min 33 s and 2 h 40 min 17 s, respectively. When comparing the temperature of gypsum with NH at the same time as pure gypsum reached 25 °C, a temperature differential of 0.2 °C, 0.5 °C and 0.5 °C, respectively, was observed.

This phenomenon occurs due to the increase in the Cp of the samples with NH, as observed in graph 11. This can be attributed to the fact that the higher the amount of NH, the higher the amount of heat stored. The variations in the heating and cooling times of pure gypsum indicate that the gypsum with NH acts as a thermal regulator in both increasing and decreasing the external temperature, which would contribute to maintaining thermal comfort and consequently to the energy efficiency of a building.







Graph 12 Thermal behaviour of gypsum pastes at 40 °C. a) Y0 vs Y05, b) Y0 vs Y1, and c) Y0 vs Y2

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Conclusions

This paper presented the preparation and incorporation of a NH of VO2/MWCNT in gypsum pastes, the corresponding additions were 0.5 %, 1 % and 2 % in relation to the weight of the cementitious agent. The results obtained showed that there is a good compatibility and homogeneous integration between gypsum and NH as the development of the hydrated compounds and the values of compressive strength, wettability and water resistance were not affected. It was found that the addition of NH had a significant effect on the thermal properties of the gypsum with an increase in thermal conductivity by 1.43 % compared to pure gypsum. The Cp value also showed an increase in its estimates.

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This influenced the heating/cooling rate of the samples with NH giving the gypsum the ability to store heat during external temperature rises and release it when the external temperature drops by acting as a thermal regulator, which makes it attractive for the preparation of energy efficient building materials.

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Proposal for a fiber cement panel with the addition of sugarcane bagasse

Propuesta de panel de fibrocemento con adición de bagazo de caña

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Abstract

The objective of the study is to propose a fiber cement panel based on sugarcane bagasse that complies with the basic requirements for non-compressed fiber cement, without using special equipment for its manufacture, reducing the processes by half without compromising the quality of the product. No crusher, kiln, hydraulic press or vacuum pump were used to produce the panels; water was used to clean the fibers and sodium silicate was used as a mineralizing agent; results were compared with NMX standards for structural mortars and fiber cement slabs together with manufacturing processes from previous research on natural fibers to obtain a methodology that optimizes the process. It is demonstrated that it is feasible to produce fiber cement panels that comply with the minimum regulatory requirements by reducing processes, without damaging the physical-mechanical values of the fiber cement. The best performance results show a MOR of 6.70 MPa and a density of 1560 kg/m^3 for a panel with 6% cane bagasse added to the dry mass of cement, a water/cement ratio of 0.95 by volume and sand/cement of 2.5 by mass. Opportunities are generated to experiment with counterpart fibers based on the tools, processes and recommendations of the project.

Composite material, Fiber cement, Cane bagasse fiber, Sodium silicate

Resumen

El objetivo del estudio es proponer un panel de fibrocemento a base de bagazo de caña que cumpla con los requisitos básicos para fibrocementos no comprimidos, sin utilizar equipos especiales para su manufactura, reduciendo los procesos a la mitad sin comprometer la calidad del producto. Para elaborar los paneles no hay necesidad de emplear: trituradora, horno, prensa hidráulica o bomba de vacío; para la limpieza de las fibras se utilizó agua y como agente mineralizante silicato de sodio; se contrastaron resultados con normas NMX para morteros estructurales y placas de fibrocemento junto con procesos de manufactura de investigaciones previas sobre fibras naturales para obtener una metodología que optimice el proceso. Se demuestra que es viable producir paneles de fibrocemento que cumplan con los requisitos mínimos normativos reduciendo procesos, sin perjudicar los valores físico-mecánicos de éste. Los resultados de mejor desempeño arrojan un MOR de 6.70 MPa y una densidad de 1560 kg/m^3 para un panel con 6% de adición de bagazo de caña respecto a la masa seca del cemento, una relación agua/cemento de 0.95 en volumen y arena/cemento de 2.5 en masa. Se genera oportunidades para experimentar con fibras homólogas basándose en las herramientas, procesos y recomendaciones del proyecto.

Material compuesto, Fibrocemento, Fibra de bagazo de caña, Silicato de sodio

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Introduction

In recent years, environmental concerns have stimulated extensive research on environmentally friendly materials. Putting attention to the use of fibers obtained from renewable plant sources for composite materials (Biagiotti, *et al*., 2008; John & Thomas, 2008; Faruk, *et al.*, 2012). The combination of interesting mechanical and physical properties, in addition to environmental benefits, has been the main driver behind their use as an alternative to traditional reinforcements (Ardanuy, *et al*., 2015).

Whereby although brittle building materials have long been reinforced with plant fibers (PV) since ancient times, the concept in cement-based reinforcement materials was first studied in the 1940s, when these were evaluated as possible substitutes for asbestos fibers due to their toxicity (Tonali, *et al*., 2011).

Therefore, the applications of cement-based composites with PV have been defined in the construction area as non-structural elements, such as: thin walls, thin sheet products for partition walls, building envelopes or roofs, flat sheets, roof tiles and prefabricated components in general (Roman, *et al.*, 2008).

Despite the above, bagasse fibers (BC) have less scientific documentation with respect to fiber cement panels, so most of them follow a series of base or standard processes in their production, unlike other PVs such as wood chips, wool or cellulose, conifers and other lignocellulosic materials such as rice husks, cotton stalks, hemp, coconut fiber, jute, sisal, etc. Therefore, it is important to create more alternative uses and methods (Fernández Rodríguez & Díaz Hernández, 2017).

Being able to use a new manufacturing methodology in coating dividing elements for this type of fiber, means replicating in a plausible way panels with half of the recommended processes but manually, with few elements, especially in communities that have this type of agro-industrial waste; therefore the method would be useful in future homologous projects for other PVs with similar characteristics in order to contrast the feasibility of the method and its limits.

Research on the treatment of BC share similarities from its cutting, separation, cleaning, drying and mineralization, all of them with different mechanical, physical or chemical means in the process, for example to define the morphology of the fiber commonly a cutting, chopping or grinding is done in addition to the one that the bagasse already has at the time of leaving the sugar mills (Lozano Zamora & Rojas Fraile, 2019), then the fiber is sieved and sifted by machinery with a certain dimension; For washing and elimination of the remaining sugars, it is subjected to cycles of rinsing with hot and cold water (Cabral, *et al*. , 2018) or otherwise with substances such as calcium hydroxide for cleaning, which also serves as a coating against aggressive agents (Osorio Saraz, *et al*., 2007; Nawrath Barros, 2015); as for chemical stabilization, so that the fiber is not affected by the alkalinity of the cement, sodium silicate is commonly used (López Barrios & Valencia Gualdron, 2006), the aforementioned calcium hydroxide or even without any agent to protect it; finally, for the drying of the fiber, forced draft ovens are used, in order to have a constant temperature control.

For the manufacture of panels, the process generally consists of the mixing of materials, addition of accelerators, casting, vibrating, pressing and curing. The making of the cementitious paste is usually carried out with motorized mixers since the fibers make it difficult to elaborate a homogeneous mortar, some use accelerants such as calcium chloride in this step since the cellulose present in the fibers delays the setting, (Osorio Saraz, *et al*., 2007) after that, fixed or disassembled molds are used which can be metallic, acrylic or wood; then a pressing with machinery or dead weight is applied for a short period of time. There are certain proposals that combine this with vacuuming to eliminate excess moisture from the paste (Khorami & Ganjian, 2011); finally, the piece is cured by immersing it in water or using curing chambers with high humidity for a period of one month.

The techniques and considerations that were taken based on the preliminary results and the information provided by the different investigations with cementitious materials and natural fibers will be handled within the development.

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Materials and methods

A series of tests were carried out to design, experiment and characterize the materials to be used, from the tools, mix composition, element thickness, casting, vibrating and curing, in order to obtain an adequate result that complies both in physical and mechanical characteristics according to the non-compressed fibrocement standard NMX-C-234-ONNCCE-2015.

A CPC 30R RS cement was used for the design of a rapid strength mortar. Along with river sand from the material bank "El Cuervo" in Huajúmbaro, Michoacán; the stone that passed the ASTM No. 4 mesh (4.75 mm opening) was used, this same contains an amount of 78% (Table 1) of silica based on x-ray fluorescence analysis (Arreola Sánchez, 2013).

Huajúmbaro Arena "El Cuervo"			
Component	%	Component	%
SiO2	78.185	CaO	1.015
AlO3	11.557	Na2O	2.666
TiO2	0.203	K2O	3.577
Fe2O3	1.567	PXC/PPI	1.19
MgO	0.239	BaO	0
MnO	0.03	P2O5	0.036
Sum = 100.29			

Table 1 Chemical composition of the river sand in Huajúmbaro *(Arreola Sánchez, 2013)*

Dry sugarcane bagasse with a relative humidity of 11.7% from the Lázaro Cárdenas sugar mill in Taretan, Michoacán was used; it was processed at the mill to be used as boiler fuel; a series of rollers and machinery squeezed it and crushed it five times until leaving a fiber with little sugar and with lengths of 1.0 to 3.0 cm and thicknesses of 1 to 15 mm, in addition to containing a high amount of fines. In order not to affect the setting and the decrease in strength of the mortar, the fiber was sieved with an ASTM No. 4 mesh in order to eliminate randomly dispersed pellets and fragments in the bagasse; the fiber used was the one retained by the ASTM No. 8 mesh in order to eliminate the high amount of fines.

The fiber was washed with water at room temperature, being submerged for 24 h and then rinsed, squeezed manually and left to dry, then to be used in the mixture, it was left to saturate again one day before being made for the same period of time (Figure 1).

Figure 1 Sifting and cleaning of sugarcane bagasse.

So that the BC does not degrade in the alkaline medium at the time of integration into the cement matrix, it is previously impregnated with sodium silicate 20º Be with a concentration of 22% w/w, a higher range was used than other research whose values ranged between 15-17% w/w (Juarez Alvarado, 2002) due to the fact that a fiber rinse with boiling water was not used.

For the binder, a type I mortar was designed based on the (NMX-C-486-ONNCCE-2014) with a cement/sand dosage of 1:2.5 in mass with respect to previous tests showing a better resistance with the sand used in this project (Mondragón Martínez, 2021); so that the fluidity was acceptable in the control without going out of the norm, a water/cement ratio in volume of 0. 95, which allowed a value of 130% in the flow table; this was used in all the test groups to maintain a good consistency when working the mix with mineralized BC.

Next, a panel morphology was used with 25.0 cm x 25.0 cm slabs in accordance with the standards for fiber cement slabs, with a thickness of 1.0 cm; BC fiber was then added in percentages of 3.0, 6.0 and 12.0% with respect to the dry mass of the cement. Before mixing with the prepared mortar, the fiber is treated as follows: once the fiber is saturated with water, the excess is emptied without squeezing for 5 minutes, then it is submerged and impregnated with sodium silicate for 5 minutes while the fiber is shaken, immediately the excess liquid is drained for 3 minutes, in order to eliminate excesses, after which it is added to the previously mixed mortar (Figure 2).

The previous water saturation helps that both the fibers and the sodium silicate do not detract hydration from the mortar and cause a drop in its quality, in addition to facilitating its handling and curing.

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Figure 2 BC saturated in water and subsequently impregnated with sodium silicate

The mixing of the components that make up the composite was carried out with a mixing shovel using a 19 L bucket as a container, as shown in Figure 3, following the following sequence:

1. Cement + Sand - mixed 30 seconds manually with a spoon until the composition was homogeneous.
2. Addition of Water - 30 seconds at medium speed with the mixer with circular movements.
3. Addition of treated BC fiber - 30 seconds at medium speed with the same movement but with an up and down motion.
4. 20 seconds at fast speed with the same motion.
5. Check the consistency, making sure that there are no fiber clumps, if there are, repeat step

Figure 3 Mixing of components

The specimens were poured into previously greased disassembled metal molds, which after being filled with the mixture were compacted by manual impact vibration, dropping it to a height of approximately 10 cm, 15 times per side (60 blows in total), then the excess mortar was leveled with the edge of a ruler that was moved in the longitudinal and transversal direction slowly through the mold with a continuous and fast transversal movement to avoid pulling out pieces of mixture and fiber, then it was tamped with an acrylic prism in parallel on the four sides of the mold and proceeded again to vibrate them.

Finally the ruler was passed again with the same procedure to eliminate surplus. They were demolded after 24 h and were superficially moistened with a sprinkler to be later left inside a sealed container in order to maintain favorable humidity and temperature conditions for their curing. The tests were carried out at 7 and 28 days of curing (Figure 4).

Figure 4 Panel manufacturing process

Subsequently, once the specimens are cured, tests are made to obtain the modulus of rupture (MOR) to five plates in a Forney brand universal machine, with a constant acceleration of 0.30 t/min supported on two smooth rods with a diameter of 20 mm and a separation between them of 200 mm for each of the four test groups in transverse and longitudinal direction (Figure 6), averaging the values and performing the test in a time of approximately 30 seconds, noting the maximum load when the rupture is recorded, all based on what is marked by the standard and using equation 1.

$$MOR = \frac{eFl_s}{2be^2} \quad (1)$$

Where: F is the ultimate load (N); l_s is the distance between the centerlines of the supports (mm); b is the width of the test specimen (mm) and e is the thickness where the failure passes (mm).

They are subjected to vertical bending tests as marked in the standard with the following arrangements and instructions as shown in Figures 5, 6 and 7.

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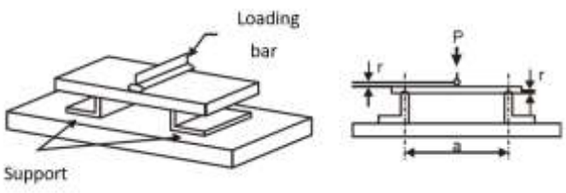

Figure 5 Bending test apparatus (NMX-C-234-ONNCCE-2015)

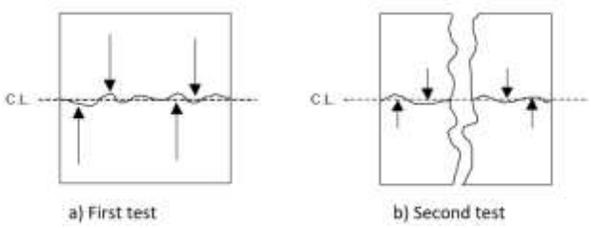

Figure 6 Transversal and longitudinal arrangement for testing by plate (NMX-C-234-ONNCCE-2015)

Where: r are the upper faces of the round supports with a radius of 20 mm; a is the distance between supports for this case will be 200 mm.

Figure 7 Attachment and arrangement for bending test (NMX-C-234-ONNCCE-2015)

Finally, permeability tests were performed which consisted of placing an acrylic frame of 20.5 cm x 25.5 cm x 5.0 cm on top of a panel, this was repeated for each test group taking only one panel from each batch which were cut to a measure of 25.0 cm x 20. 0 cm according to the standards (Figure 8) and this was sealed in the part that made contact with the plate, then water was placed until a height of 20 mm was obtained on the upper face of the panel, it was left for 24 h in ambient conditions without moving the specimen to finally report, at the end of this time, if visually drops were present on the lower face (Figure 9). In this test the panels may show traces of moisture on the bottom of the plate, but in no case should water droplets be observed.

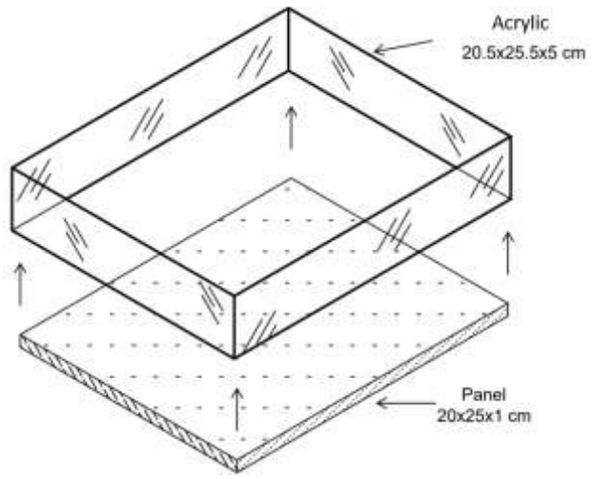

Figure 8 Permeability test frame assembly and dimensions

Figure 9 Performance of permeability test on control specimen (control)

Results and discussion

After testing the specimens with the different dosages of BC and control specimens (Table 2), it is observed that the test group with 6% addition of BC fibers is the one that shows the highest modulus of rupture at 28 days; with respect to the values at 7 days, the specimen containing 3% BC is shown as the best specimen, but its resistance shows a decrease of 32.

The remaining specimens with the addition of BC show an improvement in flexural strength with respect to the control, none of them worsens or decreases below the value of the same once the month is reached, which indicates that the fibers fulfill their function as reinforcement without reducing the panel mechanically (Table 3) (Graph 1).

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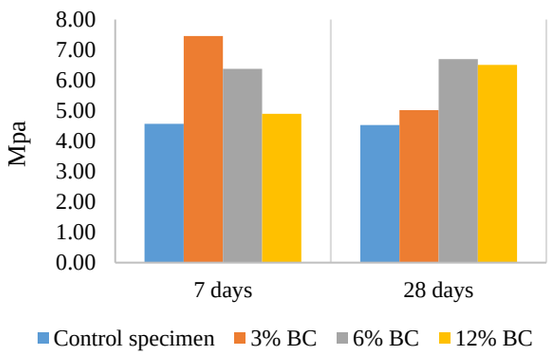
Dosage to manufacture a panel				
Components	Witness	3% BC	6% BC	12% BC
Cement (gr)	357	350	340	320
Sand (gr)	892	875	850	800
Water (ml)	339	332	323	304
Fiber (gr)	0	10.50	20.40	38.40

Table 2 Dosage of each test group to produce a 25x25x1 cm panel

MOR results for the test groups		
Mix	MOR prom. 7 days (MPa)	Average MOR at 28 days (MPa)
Witness	4.56	4.53
3% BC	7.46	5.02
6% BC	6.37	6.70
12% BC	4.90	6.51

Table 3 Average modulus of rupture of the different specimens and cores at 7 and 28 days.

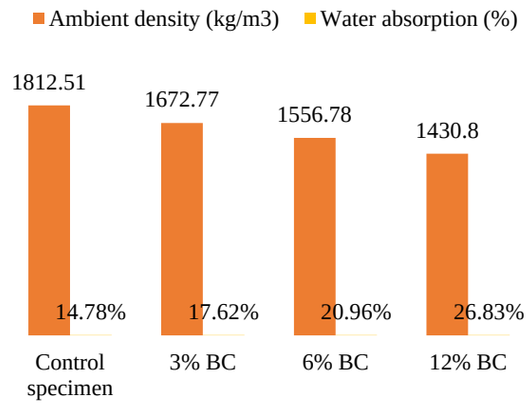


Graph 1 Average modulus of rupture of the different specimens and samples at 7 and 28 days

As for the ambient density, the values are shown in Table 4, in which all of them show a decrease in density as the amount of fiber addition increases, as well as the water absorption capacity (Graph 2).

MOR results for the test groups		
Mix	Ambient density (kg/m^3)	Water absorption (%)
Witness	1812.51	14.78
3% BC	1672.77	17.62
6% BC	1556.78	20.96
12% BC	1430.80	26.83

Table 4 Density and absorption capacity of the test groups



Graph 2 Physical properties of the panels

The permeability test showed that none of the specimens with the addition of BC passed the 24 h test, since they showed water droplet formation before the end of the test (Figure 10). The higher the concentration of BC, the shorter the time in which drops appear, with times of appearance ranging from 20 h to only 7 h (Table 5).

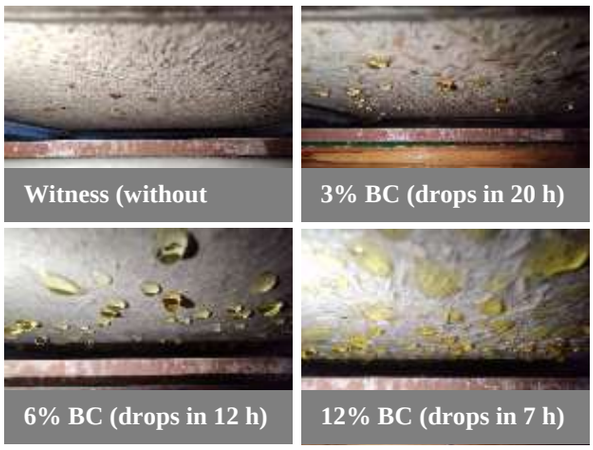


Figure 10 Photographic report of the visual inspection of the four analysis groups in the permeability test

24 h permeability test report	
Mix	Droplet formation
Witness	None
3% BC	20 h
6% BC	12 h
12% BC	7 h

Table 5 Comparative table of the test groups to permeability for 24 h

According to the above results MOR was classified as a type B fiber cement board, which is used for boards in interior applications, such as interior walls, floors, ceramic substrate or walls, which can be subjected to heat, moisture but not freezing as shown in (Table 6).

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Applications and categories of NT flat plates for category B
Application
1. Substrate for internal walls or floor tiles.
2. Ceilings.
3. Interior substrate for walls to be painted or wallpapered.
4. Mezzanine or base floors (internal).

Table 6 Possible uses for non-compressed fiber cement boards (NMX-C-234-ONNCCE-2015)

According to the minimum MPa values established by the standard, the fiber cement manufactured with this proposal is classified in Class 1; the standard does not specify its use, it only classifies it in that category (Table 7)

Minimum performance requirements	
Category B in ambient condition (MPa)	
Class 1	4
Class 2	7
Class 3	13

Table 7 Minimum MPa values (NMX-C-234-ONNCCE-2015)

There are more applicable tests according to NMX-C-234, but they are not of high relevance they are considered of lower hierarchy and as the category is type B it is not necessary to perform the other tests.

Conclusions

The fiber cement panel composed of BC and mortar, denotes adequate MOR values for its use, being supported by the marked in the NMX-C-234-ONNCCE-2015, with results above 4 MPa, which is designated as Class 1, getting very close to a class 2 panel of 7 MPa, having the possibility to perform as a standard panel category B with uses such as: substrate for internal walls or floor tiles, ceilings, interior substrate for walls to be painted or wallpapered and mezzanine or floor base (internal), but nevertheless by not successfully passing the permeability test, considered as a higher grade, which is below the flexural test, the objective is semi-complete, exposing a new problem: how to increase the impermeability, in order to guarantee its durability, which is implicit in the failed test of the test groups with BC. It is remarked that even so, the mechanical values reflected by the method used, which leaves out several of the usual processes carried out for woody fibers, did not impair the final flexural performance of the panels.

The general objective of the feasibility of being able to produce a fiber cement panel based on sugarcane bagasse and mortar using a smaller amount of resources and procedures for its production is partially confirmed, for which it is now necessary to modify the process, in such a way that it allows improving the impermeability, in order to complete the control requirements that guarantee the permanence of the element.

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Objectives
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Objectives
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Citation: Surname (IN UPPERCASE), Name 1st Author, Surname (IN UPPERCASE), Name 1st Co-author, Surname (IN UPPERCASE), Name 2nd Co-author and Surname (IN UPPERCASE), Name 3rd Co-author. Paper Title. Journal Civil Engineering. Year 1-1: 1-11 [Times New Roman No.10].

* Correspondence to Author (example@example.org)
† Researcher contributing as first author.

Introduction

Text in Times New Roman No.12, single space.

General explanation of the subject and explain why it is important.

What is your added value with respect to other techniques?

Clearly focus each of its features

Clearly explain the problem to be solved and the central hypothesis.

Explanation of sections Article.

Development of headings and subheadings of the article with subsequent numbers

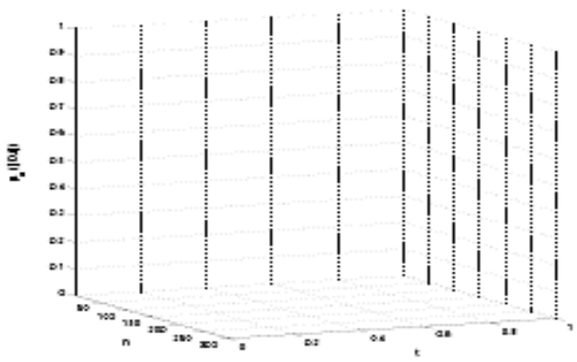
[Title No.12 in Times New Roman, single spaced and bold]

Products in development No.12 Times New Roman, single spaced.

Including graphs, figures and tables-Editable

In the article content any graphic, table and figure should be editable formats that can change size, type and number of letter, for the purposes of edition, these must be high quality, not pixelated and should be noticeable even reducing image scale.

[Indicating the title at the bottom with No.10 and Times New Roman Bold]



Graphic 1 Title and *Source (in italics)*

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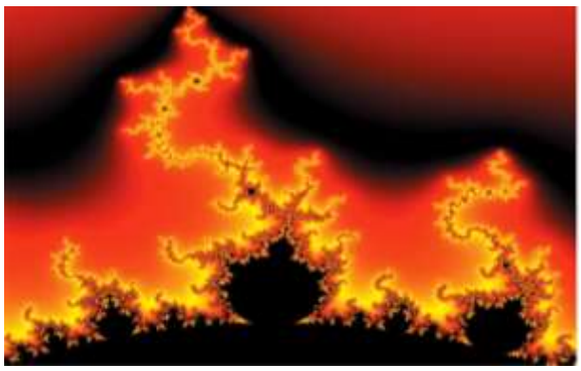

Figure 1 Title and *Source (in italics)*

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Table 1 Title and *Source (in italics)*

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Each article shall present separately in **3 folders**: a) Figures, b) Charts and c) Tables in .JPG format, indicating the number and sequential Bold Title.

For the use of equations, noted as follows:

$$Y_{ij} = \alpha + \sum_{h=1}^{r} \beta_h X_{hij} + u_j + e_{ij} \quad (1)$$

Must be editable and number aligned on the right side.

Methodology

Develop give the meaning of the variables in linear writing and important is the comparison of the used criteria.

Results

The results shall be by section of the article.

Annexes

Tables and adequate sources

Thanks

Indicate if they were financed by any institution, University or company.

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Conclusions

Explain clearly the results and possibilities of improvement.

References

Use APA system. Should not be numbered, nor with bullets, however if necessary numbering will be because reference or mention is made somewhere in the Article.

Use Roman Alphabet, all references you have used must be in the Roman Alphabet, even if you have quoted an Article, book in any of the official languages of the United Nations (English, French, German, Chinese, Russian, Portuguese, Italian, Spanish, Arabic), you must write the reference in Roman script and not in any of the official languages.

Technical Specifications

Each article must submit your dates into a Word document (.docx):

Journal Name
Article title
Abstract
Keywords
Article sections, for example:

1. Introduction
2. Description of the method
3. Analysis from the regression demand curve
4. Results
5. Thanks
6. Conclusions
7. References

Author Name (s)
Email Correspondence to Author
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