



TÚLIO CAETANO GUIMARÃES

**JUTE TEXTILE WITH THERMAL ENERGY STORAGE
PROPERTIES**

LAVRAS – MG

2023

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Dissertação apresentada à Universidade Federal de Lavras, como parte das exigências Programa de Pós-Graduação em Engenharia de Biomateriais, área de concentração em Compósitos e Nanocompósitos Lignocelulósicos, para a obtenção do título de Mestre.

Prof. Dr. Saulo Rocha Ferreira
Orientador

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**TECIDO DE JUTA COM PROPRIEDADES DE ARMAZENAMENTO DE ENERGIA
TÉRMICA**

Dissertação apresentada à Universidade Federal de Lavras, como parte das exigências Programa de Pós-Graduação em Engenharia de Biomateriais, área de concentração em Compósitos e Nanocompósitos Lignocelulósicos, para a obtenção do título de Mestre.

APROVADA em 17 de janeiro de 2023.

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**LAVRAS – MG
2023**

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RESUMO

A crescente demanda energética global requer soluções que melhorem a eficiência energética. O setor de construção civil é responsável por grande parte deste consumo. Neste contexto, o objetivo deste trabalho foi conferir propriedades de armazenamento de energia térmica a um tecido de juta, para ser aplicado em construção civil como reforço para matrizes de cimento. Para isso, foi desenvolvido um método para incorporar um material de mudança de fase (PCM) ao tecido. A juta era imersa no PCM líquido, e em seguida era realizado o revestimento com um polímero através de imersão em uma suspensão polimérica. Foram avaliados os copolímeros de estireno-butadieno carboxilado (com ou sem a adição de 5% de óxido de zinco) e a borracha nitrílica carboxilada. Foi realizada a caracterização mecânica, morfológica e química do tecido de juta contendo PCM. Os resultados verificaram a contenção de PCM nos poros capilares do tecido com todos os tratamentos, porém com certo prejuízo nas propriedades mecânicas do mesmo. O tratamento com borracha nitrílica carboxilada conferiu melhores propriedades mecânicas, além de formar uma camada de revestimento mais fina e leve. O tecido contendo PCM foi utilizado na fabricação de um compósito laminado denominado concreto reforçado com têxtil. A avaliação das propriedades mecânicas e térmicas revelaram que a presença do PCM causa um efeito negativo nas propriedades mecânicas do material, porém foi possível verificar o efeito de armazenamento de energia térmica conferido pelo PCM, atrasando variações de temperatura do compósito na faixa de temperatura de mudança de estado físico do PCM. Concluiu-se pela validade do método proposto para a produção de compósitos cimentícios reforçados com fibras vegetais com propriedades de armazenamento de energia térmica.

Palavras-chave: Tecido de juta. PCM. Armazenamento de energia térmica. Concreto reforçado com têxtil. Revestimento.

ABSTRACT

The growing global energy demand requires solutions that improve energy efficiency. The civil construction sector is responsible for a large part of this consumption. In this context, the objective of this work was to confer thermal energy storage properties to a jute fabric, to be applied in civil construction as reinforcement for cement matrices. In order to do this, a method to incorporate a phase change material (PCM) into the tissue was developed. Jute textiles were immersed in liquid PCM, and then coated with a polymer by immersion in a polymeric suspension. Carboxylated styrene-butadiene copolymers (with or without the addition of 5% zinc oxide) and carboxylated nitrile rubber were evaluated. The mechanical, morphological and chemical characterization of the jute fabric containing PCM was carried out. The results verified the containment of PCM in the capillary pores of the textile with all treatments, but with some damage to its mechanical properties. The treatment with carboxylated nitrile rubber provided better mechanical properties, in addition to forming a thinner and lighter coating layer. The fabric containing PCM was used in the manufacture of a laminated composite called textile reinforced concrete. The evaluation of the mechanical and thermal properties revealed that the presence of PCM causes a negative effect on the mechanical properties of the material, but it was possible to verify the effect of thermal energy storage conferred by the PCM, delaying temperature variations of the composite in the temperature range of the physical state transition of the PCM. It was concluded that the proposed method was valid for the production of cementitious composites reinforced with vegetable fibers with thermal energy storage properties.

Keywords: Jute textile. PCM. Thermal energy storage. Textile reinforced concrete. Coating.

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1 INTRODUCTION

Technological and economic development, along with population growth, has caused the demand for energy on a global scale to increase rapidly over the past decades (SMIL, 2017). At the same time, the effects of global warming and the eventual depletion of natural resources demand a change in the way of continuing this development. Thus, there is a need to explore renewable and sustainable resources and technologies.

In the International Energy Agency's (IEA) (IEA, 2021b) Net Zero Emissions by 2050 Scenario, energy efficiency is the key factor enabling growth in clean energy sources to outpace the growing demand for energy services. This scenario envisages that policy and technological measures in the building sector will allow immediate and rapid improvements in the energy efficiency of buildings, particularly large-scale retrofit programs that comply with zero-carbon building standards (IEA, 2021a). In this way, the development of technologies that can improve energy efficiency is essential to mitigate the effects of global warming and make our energy consumption more sustainable.

In this context, thermal energy storage (TES) is a way to improve energy efficiency of buildings. The so-called phase change materials or "PCMs" present themselves as a promising option for passive thermal energy storage systems, due to their properties of storing large amounts of energy in the form of latent heat, and the possibility of being used in applications that require the most diverse temperatures. In the building sector, the incorporation of PCMs into construction materials allows for thermal regulation, increasing thermal comfort and energy efficiency in buildings. The use of these materials makes a significant contribution to sustainable construction (CUNHA; AGUIAR, 2020).

Concrete is a widely used construction material, used in the most varied building applications, from conventional buildings to skyscrapers, bridges, dams etc. Necessary in the concrete mix, and comprising up to 15% in volume, cement is the second most produced mineral material in the world (U.S. Geological Survey, 2022), corresponding to approximately 7% of all annual anthropogenic greenhouse gasses (GHG) emissions (MILLER et al., 2021), with an emission ratio of 0.9 kg of CO₂ per kg of cement produced (MAHASANAN; SMITH; HUMPHREYS, 2003).

Due to the inherent brittleness of ceramics, reinforcement of cement matrices is suitable for certain applications, in order to improve the material's fracture behavior. Reinforcements can comprise of particles, fibers or textiles (LOPES et al., 2011; FONSECA et al.,

2016). Environmental, economic and health factors have been favoring the use of plant textiles as reinforcement instead of conventional materials (AKINYEMI; OMONIYI; ONUZULIKE, 2020; ARDANUY; CLARAMUNT; Toledo Filho, 2015; Da Fonseca; ROCHA; CHERIAF, 2021; KHORAMI; GANJIAN, 2011). Due to the natural porosity of plant fibers, textiles can be potential support matrices for PCMs, enabling their incorporation into construction materials.

However, plant fibers present challenges related to their durability in cementitious matrices, which entails the need for additional treatments (TONOLI et al., 2010; SILVA et al., 2011; YAN; KASAL; HUANG, 2016). Polymeric coating treatments reduce water absorption and are effective in the process of sealing natural fibers (FERREIRA et al., 2015). This fiber sealing provided by polymer treatments can potentially be used as a way to also contain a PCM absorbed by the pores of plant fibers. With this premise, plant fibers and textiles could acquire thermal energy storage properties.

In this work, the possibility of utilizing PCM-containing jute textiles in cementitious composites, with the objective of improving their thermal performance, was investigated. These composites are intended to be applied in civil construction or animal comfort. The PCM was incorporated into jute textiles, which were then coated with polymers of carboxylated styrene-butadiene rubber (XSBR) or carboxylated nitrile rubber (XNBR) and later used as reinforcement for the manufacture of laminated composites with cement matrices.

1.1 Objectives

1.1.1 General objective

The general objective of this work was to confer thermal energy storage properties to jute fabric through the absorption of a PCM by the fabric, incorporating it into a cement matrix and producing a composite for application in civil construction.

1.1.2 Specific objectives

- Validating the proposed method of incorporation of PCM to jute textiles through immersion and containment of this PCM through a polymer coating treatment;

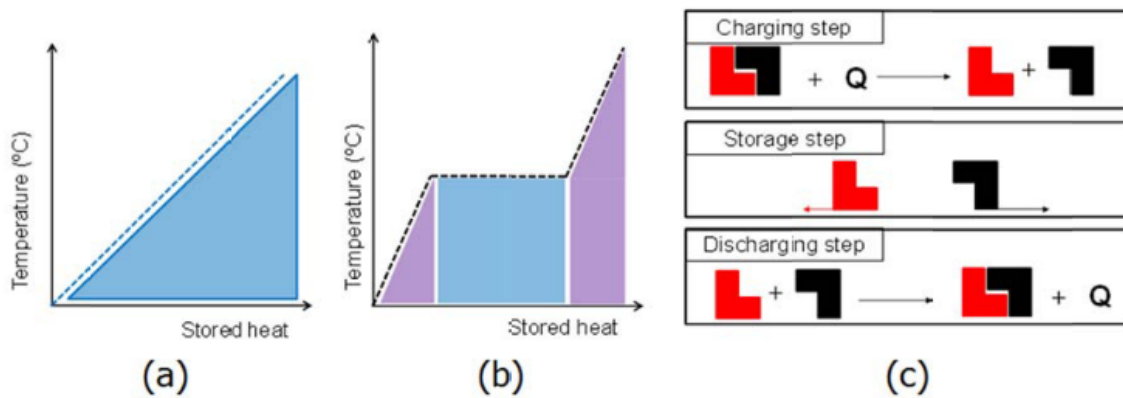
- Verifying the effect of the presence of the PCM on the mechanical and thermal properties of cementitious composites reinforced with jute fabrics;
- Evaluating the effectiveness of using different polymers for coating treatment and their effect on the chemical and mechanical properties of jute textiles and on their adhesion to a cement matrix.

2 LITERATURE REVIEW

2.1 Thermal Energy Storage

Thermal energy storage (TES) is the technology of storing energy by heating or cooling a storage medium so that the stored energy can be used later for heating and cooling applications and for power generation (SARBU; DORCA, 2019; YUAN et al., 2014). Thermal energy can be stored by physical methods (sensible heat and latent heat) and thermochemical methods (chemical reactions). The scheme in Figure 2.1 represents these three methods.

Figura 2.1 – Thermal energy storage methods. Sensible Heat (a), Latent Heat (b) and Thermochemical Reaction (c).



Source: (GRACIA; CABEZA, 2015)

Sensible heat storage is the simplest method, based on storing thermal energy by heating or cooling a liquid or solid storage medium (e.g. water, sand, molten salts or rocks) (SARBU; SEBARCHIEVICI, 2018). Due to its simplicity and low cost, it is widely used in residential and industrial applications. Water has a high specific heat, is chemically inert and has a low price, and is therefore well suited for refrigeration applications using sensible heat storage.

The amount of heat stored is a function of the specific heat of the medium, the change in temperature and the mass of the storage medium (PIELICHOWSKA; PIELICHOWSKI, 2014), according to Equation 2.1:

$$Q = \int_{T_i}^{T_f} mC_p dT = mC_{p,m}(T_f - T_i) \quad (2.1)$$

where Q - heat stored (J), T_i - initial temperature ($^{\circ}$ C), T_f - final temperature ($^{\circ}$ C), m - mass of the heat storage medium (kg), C_p - specific heat (J / kg K), $C_{p,m}$ - average specific heat between T_i and T_f (J / kg K).

Latent heat storage is based on the absorption or release of heat during the phase change of the material. For this reason, latent heat storage materials are called Phase Change Materials, or PCMs. Unlike sensible heat storage, heat is absorbed or released at virtually constant temperature, as shown in Figure 2. Furthermore, PCMs have several times the storage capacity compared to sensible heat storage materials, ranging from 50 to 150 kWh / t with efficiency between 75 and 90% (REDDY; MUDGAL; MALLICK, 2018).

Heat storage capacity of a PCM thermal storage system is given by Equation 2.2 (PIELICHOWSKA; PIELICHOWSKI, 2014):

$$Q = \int_{T_i}^{T_m} mC_p dT + ma_m \Delta H_m + \int_{T_m}^{T_f} mC_p dT \quad (2.2)$$

where a_m - melted fraction, ΔH_m - heat of fusion per unit mass (J / kg).

In thermochemical energy storage, energy is stored and released in the form of a chemical reaction. The thermochemical material, used to store thermochemical energy, undergoes a reversible physical process involving two substances or a reversible chemical reaction (Singh Rathore; SHUKLA; GUPTA, 2020), in the following form (Equation 2.3):



where Q is the heat required to dissociate components A and B of compound AB. Energy is absorbed in the endothermic direction of the reaction and released in the exothermic direction. The heat (Q) absorbed or released in the reaction is given by Equation 2.4 (BAPTISTA, 2017):

$$Q = ma_m \Delta H_R \quad (2.4)$$

Thermochemical energy storage is still in the early stages of development. There are practical limitations that preclude the use of potential reagents, such as poor operating conditions (i.e., too high a charge temperature), too low an energy density and discharge temperature, corrosivity, thermal/chemical instability, environmentally unfriendly production, or high cost (KRESE et al., 2018).

2.2 Phase Change Materials (PCMs)

Phase Change Materials (PCMs) are substances that absorb or release large amounts of latent heat when undergoing a change in their physical state, i.e. from solid to liquid and vice versa. In a heating or cooling process, this phase change occurs as soon as the material reaches its specific phase change temperature. During absorption or release of latent heat, the temperature of the PCM remains practically constant. PCMs' property to absorb and release large amounts of heat in a controlled manner can be utilized to improve the thermal performance of various end-use products to which PCMs are applied. The latent heat absorbed by the PCM can be stored there. Therefore, PCMs are considered highly efficient thermal storage media (PAUSE, 2010).

The most common PCM that can be cited is water. Water is widely used as a phase change material for cold storage because it is cheap, safe and has a high specific heat (334 kJ kg^{-1}). However, the high level of supercooling of the water during the freezing process causes degradation of the system's performance and can cause charge storage problems when the heat transfer medium is not at sufficiently low temperatures to overcome the effect of supercooling (RASTA; SUAMIR, 2019).

According to ASHRAE (American Society of Heating, Refrigerating and Air Conditioning Engineers), thermal comfort can be defined as a "condition of mind that expresses satisfaction with the thermal environment and is subjectively evaluated". Surprisingly, although climates, living conditions and cultures vary widely around the world, the temperature people choose for comfort under similar conditions of clothing, activities, humidity and air movement are very similar (ASHRAE, 2017).

There are two types of constructive solutions to achieve thermal comfort in a given environment: passive and active. Passive solutions are characterized by the use of strategies such as: the implementation of the architectural project, the shape of the building, the distribution of spaces, use of the local climate, location and size of the frames, protections, the dimensioning of the constructive elements and the definition of the materials according to their thermal inertia. When passive strategies do not achieve thermal comfort, active solutions that allow for acclimatizing the built environment are considered (PINTO, 2018). Due to their properties, PCMs are typically used for passive thermal energy storage systems.

PCMs find applications in construction industries, automotive sector, solar energy installations and textiles. In recent years, a growing number of applications have emerged, including those in electronics and medicine. Traditional sectors such as the construction industry are being modernized by new and more sophisticated TES materials for smart textiles and thermo-regulated biomaterials, etc. (FARID et al., 2004; TYAGI et al., 2012).

The use of PCMs in construction appears to be very beneficial; PCM can lower energy consumption, offset peak loads of cooling energy demand, lessen temperature fluctuations by providing a thermally comfortable environment, and reduce electricity consumption (SOUAYFANE; FARDOUN; BIWOLE, 2016). Life cycle assessment studies of phase change materials for construction applications have concluded that, considering the manufacturing, operating and disposal phases, the construction of buildings with PCM incorporation is more environmentally friendly than their reference cases (KYLILI; FOKAIDES, 2016).

Despite presenting themselves as promising alternatives for conventional materials in thermal energy storage applications, PCMs are not yet widely used due to practical and financial limitations. Most PCMs have low thermal conductivity, and can undergo large volume changes during phase change. Many of the papers about PCMs published recently look for alternatives to improve the performance of these materials and overcome the inherent problems that limit their use. Furthermore, the costs of thermal energy storage systems based on latent heat are generally higher than those based on sensible heat. The economic viability of a TES greatly depends on the application and operation needs, including the number and frequency of storage cycles (SARBU; SEBARCHIEVICI, 2018).

2.2.1 Relevant properties of phase change materials

PCM properties have a direct impact on human comfort. Therefore, the choice of a PCM for a given thermal energy storage application requires careful examination of the thermophysical, kinetic, chemical, economic, and environmental properties of the various available candidates, comparing their merits and demerits, and in some cases requires some degree of compromise. (MEMON, 2014).

Among the relevant thermal properties for phase change materials for applications in thermal storage systems, the temperature at which the phase change occurs is highlighted. This temperature should be as close as possible to the operating temperature of the potential ap-

plication for the system to effectively absorb or release the required heat. Under temperature conditions outside the phase change temperature range, PCM behaves like any other material.

The latent heat of fusion indicates the material's ability to store energy. In addition, the specific heat of the material can also contribute to the storage of energy in the form of sensible heat, although the magnitude of the latent heat is much higher. Therefore, it is desirable for the material to have a high latent heat of fusion and a high specific heat. Thermal conductivity is also an important factor to consider. A high thermal conductivity implies efficient heat transfer between the thermal storage system and the environment. PCMs, however, generally have low thermal conductivities (CABEZA et al., 2011).

Long-term stability of PCMs is required by practical applications of latent heat storage, and therefore there should not be major changes in the thermal properties of PCMs after going through a large number of thermal cycles (ZHOU; ZHAO; TIAN, 2012).

Economic aspects should also be considered when selecting a PCM; it is desirable that the material is highly available, abundant and competitive with other heat storage alternatives.

It would be difficult to obtain a material that meets all these criteria, mainly due to the fact that most PCMs have low thermal conductivity. Other challenges such as supercooling, leakage during phase change and flammability also limit the application of these materials. However, several techniques have been researched to improve the performance of PCMs and reduce these limitations. There are also several online tools and software with data on a vast amount of materials that help in the selection process, such as CES (Cambridge Engineering Selector), MatWeb, MATERIA etc (NAZIR et al., 2019).

The PCMs used in this work are CrodaTherm™ 15 and CrodaTherm™ 24. These organic PCMs based on biomaterial have transition temperatures of about 15 and 24°C, respectively, thus being in the liquid state at room temperature, which facilitates their handling. They have low flammability, high renewable carbon content and are expected to be readily biodegradable. Table 2.1 contains detailed information about the properties of these PCMs, according to manufacturer data.

Tabela 2.1 – Physical properties of CrodaTherm 15 and CrodaTherm 24 PCMs

PROPERTY	CrodaTherm 15	Crodatherm 24	UNIT
Thermal properties by differential scanning calorimetry (DSC)			
Peak melting temperature	15	24	°C
Latent heat, melting	177	184	kJ/kg
Peak crystallisation temperature	9.5	22	°C
Latent heat, crystallisation	-176	-182	kJ/kg
Thermal properties by three-layer calorimetry (3LC)			
Peak melting temperature	13	23	°C
Total heat capacity, melting	207	207	kJ/kg
Peak crystallisation temperature	12	23	°C
Total heat capacity, crystallisation	212	213	kJ/kg
Other properties			
Bio-based content	100	100	%
Density at 10/20°C (solid)	986	906	kg/m ³
Density at a 20/40°C (liquid)	859	843	kg/m ³
Flash point	206	226	°C
Specific heat capacity (solid)	2.0	3.7	kJ/kg °C
Specific heat capacity (liquid)	1.9	2.2	kJ/kg °C
Volume expansion (10–25/20–40°C)	4.3	7.5	%
Thermal conductivity (solid)	0.29	0.22	W/m °C
Thermal conductivity (liquid)	0.10	0.16	W/m °C
Thermal cycles without change in properties	10000	10000	cycles

2.2.2 Challenges in using PCMs

Despite their potential for application in thermal energy storage systems, PCMs present some challenges that limit their use. Some of these are discussed below.

Supercooling is a phenomenon in which the PCM remains in the liquid state at a temperature lower than its solidification temperature, due to a delay in the onset of crystallization. This causes latent heat to be released over a wider temperature range, which is inefficient for energy storage applications. Thus, supercooling is a key figure and a critical issue from a practical point of view and understanding the factors and methods to control supercooling are fundamental for advancing thermal energy research and technology (SAFARI et al., 2017).

Non-pure PCM substances, such as hydrated salts and some eutectic PCMs, may separate into two or more different layers during the melting process due to their difference in density, which makes the PCM no longer homogeneous (ZHANG; XIAO; MA, 2016).

In applications where the PCM is incorporated directly into the material, leakage may occur due to the increase in volume during the phase change. Leakage of PCMs causes a decline in heat storage capacity and also affects the appearance and safety of buildings (GUAN et al., 2015).

The thermal conductivities of most organic PCMs are less than 0.4 W/(m.K), while those of most inorganic PCMs are less than 1.0 W/(m.K). This low thermal conductivity of pure PCMs leads to a slow rate of heat transfer, resulting in slow charging and discharging of heat in thermal energy storage applications, as well as the inability to maintain constant temperature in temperature control systems (ZHANG; XIAO; MA, 2016).

In recent years, concern has grown about fire resistance to protect buildings from fire risks. Organic phase change materials, in particular paraffin waxes, present some flammability hazards which raise some concerns about the fire resistance of products where this material is applied. One solution that has been studied is the introduction of a stabilizer material with flame retardant properties, such as magnesium hydroxide and silica (CUNHA; AGUIAR, 2020).

2.2.3 PCM performance improvements

Several efforts in recent research have been carried out in order to improve the performance of PCMs for applications in thermal storage systems. In general, most techniques are aimed at improving heat transfer or containing PCMs. Some of these are discussed in this section.

Encapsulation is the process by which a particle is surrounded by a coating material or embedded in a matrix (homogeneous or heterogeneous) in order to form a capsule. Capsules can have a regular shape (for example, spherical, tubular and oval) or they can be made of an irregular shape (NAZIR et al., 2019). Encapsulation of PCMs is mainly useful to contain the materials, preventing leakage during the phase change due to the increase in volume, which can occur in direct incorporation. In addition, the reactivity of the PCM with the external environment can be reduced and the thermophysical properties and lifespan of the material can be improved. Based on the size of the capsule, the PCM encapsulation can be classified as follows (SALUNKHE; SHEMBEKAR, 2012):

- macroencapsulation (above 1 mm);
- microencapsulation (1-1000 μm) and

- nanoencapsulation (1-1000 nm).

Macroencapsulation consists of including the PCM in some form of container, such as tubes, bags, spheres, panels or others, with dimensions greater than 1 mm. Macroencapsulation makes material transport and handling easier, as well as allowing for appropriate designs for each application.

Compared to macroencapsulation, microencapsulation of PCMs provides faster charging and discharging rates due to shorter distance for heat transfer. However, the lower mass ratio of PCM to coating (1:1) greatly reduces the energy storage density of the storage media and increases the capital cost.

In the microencapsulation process, PCM particles are surrounded or embedded in a homogeneous matrix of size 1-1000 μm , thus forming the 'MPCM (Microencapsulated Phase Change Material)'. The main advantage of microencapsulation is related to the large surface area of the capsules, which allows a higher rate of heat transfer per unit volume. In addition, microencapsulation can improve the chemical and thermal stability of the material and its ability to resist volume change during phase change (MEMON, 2014).

Microcapsules can be obtained by many methods, which include physical methods, chemical methods and physico-chemical methods (CHEN; FANG, 2011; HUANG et al., 2019; NAZIR et al., 2019):

- Physical methods: spray drying, spray cooling, air suspension coating, supercritical fluid method, centrifugal extrusion, electrostatic precipitation, etc.
- Chemical methods: interfacial polymerization, in situ polymerization, suspension cross-linking method, emulsion polymerization, electroplating, etc.
- Physical-chemical methods: coacervation, sol-gel method, ionic gelation.

Nanoencapsulation is one of the most recently developed techniques to encapsulate PCMs to prevent PCM leakage, improve thermophysical properties, improve heat transfer and increase reliability (charge-discharge life cycle) (NAZIR et al., 2019).

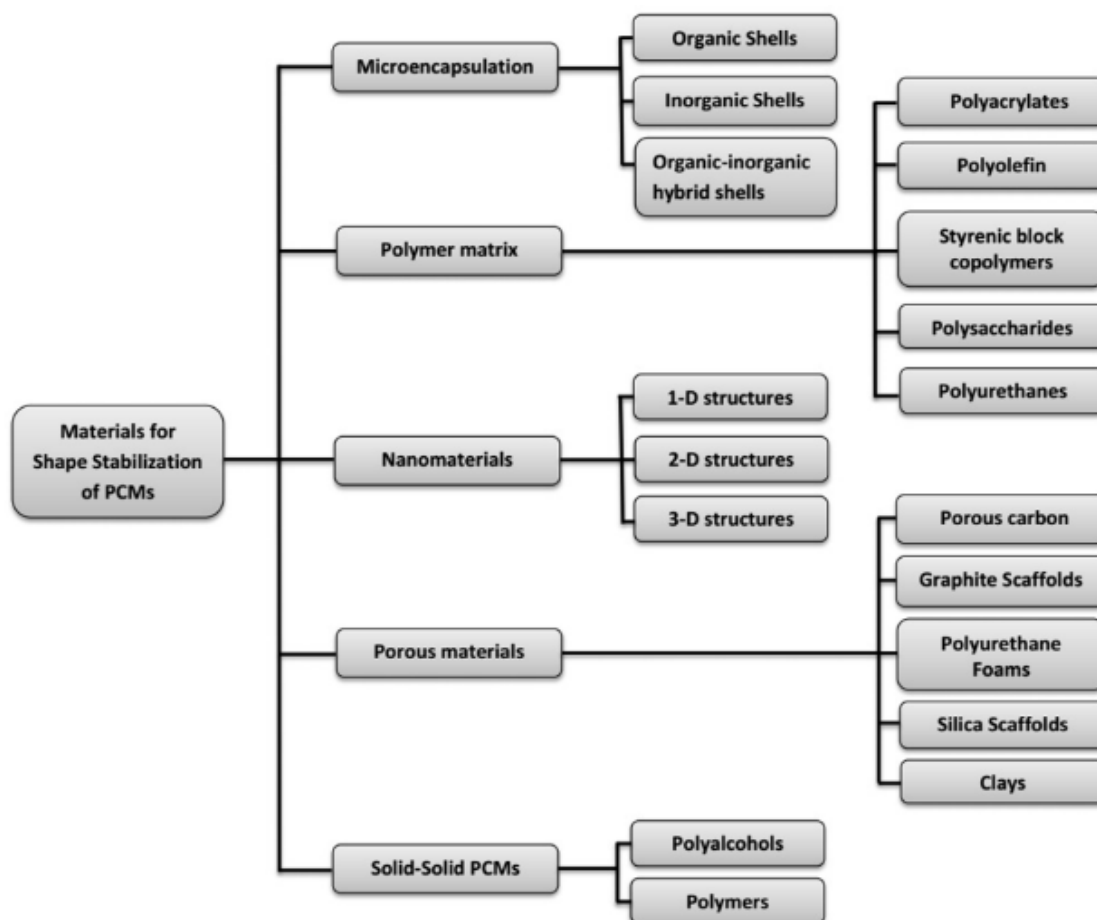
Shape stabilization of organic PCMs consists of wrapping them in a support matrix, which can consist of a variety of materials, including polymers, porous materials and nanomaterials, as shown in figure 2.2. Most researchers look at shape stabilized PCMs (in English: shape-stabilized phase change materials, or ss-PCMs) attribute to the main advantages of these

the insignificant volume change in the phase change compared to the solid-liquid phase change, which makes them efficient to prevent leakage, and the opportunity to use them in some cases without encapsulation. Furthermore, shape stabilization of PCMs is a great opportunity to use inexpensive and commonly available materials, such as naturally occurring materials, as supports. Currently, porous inorganic minerals are considered viable support matrices for organic PCMs (CÁRDENAS-RAMÍREZ; GÓMEZ; JARAMILLO, 2019; KENISARIN; KENISARINA, 2012).

In Figure 2.2, there are examples of the various types of support materials for shape stabilization. Shape stabilization of PCMs can be done through (UMAIR et al., 2019):

- Microencapsulation: consists of involving a PCM core by a protective layer of an appropriate material, as previously discussed;
- Polymer matrix: shape stabilization using polymer matrix is a simpler and cheaper method than microencapsulation. High-density polyethylene and polymethylmethacrylate (PMMA) matrices provide excellent thermal stability and chemical compatibility with organic PCMs;
- Nanomaterials: The advent of nanomaterials has opened a way to manufacture PCM composites with superior properties. The development of a 3D structured support matrix allows for maximum PCM entrapment and a superior capacity to store thermal energy in the form of latent heat;
- Porous materials are attractive as support materials for shape stabilization of PCMs due to their low density, relatively good thermal conductivity and excellent adsorption ability. Expanded graphite, expanded perlite and diatomite are widely used with organic PCMs in construction;
- Solid-solid: although solid-solid phase changes have less thermal storage capacity, their small volume variation makes PCMs with this type of phase change achieve shape stability without the need for encapsulation. These PCMs can also be used as matrices for shape stabilization of solid-liquid phase-change PCMs.

Figura 2.2 – Classification of support materials used for shape stabilization of organic PCMs.



Source: (UMAIR et al., 2019)

2.2.4 PCMs in civil construction

The incorporation of PCMs in construction materials allows thermal regulation, increasing thermal comfort and energy efficiency in buildings. The use of these materials significantly contributes to sustainable construction, based on their contribution to the three dimensions of sustainable development: social, economic and environmental (CUNHA; AGUIAR, 2020).

There are several possibilities for incorporating a PCM into building materials with the aim of changing their thermal properties: the PCM can be incorporated into floors, walls or ceilings and can also be part of more complex thermal systems, such as heat pumps and solar panels. The great advantage of incorporating PCM in buildings is the vast area they offer for storage and heat transfer (LI et al., 2018).

PCM can be incorporated into concrete by direct incorporation, immersion, form-stable PCM composites, and encapsulation. In the immersion method, construction products are dipped in liquid PCM to absorb the material by capillarity. However, PCM can interfere with the hydration products of building materials and affect their mechanical properties and durability, especially after a large number of thermal cycles, due to leaks. Typically, this technique is used to incorporate PCM into lightweight aggregates. The porous structure of lightweight aggregates is ideal for containing PCM in concrete. However, these materials need to have adequate characteristics, such as porosity, pore size, aggregate size and surface area (CUNHA; AGUIAR, 2020; MEMON et al., 2015).

The study by Mankel et al. (2019) reported the results of an extensive experimental research program that aimed to investigate the thermal energy storage performance of various mortars made with Recycled Brick Aggregates (RBAs) while filled with PCMs. In particular, this work showed the ability to immobilize significant amounts of PCMs (here paraffins) inside the highly capillary pores of RBAs (including fissures) (MANKEL et al., 2019).

Vegetable fibers are naturally porous, which makes them potential candidates for incorporation of PCMs through the immersion method. In addition, existing treatments with polymeric coating are capable of sealing these fibers, in order to potentially immobilize the PCM inside, preventing leaks. With this, it is expected to produce composite materials capable of storing significant amounts of thermal energy. The use of plant fibers and fabrics as reinforcement for cement matrices is discussed below.

2.3 Natural fibers for reinforcement

Cement-based matrices are brittle and, under the action of small tensile efforts or deformations due to elongation, tend to form cracks (LOPES et al., 2011). The inclusion of fibers, forming composites, is intended to reinforce its microstructure, increasing the impact resistance of this brittle matrix and minimizing the effects of shrinkage, mainly to reduce matrix cracking (FONSECA et al., 2016). The presence of fibers within the cement matrix promotes an anchoring effect through the cracks and, consequently, the post-cracking behavior of the composite is significantly improved (FERREIRA et al., 2016).

However, the final properties of the composite do not depend only on the presence of fibers, but also on the manufacturing method used. According to Ardanuy et al. (2015), in

order to develop composites with desirable mechanical properties, one should seek to obtain a homogeneous dispersion of fibers in the matrix; a well-balanced interaction between the cement matrix and the fibers to allow for fiber pullout; low matrix porosity; an optimized percentage of fibers: enough to reinforce the material, but allowing the continuity of the matrix (ARDANUY; CLARAMUNT; Toledo Filho, 2015).

Due to its good mechanical properties, good thermal insulation and resistance to environmental influences such as solar radiation, rain and frost, asbestos fibers were commonly used as reinforcement for cement-based building materials (WINKLER, 2015). However, exposure to this material and its handling are related to several health problems. Asbestos has been declared a proven human carcinogen by the US Environmental Protection Agency (EPA) and the International Agency for Research on Cancer (IARC) (KANG et al., 2013). As a result, asbestos has been banned in more than 60 countries (KAZAN-ALLEN, 2019) and many materials that were previously made with asbestos have been replaced by safer products.

Currently, the main substitute for asbestos fibers are synthetic fibers, produced from polymers. Synthetic fibers, such as polypropylene (PP) and polyvinyl alcohol (PVA), are alkali resistant and are commonly used as the main reinforcement in cement-based composites. However, its production consumes a large amount of energy and raw material of chemical and petrochemical products (SANTOS et al., 2015), which can considerably increase the cost of fiber cement production, in addition to not being renewable (DAI; FAN, 2013).

Plant fibers are increasingly recognized as a favorable substitute for synthetics that use unsustainable inputs. There are several advantages associated with plant fibers, such as their abundance, low cost, biodegradability, simple production processes, interesting physical, mechanical (low density, stiffness, tenacity and strength balanced) and thermal properties, and the fact that they are renewable and recyclable (AKINYEMI; OMONIYI; ONUZULIKE, 2020; ARDANUY; CLARAMUNT; Toledo Filho, 2015; Da Fonseca; ROCHA; CHERIAF, 2021; KHORAMI; GANJIAN, 2011). In addition to the technical and cost advantages, such products have the added attraction of meeting the growing consumer awareness of environmental, sustainability and social standards. Among the most prominent plant fibers are abaca, coconut, sisal and jute fibers, the so-called “future fibers” by the FAO (Food and Agriculture Organization of the United Nations). As the popularity of plant fibers in industrial uses expands, there are new opportunities for them to reach high end value markets. The scope of possible uses for the fibers

of the future is enormous. This was highlighted by the United Nations' declaration of 2009 as the International Year of Natural Fibers (FAO, 2021).

However, there are still challenges related to the use of plant fibers as reinforcement for cement matrices in composites. These challenges are mainly related to the durability of the fibers within the matrix, and the interaction between fiber and matrix. Results from previous studies indicate that there is a reduction of 50%, on average, in the mechanical resistance of cement tiles reinforced with vegetable fibers, after one year of exposure to natural conditions (TONOLI et al., 2010). The hydrophilic nature of natural fibers is responsible for the low adhesion to Portland cement-based matrices which, according to the literature, vary between 0.02 and more than 1 MPa, depending on the type of fiber, chemical composition and morphology (SILVA et al., 2011). Furthermore, Yan et al. (2016) maintain in their review of the use of plant fibers in cement composite materials that cellulosic fibers have highly variable mechanical properties and that the main reasons for degradation of cellulose tissue are alkaline degradation (hydrolysis) and fiber mineralization (YAN; KASAL; HUANG, 2016).

Fiber mineralization is the process of re-precipitation of cement hydration products in its interior (fiber lumen), caused by the alkalinity of the water present in the pores of the cement matrix. The matrix region (interface or transition zone) is characterized by high porosity, allowing the accumulation of water and a higher concentration of Ca(OH)_2 (TONOLI, 2009). In addition to mineralization, fiber degradation can also occur due to damage to the fiber-matrix interface. The severe weathering conditions to which composites are exposed induce the absorption and release of water, resulting in dimensional changes in the porous cement matrix and cell walls of cellulosic fibers, which generally promotes loss of adhesion at the interface between fiber and cement, resulting in detachment of the reinforcing element and degradation of the mechanical properties of the composite (TONOLI, 2009; YAN; KASAL; HUANG, 2016).

Given the relevance of the degradation mechanisms of plant fibers in the context of reinforcements for cement matrices, it is important that studies be carried out on the durability of new composites, through accelerated or natural aging tests.

Several treatments have been proposed by recent research to improve the interaction between fiber and matrix and the durability of composites, both for the cement matrix and for the plant fibers. To increase the durability of cement composites reinforced with cellulosic fibers/fabrics, some authors have suggested modifying the cement matrix, for example using low-alkali concrete and adding pozzolans such as shell ash, blast furnace slag, metakaolinite or

fly ash to Portland cement (AGOPYAN et al., 2005; NITA et al., 2004; SAVASTANO; WAR-
DEN; COUTTS, 2005). Accelerated carbonation of the cement matrix can also be used to
improve the durability of cellulose fiber cement composites, as it reduces the alkalinity of the
cement matrix, lowering the pH and making it less aggressive to cellulose fibers (CORREIA;
SANTOS; Savastano Júnior, 2015).

Treatments for plant fibers, in general, aim to ensure the dimensional stability, strength
and durability of plant fibers (Da Fonseca; ROCHA; CHERIAF, 2021). These treatments work
in several different ways, leading to increased fiber surface roughness, decreased amount of
waxes, and partial removal of most critical sugars (eg, pectin and glucose). All the presented
modifications can lead to an improvement in chemical and mechanical anchorage to a cement-
based matrix (Rocha Ferreira et al., 2020). Fiber treatments include hornification (FERREIRA
et al., 2017; FERREIRA et al., 2012), alkaline solution treatments (FERREIRA et al., 2015;
AKINYEMI; OMONIYI; ONUZULIKE, 2020) and polymer coating treatments (FIDELIS et
al., 2016; Rocha Ferreira et al., 2020), among others.

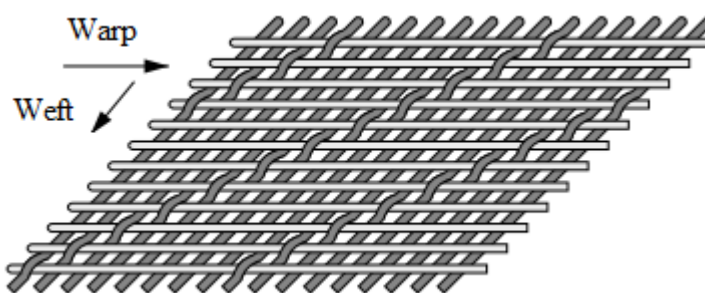
Polymeric coating treatments reduce water absorption and are effective in the natural
fiber sealing process. Styrene butadiene polymers (SBR) can be used to improve mechanical
and physical properties in cement pastes as a result of chemical reactions with calcium hydro-
xide and silicates (FERREIRA et al., 2015). The polymer forms a film that protects the fiber,
preventing contact between the fiber and hydration products; reduces water absorption by the fi-
ber, improving its volumetric stability and consequently reducing pore variation at the interface
(FIDELIS et al., 2016).

The use of carboxylated styrene butadiene rubber (XSBR) is indicated as an efficient
treatment due to its good compatibility with both the cement matrix and natural fibers. The main
reason for its efficiency may be due to the possibility of reducing the water absorption of natural
fibers and improving their chemical interaction with a cement matrix due to their carboxyl
groups. Ferreira et al. (2020) studied the influence of the XSBR coating on the mechanical
properties of plant fibers and their interface with a cementitious matrix. The results indicated
an increase in the tensile strength of all fibers studied. An increase in the maximum load was
observed during fiber pulling, as a consequence of the increase in the chemical bond between
the treated fibers and the cementitious matrix (Rocha Ferreira et al., 2020).

2.4 Plant textiles

The weaving process is one of the oldest known material processing techniques. The simplest and most common form of textile reinforcement for composites are fabrics, which consist of classic bidirectional structures, as illustrated in Figure 2.3. Fabrics are produced on looms whose rovings of fibers intertwine in mutually perpendicular positions, alternating upper and lower, which follow a certain pattern. Fabrics have two main directions: warp and weft. The warp refers to the length direction of the fabric, and the weft, in turn, is transverse to the warp. The bidirectional structure of fabrics is characterized by spacing between adjacent yarns, yarn size, percentage of fibers in each direction, and the degree of yarn packing (NETO; PARDINI, 2016; PARDINI, 2000).

Figura 2.3 – Schematic drawing of an 8HS twill fabric with respective directions.



Source: adapted from (PARDINI, 2000)

With a standardized manufacturing process, cellulosic fabrics allow control of fiber orientation and quality, good reproducibility and high productivity. Consequently, problems such as significant variations in monofilament properties, difficult dispersion and random distribution of cellulosic fibers in the cementitious matrix (i.e., which may not be along the loading direction and may not provide effective reinforcement), can be overcome with the use of cellulosic fiber fabrics. Furthermore, compared to monofilament cellulosic fibers in cementitious matrices, which only provide reinforcing effect in the longitudinal direction of the fiber, cellulosic fabrics can offer effective reinforcements to the matrix in multiple directions (YAN; KASAL; HUANG, 2016).

Textile-reinforced concrete is a high-performance cementitious composite that uses straight, parallel aligned fibers of suitable materials, for example glass or carbon, as continuous reinforcement in the form of textiles. Textile reinforced concrete is generally used for thin concrete elements or as reinforcing layers for concrete structures. Textile reinforced concrete presents

a multilinear stress-strain behavior with three distinct stages (no cracking, multiple cracking, complete cracking) (RILEM Technical Committee 232-TDT (Wolfgang Brameshuber) et al., 2016). In the case of textile-reinforced concrete, the bonding behavior is completely different from other materials used as reinforcement, since the cross section of the fabric is not homogeneous. The outer filaments of a yarn have direct contact with the matrix, that is, only a part of the fabric is anchored in the matrix. The inner filaments are not affected by cement hydration products. Thus, there is only friction between the filaments. When the reinforcement is subjected to polymer treatment, there is a complete anchorage of all the filaments, making the adhesion between the filaments greater than the adhesion of the fiber with the matrix (FIDELIS; de Andrade Silva; Toledo Filho, 2014)).

2.5 Jute fibers

Jute (*Corchorus capsularis*), illustrated in Figure 2.4, is a fiber-producing plant, well adapted to the lowland areas of the Amazon Region. It reaches a height of 3 to 4 meters and its main stem is approximately 20 mm thick, from which the fiber is extracted (HOMMA, 2016). Jute is an annual crop that takes about 120 days to grow. Jute fibers are among the most commonly used as reinforcement in concrete and mortar.

Figura 2.4 – Jute plant.



Source: (YAN; KASAL; HUANG, 2016)

Jute is mainly grown in countries like India, Bangladesh, China, Nepal and Thailand. Together they produce around 95% of the global production of jute fibers (ALVES et al., 2010). Japanese immigration in the Amazon can be pointed out as the main responsible for the introduction of jute, black pepper, among other productive chains in the north of Brazil. This

insertion began to outline a new model of agriculture for the floodplain in the Amazon, becoming an integral part of the region's economy and natural resources (SOARES et al., 2020).

Jute fibers are obtained from the plant through an extraction process that consists of cutting, maceration, crushing, drying, packaging and classification (FERREIRA et al., 2018). Maceration can be done by different techniques of bundling jute stems and soaking them in water to help separate the fibers from the stem before removal. In Table 2.2, the properties of jute fibers can be observed.

Tabela 2.2 – Physical and mechanical properties and composition of jute fibers.

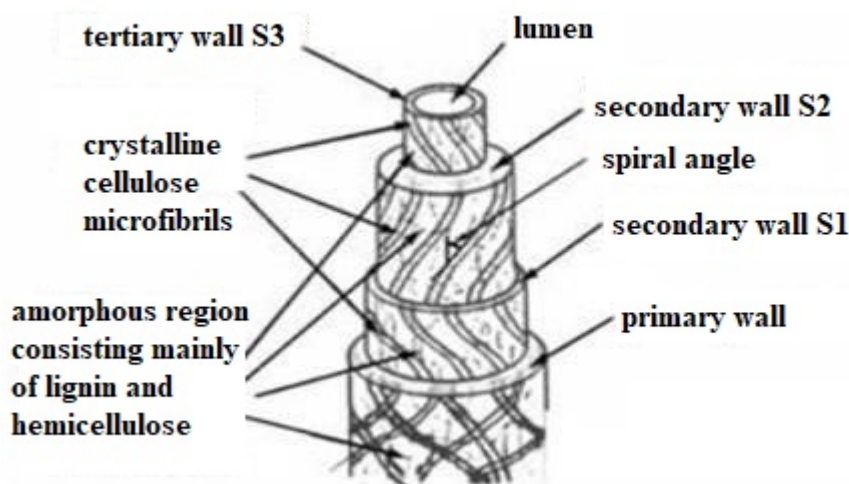
Physical properties	
Density (g/cm ³)	1.3–1.5
Length (mm)	1.5–120
Diameter (μm)	20–200
Helical Angle	7–9
Mechanical properties	
Tensile strength (MPa)	200–800
Young's Modulus (GPa)	8–78
Elongation at rupture (%)	1–8
Chemical composition	
Cellulose (%)	59–71.5
Hemicellulose (%)	13.6–20.4
Lignin (%)	11.8–13
Pectin (%)	0.2–0.4
Extractives (%)	0.5

Source: (Fornari Junior, 2017)

Vegetable fibers have a highly complex hierarchical structure. Each fiber is composed of several fibro-cells. Each fibro-cell, in turn, is formed by four main parts: primary wall, secondary wall, tertiary wall and lumen (FERREIRA, 2012).

Figure 2.5 illustrates the structure of a vegetable fiber. The main component of the cell wall is cellulose, which largely determines its architecture. Cellulose is made up of numerous glucose monomers linked end to end. Cellulose polymers are bundled into microfibrils that are about 10 to 25 nanometers in diameter. Cellulose has crystalline properties due to the orderly arrangement of its molecules in certain parts, the micelles of microfibrils. Cellulose microfibrils intertwine to form thin filaments that can wrap around each other, similar to strands on a cable. Cellulose molecules intertwined in this way have greater strength than steel of equivalent thickness (EVERT; EICHHORN, 2014).

Figura 2.5 – Structural constitution of a vegetable fiber.



Source: (KNUTH, F. A. et al., 2017)

The crystalline areas in the fiber structure are primarily responsible for stiffness, density and resistance to swelling; while the less ordered or amorphous areas are mainly related to softness, flexibility and reversible extensibility (MOHANTY; MISRA, 1995).

In addition to cellulose, the main components of plant fibers are hemicellulose and lignin, which is why they are known as lignocellulosic fibers. Several substances can also be present in natural fibers, such as acidic resins, fatty acids and alcohols. Many of these substances are soluble in water or neutral organic solvents, and are collectively referred to as extractives (SMOOK, 2001).

In contrast to cellulose, which is composed only of glucose, hemicelluloses are polymers composed of five different sugars (glucose, mannose, galactose, xylose and arabinose) (ALEXANDRE, 2018). Unlike cellulose, hemicelluloses are not crystalline and generally have an amorphous arrangement, with sugar molecules arranged in chains with some branches. In the cell wall, hemicelluloses act as a support matrix for microfibrils, apparently having a uniform distribution throughout the wall (KNUTH et al., 2017).

Lignin is a biochemically inert polymer that functions as a structural support material in plants. During the synthesis of plant cell walls, polysaccharides such as cellulose and hemicelluloses are deposited first, and lignin fills the spaces between the polysaccharide fibers, cementing them together. This lignification process causes the cell walls to harden and the carbohydrate is protected from chemical and physical damage (MOHANTY; MISRA, 1995).

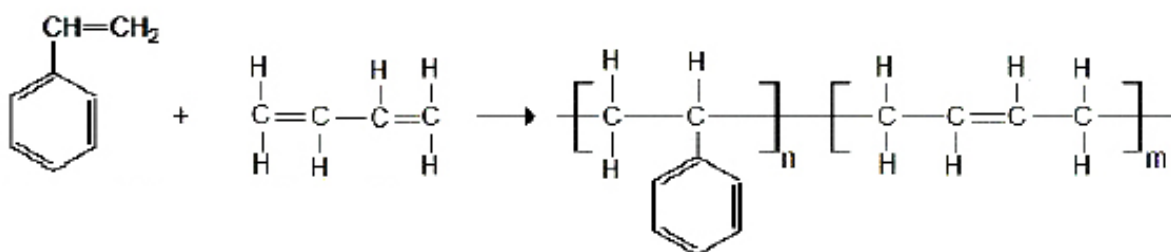
Among naturally occurring lignocellulosic fibers, jute contains one of the highest proportions of rigid natural cellulose. In the textile industry, the high stiffness of jute is often a limitation due to difficulties in fine spinning; however, in fiber-reinforced composites, jute stiffness is an important basis of design criteria (MOHANTY; MISRA, 1995).

2.6 Styrene-butadiene rubber (SBR) and nitrile rubber (NBR)

Styrene-butadiene rubber, known as SBR, is one of the most produced and commercialized synthetic elastomers. It is a co-polymer that meets numerous demands and has a relatively low cost, which justifies its wide use and consumption (Fornari Junior, 2017).

The main Raw Materials for the production of SBR are styrene and butadiene, however, other chemical components are also of fundamental importance in the co-polymerization processes, these are used in minimal quantities, such as; emulsifiers, initiators, modifiers, catalysts, reaction terminators, coagulating agents, antioxidants and antiozonants. Figure 2.6 illustrates how to obtain SBR from styrene and butadiene.

Figura 2.6 – Synthesis and structure of SBR.



Source: (CAETANO, 2010)

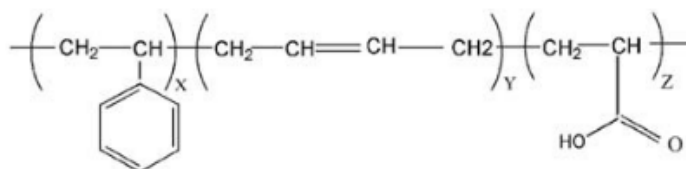
SBR is used to manufacture treads for light vehicles, shoes, carpets, hoses, among others. It is soluble in benzene, hexane, toluene and has a density of approximately 0.94 g/cm³ (Fornari Junior, 2017).

Carboxylated styrene-butadiene rubber, or XSBR, is a modified type of SBR containing carboxyl groups. With the introduction of carboxyl groups, properties such as elasticity, strength, compatibility with functional fillers and polymers and resistance to hydrocarbon solvents increase and crosslinking by non-sulfur reagents becomes possible. Due to the polarity offered by the carboxyl groups, XSBR composites with fibers or particles exhibit improved physical and mechanical properties. The presence of the carboxyl group also allows the rubber

to react with new and additional reagents, which is certainly of great importance to achieve desirable adhesion to polar substrates such as textiles, paper, etc. It also allows the polymer to be cured without any restriction with normal vulcanizing agents like sulfur, but with epoxies, polyamines and metal oxides (ALIMARDANI; ABBASSI-SOURKI, 2015).

XSBR is prepared by the emulsion polymerization of butadiene, styrene, and one or more unsaturated acids, such as acrylic acid, methacrylic acid, crotonic acid, maleic acid, fumaric acid, tectonic acid, and 3-butene-1,2,3-tricarboxylic acid. In Figure 2.7, the chemical structure of XSBR can be observed.

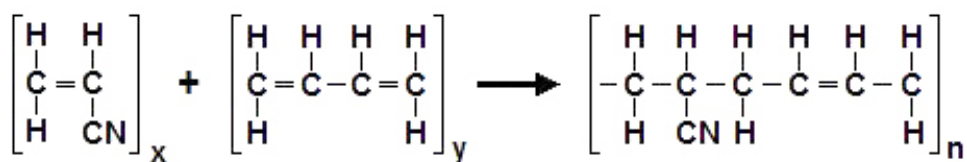
Figura 2.7 – Chemical structure of XSBR. X, Y and Z represent different concentrations of monomers.



Source: (ALIMARDANI; ABBASSI-SOURKI, 2015)

Nitrile rubber, or NBR, is another type of synthetic rubber. It is a co-polymer of butadiene and acrylonitrile, hot or cold polymerized. This type of rubber is used in applications that require resistance to oils, fuels and solvents, good abrasion resistance, elasticity or resistance to heat aging. Applications are found in textile, automotive and mineral oil industries. Figure 2.8 represents a scheme for obtaining NBR from acrylonitrile and butadiene.

Figura 2.8 – Nitrile rubber synthesis reaction.



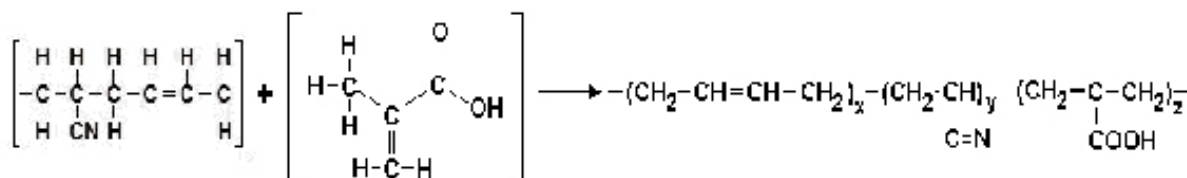
Source: (CAETANO, 2010)

The presence of acrylonitrile groups in this polymer favors the interaction with the cement. The chemical interaction between this functional group and the hydrating phases of the cement causes the formation of new phases which, in turn, cause the formation of more chemical bonds, leading to increased strength (SINGH; KHATHRI; SINGH, 2007).

As with SBR, there is a modified type of nitrile rubber containing carboxyl groups known as XNBR. As with XSBR, the carboxylic groups promote the improvement of seve-

ral properties to the carboxylated nitrile rubber, such as tensile, chemical, tear and abrasion resistance, when compared to NBR (COSSA; CARVALHO; SIRQUEIRA, 2015). Figure 2.9 illustrates the reaction to obtain XNBR from acrylonitrile butadiene and methacrylic acid.

Figura 2.9 – Synthesis reaction of carboxylated nitrile rubber.



Source: (CAETANO, 2010)

2.7 Concluding remarks

The literature review presented in this chapter included information on: thermal energy storage technology; phase change materials, including their properties, limitations, selection criteria, potential performance improvements, and applications in civil construction; plant fibers and textiles applied as reinforcements for cement matrices, including the treatment of fibers with polymer; jute fibers and styrene-butadiene and nitrile rubbers.

These were all key topics for the development of a jute fabric that can absorb and contain a PCM in its pores, at the same time as it acts as a reinforcement for a cement matrix, providing the composite with adequate thermal properties so that it can be used in applications aimed at thermal comfort.

The next two chapters were written in the form of articles, which report the methods used throughout this project, as well as the results obtained and the conclusions drawn from them. The first article focuses on the validation of the proposed method of incorporation of PCM into construction materials and the evaluation of mechanical and thermal properties of composites containing PCM. The second article focuses on the influence of utilizing different polymer treatments for the sealing of jute textiles containing PCM.

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3 ARTICLE 1: PCM-IMPREGNATED TEXTILE-REINFORCED CEMENTITIOUS COMPOSITE FOR THERMAL ENERGY STORAGE

Article

PCM-Impregnated Textile-Reinforced Cementitious Composite for Thermal Energy Storage

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Abstract: The growing global energy demand requires solutions that improve energy efficiency in all sectors. The civil construction sector is responsible for a large part of global energy consumption. In this context, phase change materials (PCMs) can be incorporated into construction materials to improve the energy efficiency of buildings. The purpose of this study was to incorporate a PCM to jute fabric, applying it in civil construction as a reinforcement for cement matrices. In order to do that, a method of immersing jute fabric in liquid phase change material, and then coating it with a polymer, was proposed. Treated jute fabric was then used to produce a laminated composite with a cementitious matrix. Morphological, mechanical and chemical characterization of jute textiles was performed, as well as an analysis of the composites' mechanical and thermal behavior. The results verified that jute textiles absorbed 102% PCM in weight, which was successfully contained in the capillary porosity of jute. The PCM was able to delay the composite's temperature increase by up to 24 °C. It was concluded that this method can be used to incorporate PCM to natural textiles, producing composites with thermal energy storage properties.

Keywords: jute textile; PCM; thermal energy storage; textile-reinforced concrete; coating

1. Introduction

Solutions related to the improvement of energy efficiency offer some of the fastest and most cost-effective actions to reduce CO₂ emissions [1]. Storage technologies, such as thermal energy storage (TES), are a central component of energy-efficient and sustainable energy systems. Phase Change Materials (PCMs) are a promising option for passive thermal energy storage systems, due to their property of storing large amounts of energy in the form of latent heat [2]. When incorporating PCM into construction materials, it

significantly contributes to sustainable construction, as it allows an enhanced thermal regulation, reducing the consumption of non-renewable energy sources [3].

Focusing on the thermal performance of buildings, which consequently helps to reduce energy consumption and GHG emission, in the last few years researchers have developed relevant works on PCM-incorporated construction materials, such as recycled materials with PCM [4,5], Bio-PCMs [6–8], cement-based systems with microencapsulated PCMs [9–11], PCM walls [12,13], PCM incorporated into lightweight aggregates [14], wood flooring with microencapsulated PCM [15], and delignified wood composite phase change materials [16], amongst others.

Despite their potential for application in thermal energy storage systems, PCMs present some challenges that limit their use, such as low thermal conductivity [17], super-cooling [18], and leakage (when PCM is directly incorporated into the material) [19]. In order to prevent PCM leakage, shape stabilization methods can be applied, consisting of employing a support matrix, such as polymers, porous materials, or nanomaterials [20–22] to contain the PCM. Numerous porous materials have been investigated as containers for PCM, including expanded graphite [23], diatomite [24], expanded vermiculite and perlite [25], kaolinite [26], pumice [27], hydroxyapatite [28], cellulose, agarose and chitosan [29], and cellulose aerogel [30]. However, these works have not yet utilized natural raw materials as containers for PCM and have not yet incorporated PCM into reinforcements for concrete.

Due to their morphological characteristics, plant fibers are naturally porous. Hemp, sisal, and jute fibers find their porosity to be 2.46, 10.85, 11.36, and 73.95%, respectively [31], which makes them potentially suitable containers for PCMs. Additionally, plant fibers used as reinforcement for cement-based matrices have demonstrated that polymeric coating treatments can reduce water absorption and are effective in the sealing process of the fibers [32–34]. Previous studies [35,36] have developed a textile-reinforced concrete utilizing polymer-coated jute fabric as reinforcement. The treatment of jute fabric with carboxylated styrene–butadiene rubber (XSBR) increased the tensile strength and strain capacity of the composites.

This work proposed a novel method of utilizing the sealing of the fibers provided by polymeric coating as a way to also contain the PCM, preventing its leakage. Jute fabric was immersed in liquid PCM, absorbing it by capillarity, and then it was coated with XSBR. Coated fabric was then used as a reinforcement for laminated composites. Bending tests were performed in order to evaluate the mechanical performance of these composites, as well as heating and cooling cycles in thermally insulated chambers to evaluate their thermal behavior.

2. Materials and Methods

2.1. Materials

Jute fibers originate from the stem of the *Corchorus capsularis* plant, through a process of cutting, retting, shredding, drying, packing, and sorting [37], while their textiles are produced via spinning and weaving processes. Acquired from the company Companhia Têxtil Castanhal (São Paulo-SP, Brazil), the textile used in this work has a mesh opening of 3 yarns/cm in the transverse direction and 3.5 yarns/cm in the longitudinal direction, as depicted in Figure 1.

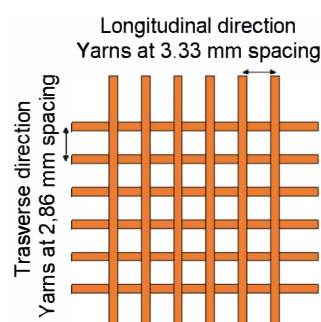


Figure 1. Jute fabric mesh opening.

A bio-based organic PCM with a phase change temperature of approximately 24 °C was selected for this work (CrodaTherm 24-LQ-(GD), from Croda do Brasil Ltda, Campinas, SP, Brazil), with a phase change temperature of approximately 24 °C. The physical properties of this PCM are found in Table 1. A carboxylated styrene–butadiene rubber, XSBR NTL-218, with a medium styrene content, manufactured by Nitriflex (Duque de Caixas-RJ, Brazil) was chosen as the polymer used for coating the natural fibers.

Table 1. Physical properties of CrodaTherm 24.

Property	Typical Value	Unit
Thermal properties by differential scanning calorimetry (DSC)		
Peak melting temperature	24	°C
Latent heat (melting)	184	kJ/kg
Peak crystallisation temperature	22	°C
Latent heat (crystallisation)	−182	kJ/kg
Thermal properties by three-layer calorimetry (3LC)		
Peak melting temperature	23	°C
Total heat capacity (melting)	207	kJ/kg
Peak crystallisation temperature	23	°C
Total heat capacity (crystallisation)	213	kJ/kg
Other properties		
Bio-based content	100	%
Density at 20 °C (solid)	906	kg/m ³
Density at 40 °C (liquid)	843	kg/m ³
Flash point	226	°C
Specific heat capacity (solid)	3.7	kJ/kg °C
Specific heat capacity (liquid)	2.2	kJ/kg °C
Volume expansion 20–40 °C	7.5	%
Thermal conductivity (solid)	0.22	W/m °C
Thermal conductivity (liquid)	0.16	W/m °C
Thermal cycles without change in properties	10,000	cicles

Finally, the following materials comprise the cementitious matrix mix constituents: Portland cement CP II F32 (equivalent to the European CEM II [38], manufactured by CSN company at Serra, ES, Brazil); metakaolinite (manufactured by Metacaulim do Brasil, at Jundiaí, SP, Brazil); river sand passing through a 1.19-mm sieve; fly ash; BASF's MasterGlenium 51 superplasticizer (PA type) with a solids content of 51%.

2.2. Methods

2.2.1. Scanning Electron Microscopy

Untreated and treated jute yarns were investigated with a FEI Quanta 400 scanning electron microscope (SEM). The specimens were sputter coated with a thin layer of about

20 nm of silver using a BAL-TEC SCD 005 sputter coater. This process is necessary to provide conductivity to the specimens of non-conductive materials, such as these natural fibers. This improves the quality of the SEM images and analysis. It is worth mentioning that silver was used, instead of the more commonly used gold, because it costs much less, and it can provide good quality SEM images in a low magnification [39]. The microscope was operated under 20 kV of acceleration voltage, in a high vacuum mode. The images were acquired using a back-scattered electrons detector.

2.2.2. Micro-Computed Tomography (Micro-CT) Scans

Micro-CT scan images of untreated and treated jute yarns embedded in cement matrices (w/c ratio = 1:0.3) were obtained in order to analyze the effect of the polymer coating treatment on the interface of the system. For the image acquisition, single yarns were placed inside 2 mL microcentrifuge tubes, and then filled with the cement paste.

For the micro-CT acquisition, a V/TOMEX/M (GE Measurement & Control Solutions, Wunstorf, Germany) was used. The micro-CT parameters for the acquisition included a voltage of 70 kV, an energy of 280 μ A, 5 frames, and an Al filter with a thickness of 0.5 mm. The geometry had a magnification of 9.37, pixel size of 21 μ m, and a total of 1200 projections.

2.2.3. Immersion of Jute Fabric in Liquid PCM and Polymer Solution

Immersion is reported as a suitable method for incorporating PCM into porous materials, such as lightweight aggregates [3,14]. Due to jute fibers' natural porosity (11%) [31], the immersion method can be used to incorporate PCM into jute fabric.

The jute fabric was immersed in liquid PCM. Depending on weather conditions, the PCM was in a liquid state at room temperature ($t \geq 24$ °C.). However, it was melted and kept in a kiln at 60 °C until the immersion procedure when necessary. PCM was impregnated into the fibers' pores by capillary absorption, as depicted in Figure 2a; excess liquid was then removed in tray 2 (Figure 2b). Since the leakage effects of liquid PCM inside the open capillary pores of jute fibers can occur during a phase change, a maximum degree of saturation of 80% volume was applied [4]. Thus, a 2-min immersion time was recommended to obtain that degree of saturation of PCM in jute fabric.

Jute textiles were then treated by total immersion in an XSBR polymer bath at room temperature for 50 min, as shown in Figure 2c, followed by drying in open air for 72 h, in accordance with Fidelis et al. [33]. In addition, the influence of a multilayered coating was evaluated in 3 cm $\times$ 3 cm fabric specimens. In a 3-time process, after a coating layer, the fabric mass was measured, as well as its thickness. Finally, the non-occurrence of liquid PCM leakage was verified: by mass loss (during 14-day mass measurement) and by the difference between densities (after 3-day water immersion).

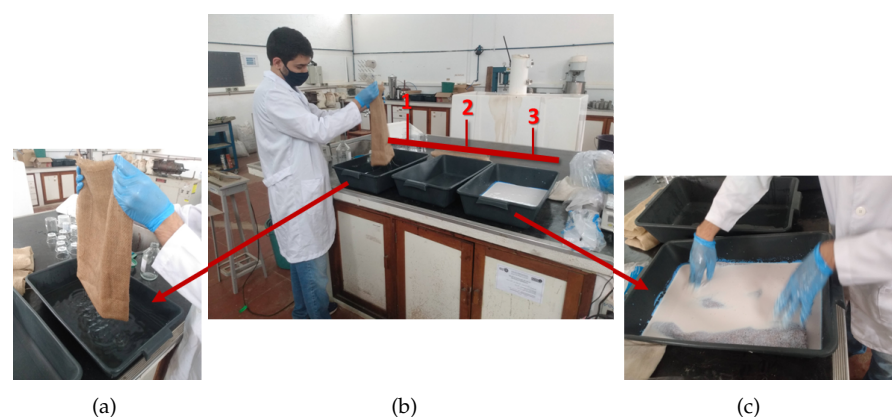


Figure 2. Treatment of jute fabric: (a) immersion in liquid PCM, (b) removal of PCM excess, and (c) coating by immersion in XSBR bath.

Along with this procedure, the fabric's capacity for PCM absorption was determined by Equation (1):

$$A(\%) = \frac{m_t - m_0}{m_0} \times 100\%, \quad (1)$$

where A is the absorption, represented by the mass gain, and m_t is the specimen mass at time t and m_0 is the initial mass; 3×3 cm untreated specimens were tested.

2.2.4. Mechanical Characterization of Jute Fibers

Under a controlled temperature of 18 °C, direct tensile tests were performed on an AROTEC WDW-20E universal testing machine (Arotec Indústria e Comércio company from Cotia-SP, Brazil), in accordance with ASTM C1557 [40]. Measured with a 5 kN load cell, with a constant displacement rate of 1.25 mm/min, 250 mm long yarn samples and 60×250 mm (width $\times$ length) samples were selected (Figure 3a). In order to avoid specimens' slippage during the test, rubber sheets were glued to each end (Figure 3b).

The tensile strength of the yarns was determined by dividing the maximum force measured in these tests by the average cross-sectional area (0.48 mm^2), as measured by Fidelis [35]. The area of the fabric was determined based on the average number of yarns in the specimens of these dimensions.

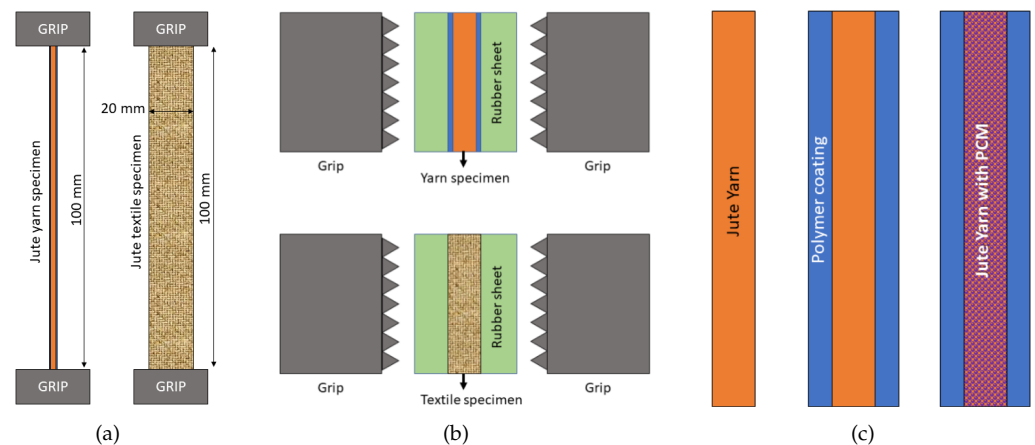


Figure 3. Samples preparation: (a) dimensions for the direct tensile tests, (b) details of rubber sheets glued at specimens' ends to avoid slippage during testing, and (c) yarn treatments.

2.2.5. Chemical Characterization of Jute Fibers

Fourier-transform infrared spectroscopy (FTIR) and thermogravimetry analysis (TGA) were carried out in order to evaluate the interaction of the jute fibers with the proposed components, i.e., PCM and XSBR; the following jute samples were used: in natura, coated with XSBR polymer, impregnated with PCM, and coated with XSBR polymer (Figure 3c).

For the FTIR analysis, the attenuated total reflectance (ATR) sampling method was used. The spectral range analyzed was from 4000 to 675 cm^{-1} , with 24 scans and a resolution of 4 cm^{-1} . For the thermogravimetry analysis, the samples were initially submitted to a constant temperature of 40 °C for 30 min in order to remove the free water present in the material. Then, the samples were heated from 40 to 1000 °C at a constant heating rate of 10 °C/min , under an N_2 atmosphere. Both tests were based on the method proposed by Ferreira et al. [34].

2.2.6. Composite Production

Laminated composites containing three layers of jute fabric were produced. As depicted in Figure 4, the composite comprises of seven layers, alternating between mortar and jute fabric, totalling 15 mm thick.

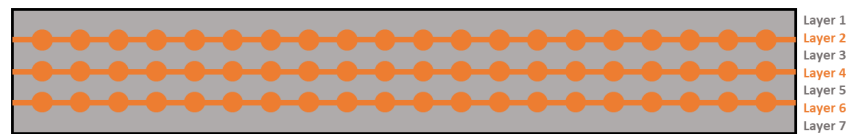


Figure 4. Composite layer distribution.

A ratio of 1:0.5:0.4 (binding:sand:water/cement ratios (% weight)) was adopted for the mortar, with 1.5% of superplasticizer [41]; the mix proportions of the mortar can be observed in Table 2. For the mortar reinforcement, in natura jute fabrics (2-hour hot water washing and 24-hour drying at 60 °C [42]), as well as XSBR-coated jute fabrics (with and without impregnated PCM), were used. The mortar was produced as follows: mixing of the dry materials; gradual addition of water; addition of superplasticizer; mixing until the matrix was homogenized.

Table 2. Mix proportions of the mortar.

Component	Materials Consumption (kg/m ³)
Cement	548
Metakaolinite	438
Fly ash	108
Sand	548
Water	438

The molding process [35] is illustrated in Figure 5. On a 485 × 650 mm mold, paper ribbons delimited the casting area (Figure 5a). After the first mortar layer was applied, the jute fabric was anchored and stretched on the threaded studs (Figure 5b,d) up to the last (and fourth) mortar layer (Figure 5e). The plates were then placed in a curing chamber for 28 days, after which they were sawed into four 75 × 325 mm specimens for the mechanical tests, and one 170 mm × 240 mm specimen for the thermal test, as illustrated in Figure 6a.

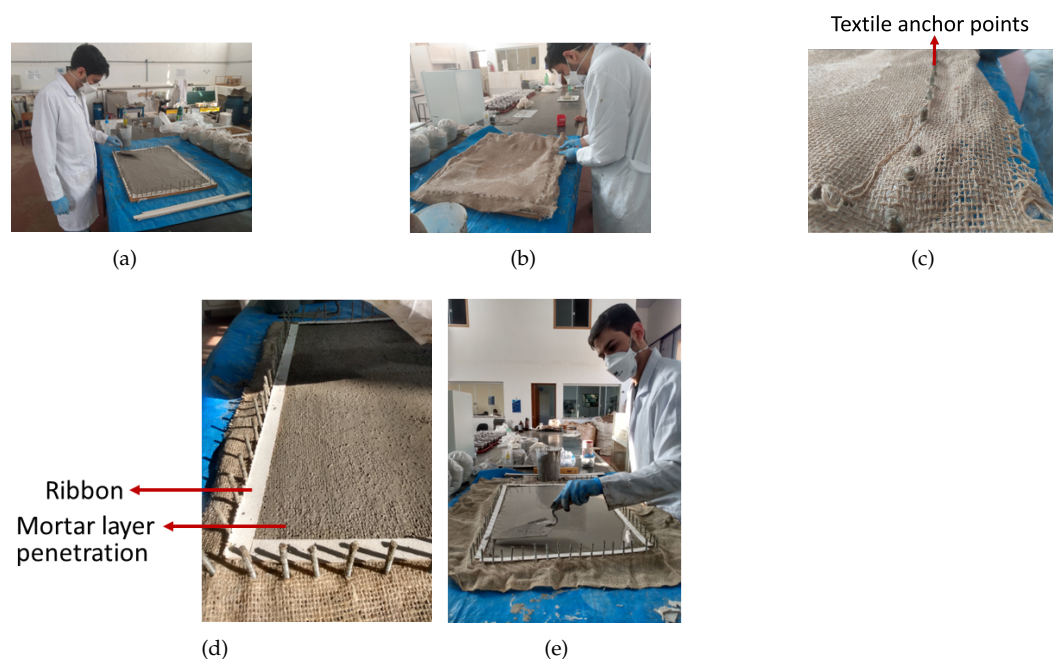


Figure 5. Composite molding: (a) first layer of mortar, (b,c) fixation of jute fabric, (d) mortar layer penetration, and (e) last layer of mortar (e).

2.2.7. Mechanical Characterization of the Laminated Composites

RILEM's four-point flexural tests were carried out to evaluate the mechanical behavior of the composites [43]. In a Shimadzu universal testing machine (Shimadzu Corporation, Kyoto, Japan), at a room temperature of 23 °C, the tests were performed under a displacement rate of 0.3 mm/min, and measured with a load cell of 100 kN. The span length between supports was 270 mm, and the distance between loading pins was 90 mm.

2.2.8. Thermal Behavior of Composites

The thermal behavior of the composites was evaluated through heating and cooling cycles of two thermally insulated chambers, simulating outdoor conditions. A 15 W incandescent light bulb was used as a heat source and eight thermocouples were used to register the evolution of the temperature in four different regions: one in each chamber (air temperature), one on the lower face of the specimen, and one on the upper face. The setup is illustrated in Figure 6b.

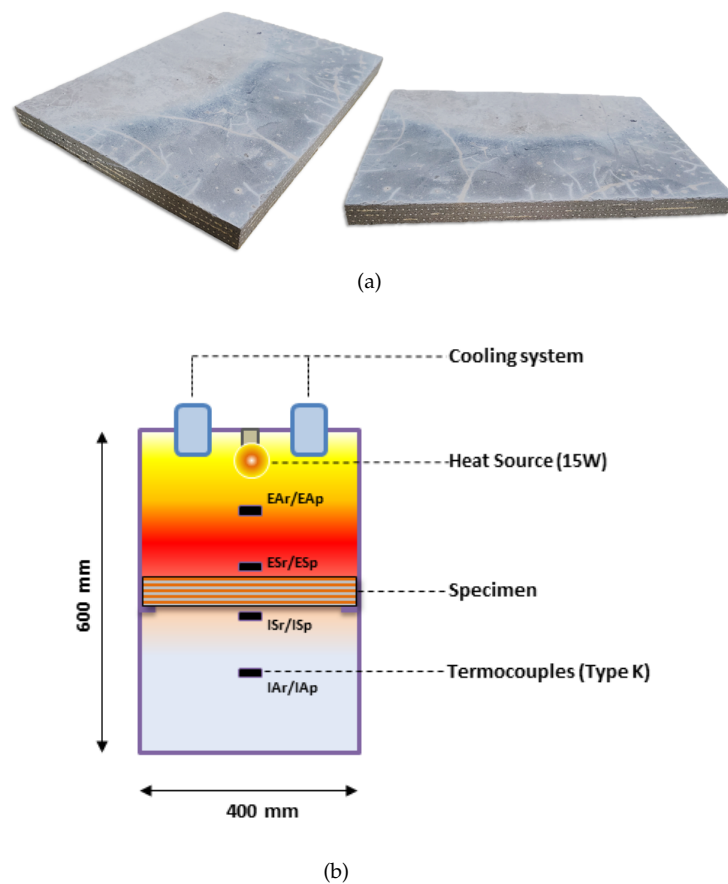


Figure 6. Thermal behavior test: (a) composite specimens and (b) test setup.

The specimens were initially kept at a temperature lower than the PCM's phase change temperature (19 °C). After the system reached a steady state, the heat source was turned on, recording the temperature every 5 s for 90 min. The test time was determined to be sufficient to observe the temperature range in which the PCM's phase change occurs. After this period, the heat source was turned off and the upper chamber was removed, releasing the hot air, and replaced with another chamber, containing glasses with ice, to cool the environment for another 90 min. Specimens from plates produced with jute containing PCM, as well as the reference material not containing PCM, were tested.

3. Results and Discussion

3.1. SEM and Micro-CT Scan Image Analysis

Figure 7 shows the cross-section images of jute yarns in the following conditions: in natura (Figure 7a), XSBR-coated (Figure 7b), and PCM-impregnated and XSBR-coated (Figure 7c). The red arrows in Figures 7b and c indicate the fibers in coated specimens. Jute yarns presented an area ranging between 0.48 and 0.55 mm², while the outer area of treated yarns, i.e., taking the coating into account, presented an average value of 1.38 mm². It is noteworthy to state that the magnitude of the diameters is in accordance with Fidelis et al. [33].

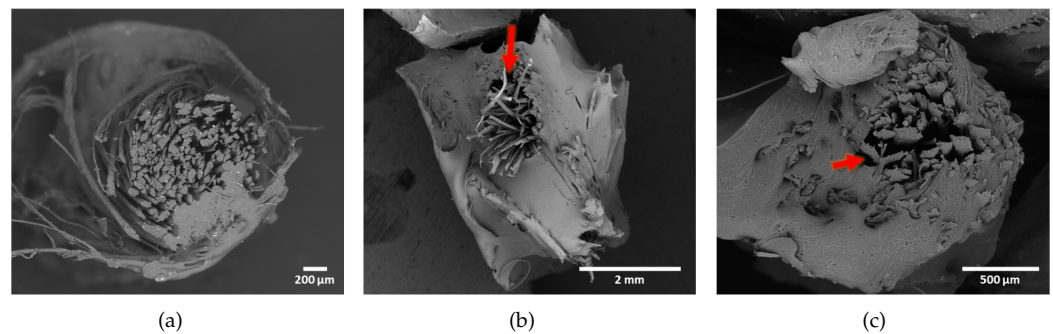


Figure 7. SEM cross-section images of (a) in natura, (b) XSBR-coated, and (c) PCM-impregnated and XSBR-coated jute yarns.

Micro-CT scan images of longitudinal sections of single jute yarns embedded in cementitious matrices are presented in Figure 8a. As observed in Figure 8b, the presence of voids on the interface region between the in natura jute yarn and the matrix occurs due to the poor interaction between them, whereas a more cohesive interface can be observed between XSBR-coated jute yarns and the matrix. Due to the polymer's interaction with both the fiber and matrix, a stronger adhesion was observed. In addition, the polymer completely coated the fibers, preventing the PCM from leaking.

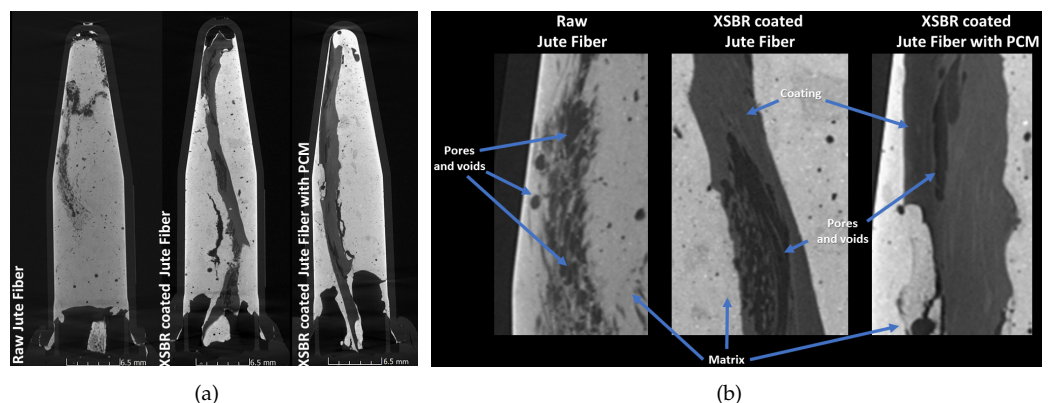


Figure 8. CT scan images of jute yarns embedded in cement: microcentrifuge tubes (a); pores and voids detail (b).

3.2. Jute Fabric PCM Absorption and Polymer Coating Treatment

Two methods were adopted to evaluate the sealing effectiveness of the polymer coating on the jute fabrics: (I) visual, which verified the absence of liquid PCM leakage after the fabric treatment process was considered as fully dried, and (II) direct measurement, which assessed the fabric's thickness and mass after each treatment process. Both methods' results can be observed in Figure 9.

PCM leakage was detected, as some droplets were observed in water after immersion of in natura fabric (Figure 9a). On the other hand, no droplets were observed after the immersion of the polymer-coated fabric (Figure 9b), thus being a good indicator of the polymer's sealing effectiveness.

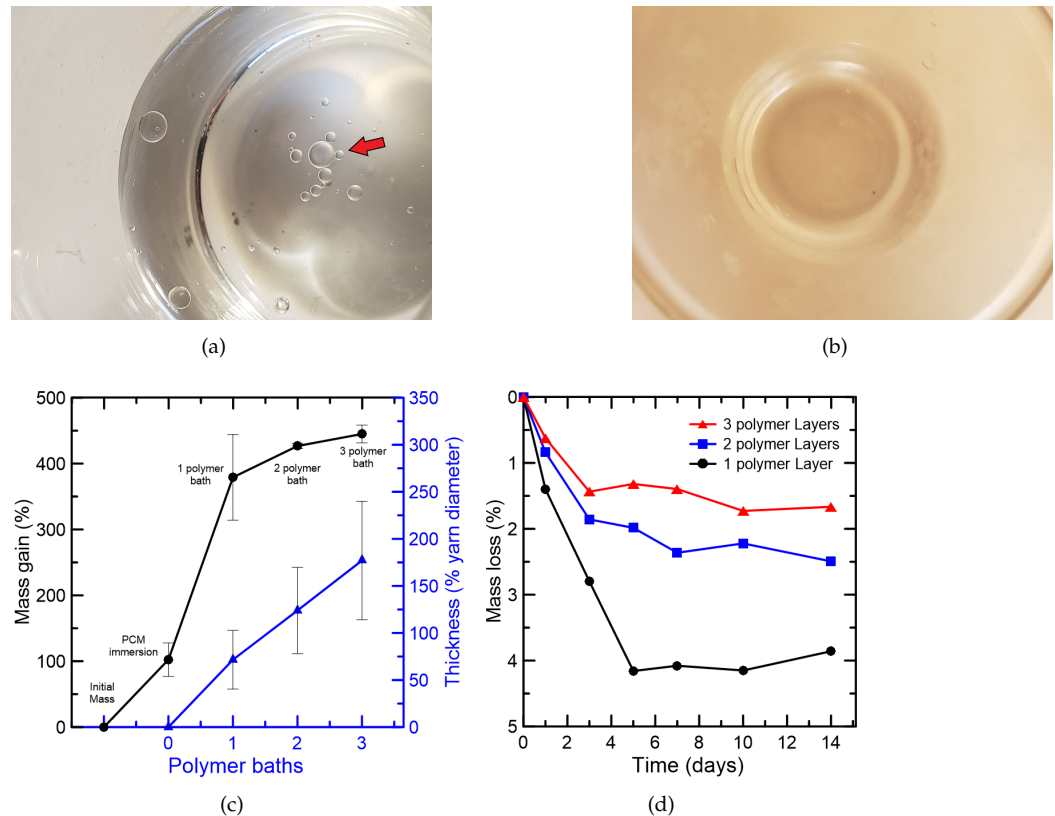


Figure 9. Evaluation of the effectiveness of the polymer coating on the jute fabrics: leakage of (a) in natura jute yarn and (b) XSBR-coated jute yarn, (c) mass and layer thickness during treatment and (d) mass loss after treatment.

Jute fabric specimens' PCM absorption after immersion in liquid PCM was, on average, 102% of the fabric's mass, which can be observed in Figure 9c. After a single bath for 50 min, the layer of XSBR formed measured, on average, 71.6% of the yarn's diameter. The mass gain was the most expressive at this step, at 276.7% of the untreated fabric's mass. This is probably due to the penetration of the polymer between the fibers, filling voids, as observed in the SEM images discussion. After three drying and immersion cycles, the average thickness of the polymer layer reached 177.0% of the yarn's diameter.

Figure 9d shows a 14-day evolution of specimens' mass loss after polymer coating, under laboratory conditions. During the first 3 to 7 days, the mass loss occurs due to the polymer drying, while on the subsequent days, no variations greater than 1% were observed, indicating that no PCM leakage was occurring.

Due to XSBR's low thermal conductivity (0.092 W/m.K) [44], the formation of a thick layer of polymer over the fabric's fibers can hinder heat exchange, which can reduce the action of the PCM. Since no PCM leakage was observed after treatment with a single immersion, no additional immersions were necessary in subsequent treatments.

3.3. Mechanical Characterization of Jute Fibers

Tensile strength tests were performed on yarn and fabric specimens in the following conditions: in natura, XSBR-coated, and PCM-immersed plus XSBR-coated. Due to the mesh opening, the fabric was tested in the transverse and longitudinal directions. Thus,

Figure 10 illustrates typical stress–strain curves of the reinforcements’ behavior, while Table 3 shows the average results of the tensile strength and elastic modulus; similar values for untreated yarns and jute fabric were found by Fidelis et al. [35].

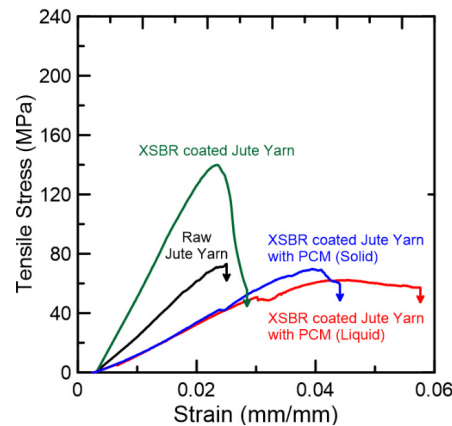


Figure 10. Typical curves from the tensile tests performed on jute yarns.

Table 3. Tensile tests of studied jute yarns and fabric.

Textile Component	Tensile Strength (MPa)	Elastic Modulus (MPa)
Yarn	102 ± 18	4627 ± 739
Fabric (transverse)	60 ± 17	2938 ± 220
Fabric (longitudinal)	62 ± 19	3208 ± 704
Coated yarn	118 ± 14	5508 ± 427
Coated yarn and PCM (liquid)	61 ± 11	2160 ± 290
Coated yarn and PCM (solid)	69 ± 9	2431 ± 258

The presence of PCM in either solid or liquid state caused a reduction of approximately 40–50% of the jute’s tensile strength when compared to yarns without this material. In addition, there was no difference in behavior due to the PCM state during the linear ascending branch. However, after reaching the limit of proportionality, the physical state of the PCM influenced the tensile behavior: jute yarns containing solid PCM kept their original slope up to their rupture, while jute yarns containing liquid PCM presented a plateau, with a strain 40% larger than the former.

3.4. FTIR

The results of the FTIR analysis were obtained through the transmittance as a function of the wave number, as depicted in Figure 11. For the untreated jute fibers, a broad peak between 3700 and 3100 cm^{-1} was observed, relative to the stretching of O–H bonds of carboxylic acid and alcohol functional groups and water absorbed by the material. This peak tends to decrease in intensity or disappear almost completely in treated samples, due to the sealing of the fiber by the polymer, preventing the entry of water and the lower content of the jute in the treated samples.

Between 3100 and 3000 cm^{-1} , low intensity peaks are observed in the treated samples, related to the stretching of =C–H bonds in alkenes, present in the polymers due to the butadiene monomer. A more intense peak in this region at about 3030 cm^{-1} refers to the stretching of C–H bonds in aromatic rings, due to the presence of styrene.

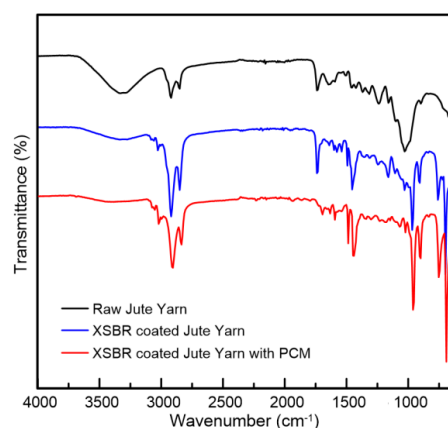


Figure 11. FTIR spectroscopy of untreated and treated jute yarns.

Two peaks can be observed at 2919 and 2850 cm^{-1} , related to the stretching of C–H bonds in alkanes. These bonds are present in the components of the jute fibers (cellulose, hemicellulose, and lignin) and the soluble extractives, but the bonds are also present in the structure of the polymers used, resulting in an increase in the intensity of these peaks in treated samples.

A peak can also be observed at 1735 cm^{-1} , relative to the stretching of C=O bonds. This peak is attributed to the acetyl and uronic ester groups of hemicelluloses or to the ester bond of the carboxylic groups of ferulic and p-coumaric acids of lignin and/or hemicellulose [45]. Furthermore, an increase in the intensity of these peaks is observed in samples treated without PCM, due to the C=O bonds of the carboxyl groups of the polymers. However, in samples containing PCM, these peaks tend to decrease in intensity and to be displaced slightly, possibly due to interactions between the PCM and C=O groups present in XSBR and jute components.

In the region between 1500 and 1400 cm^{-1} , the occurrence of several peaks can be observed. At 1492 cm^{-1} , it is observed that the samples treated with XSBR present a more intense peak, related to the C=C bonds of aromatic rings. At 1450 cm^{-1} , peaks related to the deformation of the C–H bond of alkanes are found in all samples, but are more intense in those treated with polymer, as well as the peaks at 2919 and 2850 cm^{-1} . Finally, it is also possible to identify the occurrence of peaks between 1440 and 1395 cm^{-1} , due to the deformation of the –OH bond in carboxylic acids.

It is observed, in the region between 1300 and 1000 cm^{-1} , the occurrence of some peaks related to the stretching of C–O bonds, characteristic of alcohol, carboxylic acids, ester, or ether functional groups, present in components of the jute fibers or the polymer. In the region between 1220 and 1133 cm^{-1} , there was a decrease in the peak intensity in samples containing PCM, presumably related to interactions between PCM and jute components or XSBR.

Finally, in the region between 1000 and 675 cm^{-1} , peaks related to the deformation of C–H bonds in mono-, di-, or tri-substituted carbons or C=C bonds are observed. The presence of sharp peaks at 966 and 910 cm^{-1} in treated samples is mainly due to the C=C bonds of the butadiene monomer, present in XSBR. Furthermore, the peaks at 760 and 698 cm^{-1} are relative to a mono-substituted benzene derivative, which is identified as the styrene monomer of the XSBR co-polymer.

3.5. Thermogravimetric Analysis

In Figure 12, thermogravimetry (TG) and differential thermogravimetry (DTG) curves are shown. It can be observed that initially, there is a loss of about 3.8% in the mass of raw jute yarn, due to the release of free water present in the material during the first 30 min, in which the temperature was maintained at 40 °C. Three other mass loss events were also observed when analyzing this curve. The initial shoulder, occurring around 250 and 320 °C, corresponds to the degradation of hemicellulose, which occurs between about 150 and

350 °C [46]. Then, a sharp peak is observed with a center at 348 °C, corresponding to the degradation of cellulose. The temperature of this peak is close to that observed by Poletto et al. (2014) for jute fibers, which was 365 °C [47]. Finally, a broad peak of lower intensity can be observed with a center at about 458 °C, which corresponds to the degradation of lignin, and typically occurs at about 250 to 600 °C.

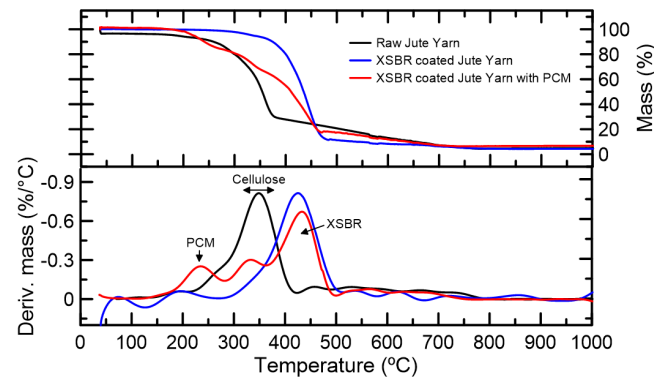


Figure 12. Thermogravimetric analysis curves: TG and DTG of raw and treated jute yarns.

The loss of mass observed in the curve of raw jute due to the free water present in the material was not observed in the other curves, since treatments with polymers provided a sealing of the fibers, reducing water absorption [34], which was confirmed by lower intensities of the bands between 3700 and 3100 cm^{-1} observed in FTIR spectra, as discussed in Section 3.4. Thermal stability, evaluated by the temperature at which a 3% mass loss occurs, was lower for the specimen that contained PCM in relation to treated specimens without PCM. These results can be observed in Table 4. This is due to the fact that the degradation of the PCM, an organic ester, starts at relatively low temperatures [47]. As opposed to the specimens that did not contain PCM, a DTG peak with a center around 234 °C was observed on the curve of the specimens containing PCM, corresponding to its degradation. These results, combined with the results from the previous section, confirm the presence of the PCM in the samples and the effectiveness of the proposed method to incorporate the PCM to jute textiles.

Table 4. Thermal degradation temperature and residue at 1000 °C of untreated and treated jute.

Treatment	Ti (°C) 3% Mass Loss	DTG Main Peak (°C)	Residue at 1000 °C (%)
Raw jute	214	350	4.8
XSBR	313	425	4.3
XSBR + PCM	195	437	6.7

The polymer coating provided greater thermal stability, with an increase of about 100 °C in the temperature at which a 3% mass loss was reached in relation to the untreated jute. It was also observed that the use of treatments shifted the main DTG peak from 367 to about 447–452 °C, a temperature range corresponding to the pyrolysis of XSBR [48], due to its higher content in the samples.

3.6. Mechanical Behavior of Composites

The setup of the flexural test is illustrated in Figure 13, in which the tests were carried out on the following specimens: in natura textile-reinforced composites (T1), XSBR-coated textile-reinforced composites (T2), and PCM-impregnated XSBR-coated textile-reinforced composites (T3). Typical curves of the four-point flexural are shown in Figure 14a. It can be observed that the composites present a linear behavior until the occurrence of the first crack. In all cases, the textile reinforcement presented a large displacement, which indicated a considerable increase in ductility when compared to a cementitious matrix.

The maximum equivalent flexural stress was, on average, 0.96, 1.94, and 1.03 MPa for T1, T2, and T3, respectively.

In Figure 14b, photographs of cracked specimens after bending tests can be observed. T1 composites presented two to three cracks, while T2 presented multiple cracks, indicating a deflection-hardening behavior. Finally, T3 composites presented two cracks, which could be related to the yarn's strength (see Section 3.3). The formation of these cracks can be identified in the curves by the abrupt decrease in flexural stress, followed by its subtle increase.

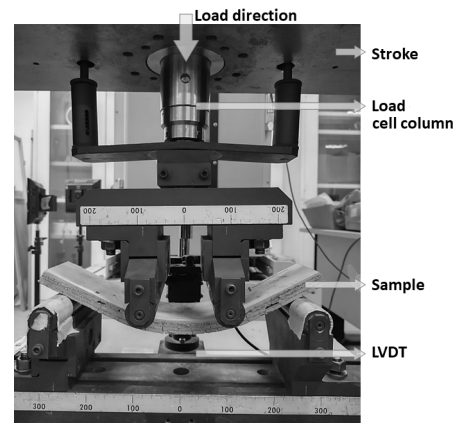


Figure 13. Four-point bending test of XSBR-coated jute fabric composite.

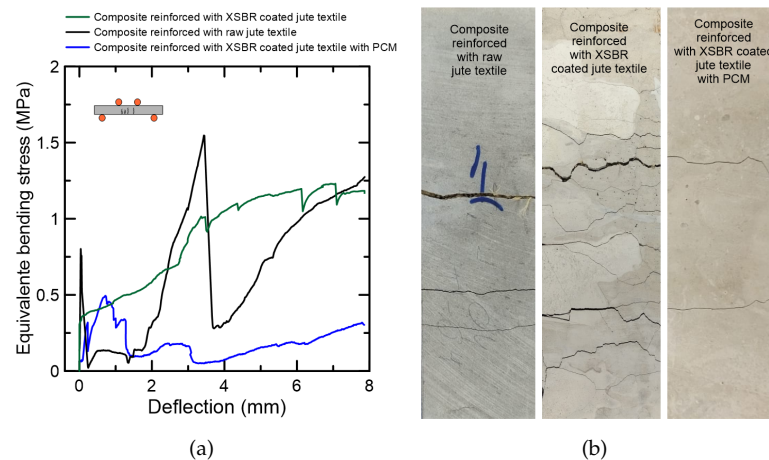


Figure 14. Results of four-point bending tests on the composites: typical curves (a) and cracks formed during tests (b).

The improvement of the mechanical behavior observed in the composites with the XSBR-coated jute fabric can be justified by a better interface between the jute textile and cement-based matrix. As indicated by [33,34], carboxylated styrene–butadiene rubber can chemically interact with calcium-based products produced during cement hydration. This improved interface can also be observed in micro-CT scan images, as discussed in Section 3.1. This enhanced adhesion between the fibers and the matrix helps promote an enhanced stress transfer to adjacent areas after a crack opens, inducing multiple cracks. During all tests, no layer delamination was observed.

3.7. Thermal Behavior of Composites

The results of the tests can be observed in Figure 15. Solid lines represent the reference material (without PCM), while dashed lines represent the material with PCM. Each color represents an average measurement of a pair of thermocouples, simultaneously positioned in different regions, as follows: upper chamber air (simulating the external air), the upper

surface of the specimen, the lower surface of the specimen, and the lower chamber air (simulating the internal air).

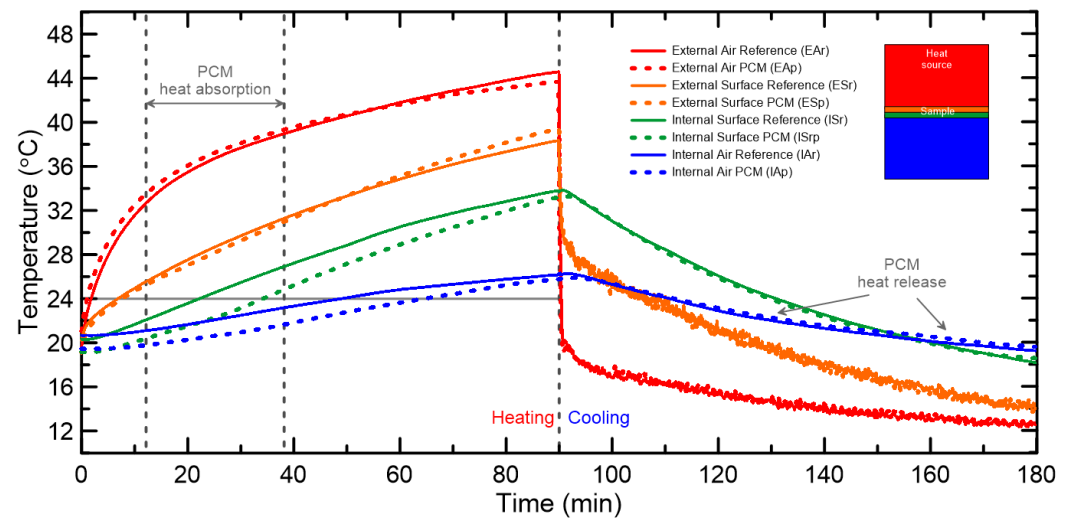


Figure 15. Temperature profiles of studied composites submitted to thermal tests during heating and cooling cycles.

When the PCM-impregnated and XSBR-coated textile-reinforced plate reaches 20–24 °C, the PCM begins to change its phase, absorbing heat and hence delaying the temperature increase. While the lower surface of the reference material takes about 25.5 min to reach 24 °C, the PCM-impregnated plate takes 34% longer, i.e., 34.3 min to reach the same temperature. This result demonstrates the effectiveness of the jute textile reinforcement containing PCM for thermal energy storage.

4. Conclusions

The proposed method of incorporating and containing a PCM within the pores of jute textiles through immersion in liquid PCM, followed by a polymer coating treatment using a styrene–butadiene carboxylated rubber (XSBR), succeeded. PCM absorption by jute textiles was, on average, 102%. FTIR and TG tests, as well as mass stability and water immersion tests, confirmed the non occurrence of PCM leakage. Additionally, SEM images supported the hypothesis that the jute fibers were properly coated by XSBR, while Micro-CT scan images helped understand the behavior of the interface between the jute fibers and cement matrix interface. Mechanical behavior tests indicated, however, a 40–50% reduction in the tensile strength of jute yarns due to the presence of PCM, which consequently affected the textile-reinforced concrete’s mechanical properties.

XSBR-coated textile-reinforced cementitious-laminated composites presented an enhanced mechanical behavior when compared to the reference material, in agreement with previous studies. Oppositely, the composite with PCM presented a similar ductility, when compared to composites without PCM, but with a reduced flexural strength. Composites containing PCM presented a delay in the increase in temperature until 24 °C due to the absorption of heat during the PCM’s phase change. This fact corroborates with the proposal of using jute textile-reinforced composite with thermal energy storage properties to promote the thermal comfort of rooms. Overall, complementary analyses are still required to provide reliable data on the heat transfer properties of the proposed composites, and hence the maintenance and thermal stability of this system, when it comes to real scale buildings.

5. Patents

Ferreira, Saulo R.; Koenders, E.; Mankel, C.; Sam, M.N.; Caggiano, A./Patent name: Energy Storage in Textile Reinforcement Systems NRG-TEX. 2021, Alemanha. Patente: Privilégio de Inovação. Número do registro: 102021126049.3, título: “Energy Storage in

Textile Reinforcement Systems NRG-TEX”, Instituição de registro: German Patent and Trademark Office. Depósito: 07/10/2021.

Author Contributions: Conceptualization, S.R.F. and E.K.; methodology, T.C.G., S.R.F., O.M.O.d.A., M.Y.R.d.-G. and A.C.; validation, T.C.G., O.M.O.d.A., C.M. and M.N.S.; formal analysis, T.C.G., S.R.F., R.G.M.d.A., O.d.F.M.G. and M.N.S.; investigation, T.C.G., R.T.L., M.Y.R.d.-G., E.K., C.M. and M.N.S.; resources, S.R.F., O.d.F.M.G., R.T.L., R.D.T.F., E.K., A.C. and C.M.; data curation, O.M.O.d.A., E.K., A.C. and C.M.; writing—original draft preparation, T.C.G., S.R.F. and R.G.M.d.A.; writing—review and editing, T.C.G., S.R.F., R.G.M.d.A. and O.d.F.M.G.; visualization, T.C.G., S.R.F., R.G.M.d.A., O.d.F.M.G., O.M.O.d.A. and R.T.L.; supervision, S.R.F. and M.Y.R.d.-G.; project administration, R.D.T.F. All authors have read and agreed to the published version of the manuscript.

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Abbreviations

The following abbreviations are used in this manuscript:

PCM	Phase Change Material
GHG	Greenhouse Gases
TES	Thermal Energy Storage
LCA	Life Cycle Assessment
SDGs	Sustainable Development Goals
XSBR	Carboxylated Styrene-Butadiene Rubber
SEM	Scanning Electron Microscope
Micro-CT	Micro-computed tomography
ASTM	American Society for Testing and Materials
FTIR	Fourier-transform infrared spectroscopy
ATR	Attenuated total reflectance
TG	Thermogravimetry
DTG	Differential Thermogravimetry

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4 ARTICLE 2: INFLUENCE OF POLYMER TREATMENT ON THE PROPERTIES OF JUTE TEXTILES CONTAINING PHASE CHANGE MATERIAL

Influence of polymer treatment on the properties of jute textiles containing phase change material

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ABSTRACT

Phase change materials (PCMs) can improve energy efficiency of buildings, reducing energy consumption and improving thermal comfort. PCMs have reportedly been contained by porous materials such as natural fibers, which can be applied as reinforcement for cement matrices in buildings. This work aimed to investigate the effectiveness of different polymer treatments to coat jute textiles containing a PCM, preventing leakage. Three treatments were studied: carboxylated styrene-butadiene rubber (XSBR); XSBR with 5% ZnO addition; carboxylated nitrile rubber (XNBR). In order to evaluate the effect of the treatments on jute's properties, mechanical and chemical characterization of textiles was also performed. Results have shown that XNBR coating provided superior mechanical properties, while also effectively coating fibers with a thinner and lighter coating layer, demonstrating its potential for this application.

1. Introduction

The development of technologies that can improve energy efficiency is a key factor in mitigating the effects of global warming and making our energy consumption more sustainable IEA (2021). Thermal energy storage (TES) is the technology of storing energy by heating or cooling a storage medium so that the stored energy can be used later for heating and cooling applications or for power generation Sarbu and Dorca (2019); Yuan, Zhang, Tao, Cao and He (2014). It has been regarded as a central component in energy-efficient and sustainable energy systems. Phase change materials' (PCMs) property to absorb and release large amounts of heat in a controlled manner can be utilized to improve the thermal performance of various end-use products to which PCMs are applied. Therefore, PCMs are considered highly efficient thermal storage materials Pause (2010).

The use of PCMs in construction appears to be very beneficial; PCM can decrease energy consumption, offset peak loads of cooling energy demand, lessen temperature fluctuations by providing a thermally comfortable environment, and reduce electricity consumption Souayfane, Fardoun and Biwolé (2016). Life cycle assessment studies of phase change materials for construction applications have concluded that, considering the manufacturing, operation and disposal phases, the construction of buildings with PCM incorporation is more environmentally friendly than their reference cases Kylili and Fokaides (2016).

Natural fibers have also been gaining importance in the context of sustainable civil construction. Due to cement's brittleness, reinforcement with fibers can be utilized to increase the impact resistance of this brittle matrix and minimize the effects of shrinkage, reducing matrix cracking Fonseca, da Silva, Silva, Oliveira, Mendes, Hein, Mendes and Tonoli (2016), while environmental, economic and health factors have been favoring the use of natural fibers instead of conventional materials Kang, Myung, Kim and Kim (2013); Akinyemi, Omoniyi and Onuzulike (2020); Ardanuy, Claramunt and Toledo Filho (2015); Da Fonseca, Rocha and Cheriaf (2021); Khorami and Ganjian (2011). However, natural fibers present challenges related to their durability in a cementitious matrix, which leads to the need for additional treatments Tonoli, Santos, Joaquim and Savastano (2010); Silva, Mobasher, Soranakom and Filho (2011); Yan, Kasal and Huang (2016). Polymeric coating treatments reduce water absorption and are effective in the sealing process of natural fibers Ferreira, Silva, Lima and Toledo Filho (2015). Carboxylated styrene-butadiene rubber (XSBR) is pointed out as an effective polymer for these treatments due to its good compatibility with both cement matrices and natural fibers Rocha Ferreira, Rodrigues Sena Neto, de Andrade Silva, Gomes de Souza and Dias Toledo Filho (2020).

In a previous work Guimarães, Gomes, Oliveira de Araújo, Tadeu Lopes, Da-Gloria, Toledo Filho, Koenders, Caggiano, Mankel, Nazari Sam, Mendes de Andrade and Ferreira (2023), the authors have tested the effectiveness of

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a method to store a PCM within the pores of natural fibers. Jute textiles were immersed in liquid PCM and then coated with a carboxylated styrene-butadiene rubber (XSBR), sealing the fibers and preventing PCM leakage. The results showed the ability of producing cementitious composites with thermal energy storage properties through this method. However, due to XSBR's low thermal conductivity and the formation of a thick coating layer around jute fibers, other possibilities for coating should be investigated. This work aimed to evaluate the effects of the addition of zinc oxide to XSBR or the substitution of this polymer for carboxylated nitrile rubber (XNBR) on the mechanical properties of jute yarns containing PCM, as well as evaluating these treatments' suitability for coating jute fibers.

2. Materials and Methods

2.1. Materials

Jute fibers are obtained from the stem of the plant *Corchorus capsularis*, through a process of cutting, retting, shredding, drying, packing and classification Ferreira, Pepe, Martinelli, de Andrade Silva and Toledo Filho (2018). Jute textiles, produced by spinning and weaving, were obtained from Castanhal Companhia Têxtil do Brasil. The textiles' mesh opening is 3 yarns/cm in the transverse direction and 3.5 yarns/cm in the longitudinal direction.

The PCM used was CrodaTherm 15-LQ-(GD), with a phase change temperature of about 15 °C. This bio-based organic PCM is a commercial product obtained from Croda do Brasil Ltda. The polymers used for coating were carboxylated styrene-butadiene rubber (XSBR NTL-218, with medium styrene content) and carboxylated nitrile rubber (XNBR NTL-525, with medium-low acrylonitrile content), manufactured by NITRIFLEX.

2.2. Methods

2.2.1. Immersion of jute textiles in liquid PCM and polymer coating treatment

The absorption of PCM by jute textiles was performed by immersion in liquid PCM for 2 min, in order to obtain a maximum degree of saturation of 80%, as proposed by Guimarães et al. (2023). Polymer coating treatment was then carried out based on the procedure described by Fidelis, de Andrade Silva and Toledo Filho (2014), by means of total immersion in a polymer bath at room temperature for 50 min, followed by drying in open air for 72 hours on a plastic film. The system mass was measured after each step, and the thickness of the polymer layer formed around the fibers was measured with the aid of a microscope and ImageJ imaging software. In order to verify the non-occurrence of liquid PCM leakage, 3x3 cm coated fabric specimens were left in open air and mass variation was measured over fourteen days. Specimens were also placed in water for three days, and the presence or absence of leaked PCM in water was verified.

2.2.2. Tensile tests

Tensile tests were performed on an AROTEC WDW-20E universal testing machine, in accordance with ASTM C1557 Anonim (2015). A 5 kN load cell was used and the displacement rate was 1.25 mm/min. 250 mm long yarn specimens were tested at a controlled temperature of 18 °C. To help secure the yarns to the equipment's grips, the ends were wrapped with 3M duct tape. In addition, a laser was used to ensure the alignment of the specimens. This setup can be observed in Figure 1.

The tensile strength of the yarns was determined by dividing the maximum force measured in these tests by the average cross-sectional area (0.48 mm²), as measured by Fidelis et al. (2014).

XSBR and XNBR coated jute yarns containing PCM were tested, in addition to raw jute yarns. In order to verify the effect of the PCM's physical state on the mechanical properties of jute fibers, tests were performed both on specimens at room temperature (in which the PCM was in its liquid state) and on specimens cooled at 5°C until the moment of testing (in which the PCM was in its solid state).

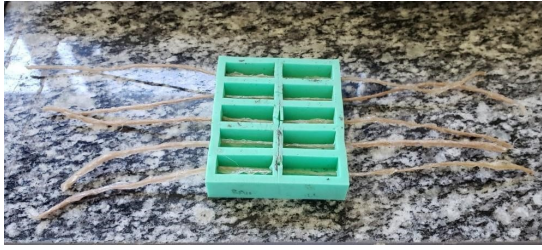
2.2.3. Pullout tests

Pullout tests were carried out in order to verify the effect of polymer treatments on the adhesion between jute fibers and a cement matrix. Specimens were produced in a silicone mold, as illustrated in Figure 2 with an internal embedment length of 25 mm, pointed out by Ferreira et al. (2015) as an effective embedment length to obtain representative pullout load curves. Jute yarns were inserted in the mold and stretched, and a cement paste with a 1:0.3 (cement:water) ratio was applied, filling the mold. Specimens were demolded after 24 h and stored in a wet chamber. Tests were then performed after 7 days from the molding, in accordance with Ferreira, Lima, Silva and Toledo Filho (2012).

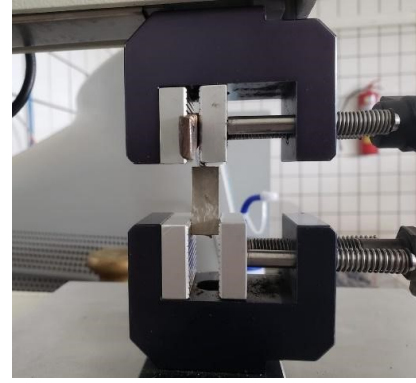
Three polymer coating treatments were tested: XSBR, XSBR + 5% (mass) ZnO and XNBR. Tests were performed on a TA.XT plus texturometer equipment at room temperature. A load cell of 50 N was used and a displacement rate



Figure 1: Direct tensile test on jute yarn.



(a)



(b)

Figure 2: Mold for pullout specimens production (a) and pullout test (b).

of 0.6 mm/min was adopted. As for direct tensile tests, specimens were tested at room temperature, in which the PCM was in its liquid state, or cooled until the moment of testing, meaning the PCM would still be in its solid state. Nominal adhesion tension can be calculated by (1):

$$\tau = \frac{P_{ad}}{2\pi rl} \quad (1)$$

In which P_{ad} is the maximum pullout load, l is the embedment length and r is the yarn's radius, calculated from the average cross section area.

2.2.4. Chemical characterization of jute fibers

Chemical characterization of jute fibers was performed through Fourier-transform infrared spectroscopy (FTIR) and thermogravimetry analysis (TGA). The following jute samples were used: *in natura*, coated with XSBR, XSBR + 5%ZnO or XNBR polymers, and impregnated with PCM + coated with polymer.

For the FTIR analysis, the attenuated total reflectance (ATR) sampling method was used. The spectral range analyzed was from 4000 to 675 cm^{-1} , with 24 scans and a resolution of 4 cm^{-1} . For the thermogravimetry analysis, the samples were initially submitted to a constant temperature of 40 °C for 30 minutes, to remove the free water in the material. Then, they were heated from 40 to 1000 °C at a constant heating rate of 10 °C/min, under an N_2 atmosphere. Both tests were based on the method proposed by Ferreira *et al.* Rocha Ferreira *et al.* (2020).

2.2.5. Micro-computed tomography (Micro-CT) scans

Single jute yarns were placed inside 2 mL microcentrifuge tubes, which were then filled with a 1:0.3 (water/cement) cement paste. Micro-CT scan images of the microcentrifuge tubes were obtained in order to analyze the effect of the polymer coating treatment on the interface of the system. Images were acquired in a V/TOMEX/M equipment. The following parameters were specified: 70 kV voltage, an energy of 280 μ A, 5 frames, and an Al filter with a thickness of 0.5 mm. The geometry had a magnification of 9.37, pixel size of 21 μ m and a total of 1200 projections.

2.2.6. Scanning Electron Microscopy

Untreated and treated jute yarns were investigated with a FEI Quanta 400 scanning electron microscope (SEM). Specimens were sputter coated with a thin layer of about 20 nm of silver using a BAL-TEC SCD 005 sputter coater. This process is necessary to provide conductivity to specimens of non-conductive materials, such as natural fibers, improving the quality of the SEM images and analysis. It is worth mentioning that silver was used, instead of the more commonly used gold, because it costs much less, and it can provide good quality SEM images in low magnification Vieira, de Souza, Enríquez-León, Aragão, Gomes, Leite and Rezende (2021). The microscope was operated under 20 kV of acceleration voltage, in high vacuum mode. The images were acquired using a back-scattered electrons detector.

3. Results

3.1. Polymer coating treatments

XSBR polymer treatment for the sealing of jute fibers containing PCM has been evaluated in a previous study Guimarães et al. (2023). The main criteria for evaluating the effectiveness of that treatment was the absence of PCM leakage on coated jute fabric specimens. This can be achieved if the fibers are completely coated by the polymer, which seals them, preventing liquid PCM leakage. However, it is also important that the coating layer formed does not hinder heat exchange to the point that the PCM's phase change can not fully take place. Therefore, thick layers of polymer around the fibers should be avoided.

Specimen mass gain after immersion in liquid PCM and after up to three immersions in 50 min in polymer suspension, as well as polymer layer thickness after each immersion, can be observed in Figure 3. Polymer layer thickness was measured with ImageJ image software as a percentage of the yarn's diameter in each image. Images were collected in each intersection of three 3 x 3 cm fabric specimens. It can be observed that XSBR forms a thicker and heavier coating layer than XNBR. After a single immersion for 50 min, XSBR and XNBR layer thicknesses were, respectively, 72 and 49% of the yarns' diameters. Mass gain after the first immersion was the most expressive one for both polymers, with specimens gaining, respectively, 276 and 213% of the untreated fabric's mass, due to the penetration of polymer between the fibers, filling voids, which does not happen after successive immersions. After a full 3 cycles of immersion and drying, XSBR and XNBR coating layer thicknesses reached 177 and 108%, respectively, of the yarns' diameters.

As it had been previously observed for XSBR coating Guimarães et al. (2023), after being left in open air for 14 days and after immersion for three days in water, no PCM leakage has been observed for XNBR-coated jute fabric specimens (after a single immersion). This result, combined with the formation of a thinner coating layer compared to XSBR, indicates that XNBR coating can also effectively be used as a method to contain PCM and can potentially lead to better heat exchange between the PCM and the environment.

3.2. Tensile tests

Influence of polymer treatment and PCM's physical state on fibers' mechanical properties were investigated through direct tensile tests. Figure 4 shows typical stress-strain curves of direct tensile tests of raw and treated jute yarns containing PCM in its solid or liquid state. Maximum tensile stress and elastic modulus results can be observed in Table 1.

In relation to raw jute yarns, treated yarns presented lower maximum tensile stress. This result is in accordance with Fidelis et al. (2014), who observed a reduction from 104.38 MPa for raw jute yarns to 87.59 MPa for XSBR-coated yarns. Furthermore, Guimarães et al. (2023) observed that the presence of PCM has a negative effect on the mechanical properties of jute yarns.

When comparing the two polymers utilized for the treatments, a statistically significant difference has been observed. XNBR coating provided higher tensile strength and elastic modulus than XSBR coating. Compared to XSBR, tensile strength increased from 61–69 MPa (with PCM in liquid/solid state) to about 77 MPa (with PCM in either state);

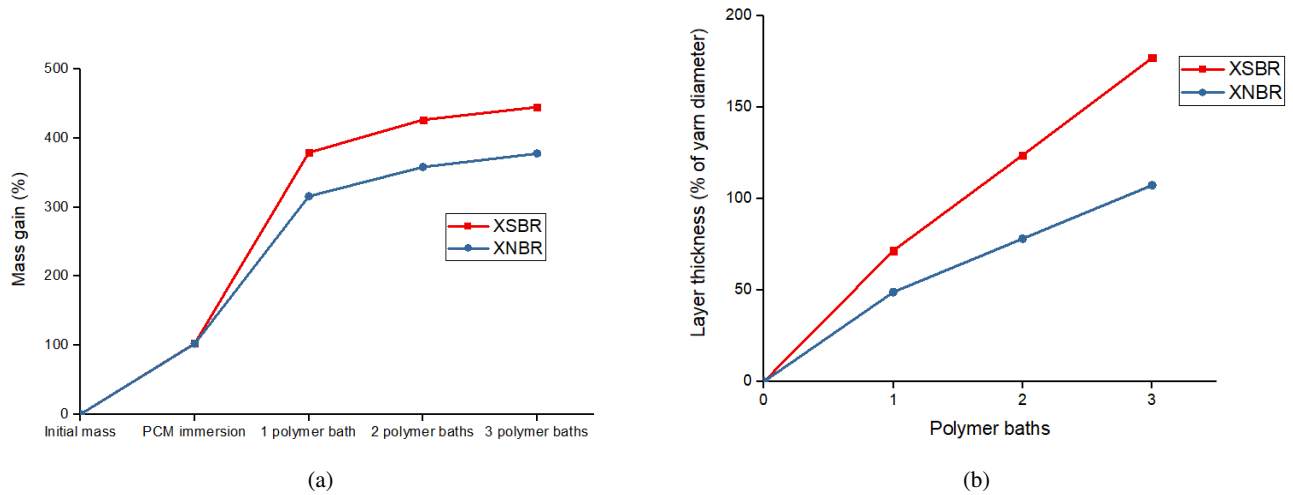


Figure 3: Treatment of jute textiles: mass gain (a); polymer layer thickness (b)

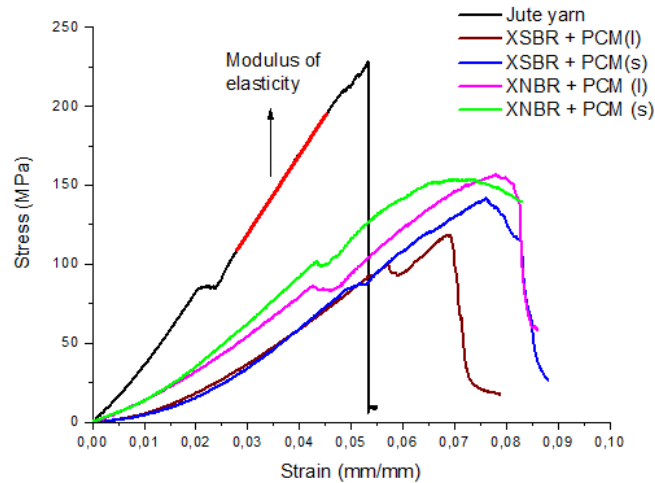


Figure 4: Typical direct tensile tests stress-strain curves.

Table 1

Direct tensile tests results.

Treatment	PCM state	$\sigma_{\max}$ (MPa)	E (GPa)
Raw jute	-	102±18	4.6±0.7
XSBR + PCM	Liquid	61±11	2.1±0.3
XSBR + PCM	Solid	69±9	2.4±0.2
XNBR + PCM	Liquid	77±13	2.6±0.3
XNBR + PCM	Solid	77±12	3.0±0.2

elastic modulus increased from 2.1–2.4 GPa to 2.6–3.0 GPa (liquid/solid PCM). Better mechanical behavior of XNBR coated yarns may be related to a good chemical interaction between hydroxyl groups present in the fibers' cellulose and XNBR's acrylonitrile groups Leão, Jesus, Bertuoli, Zattera, Maia, del Menezzi, Amico and da Luz (2020).

PCM physical state had little effect on the fiber's tensile strength, with no statistical differences being observed, indicating that phase change does not compromise the fibers' mechanical properties. However, an increase in stiffness has been observed when the PCM was in solid state for both treatments, with the elastic modulus increasing from 2.1 to 2.4 GPa for XSBR and from 2.6 to 3.0 for XNBR.

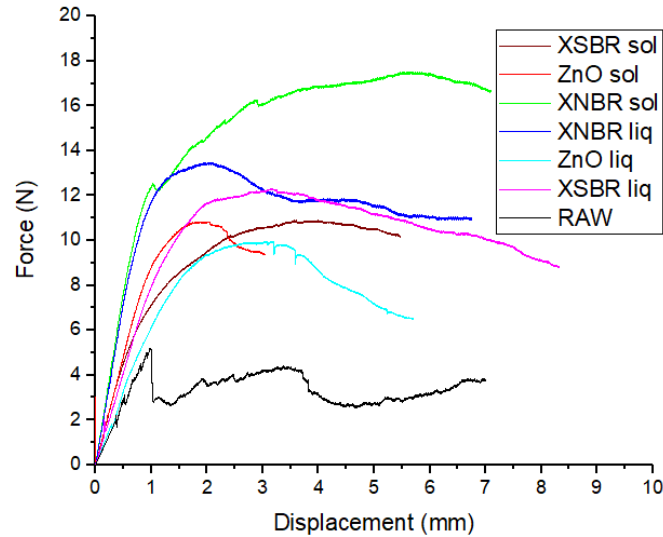


Figure 5: Typical pullout curves.

3.3. Pullout tests

Typical pullout curves of raw, XSBR coated, XSBR + 5% ZnO coated and XNBR coated jute yarns can be observed in Figure 5. Adhesion strength results can be observed in Table 2.

The influence of treatments on adhesion to the cement matrix can be observed. All treatments used resulted in higher adhesion stresses compared to the untreated material. This is due to the greater anchorage provided by the polymeric coating (ROCHA FERREIRA et al., 2020). Stronger adhesion of treated yarns was also observed by (FIDELIS, 2014). However, the author observed higher values of adhesion stress, 0.47 MPa for untreated cords and 0.64 MPa for cords treated with XSBR.

Table 2
Pullout tests results.

Treatment	PCM state	Fad (N)	Tad (MPa)
Raw jute	-	5.6±1.1	0.091±0.017
XSBR + PCM	Liquid	10.4±3.1	0.170±0.51
XSBR + PCM	Solid	9.6±3.6	0.156±0.059
XSBR/ZnO + PCM	Liquid	9.9±1.7	0.162±0.028
XSBR/ZnO + PCM	Solid	8.9±3.2	0.145±0.053
XNBR + PCM	Liquid	12.6±1.2	0.206±0.020
XNBR + PCM	Solid	15.7±3.7	0.256±0.060

Regarding the addition of 5 % zinc oxide to XSBR, no significant differences in adhesion were observed. Finally, it was observed that the adhesion provided by the treatment with XNBR was superior to the others, with an adhesion strength of 0.206 and 0.256 MPa with PCM in the liquid and solid states, respectively, in agreement with the results obtained in the direct tensile tests, possibly due to the interaction between the acrylonitrile groups of the polymer and the hydroxyl groups of the cellulose present in the fiber (LEÃO et al., 2020).

The physical state of PCM had little effect on adhesion to the cement, since the results of samples containing PCM in solid state were statistically equal to those of samples containing PCM in liquid state, meaning that adhesion was not compromised by changes in the physical state of the PCM. These results were also in agreement with results from direct tensile tests.

3.4. Infrared spectroscopy (FTIR)

Spectra of analysed jute samples can be observed in Figure 6. Raw jute fibers samples present broad bands between 3700 and 3100 cm^{-1} , relative to stretching of -OH bonds due to the water present in the fibers. In treated samples, these

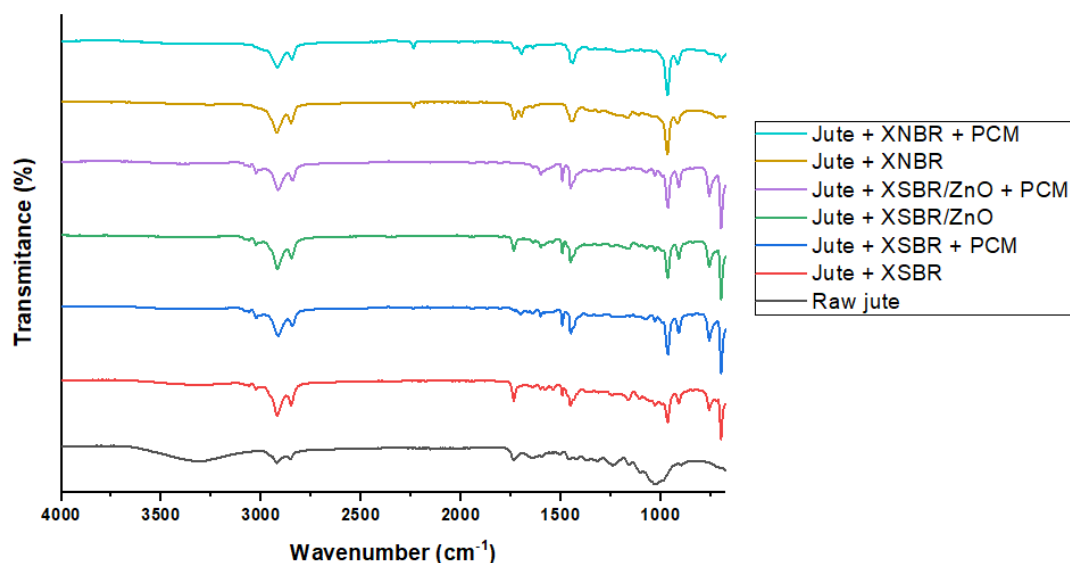


Figure 6: Infrared spectra of jute textile samples.

bands present lower intensity due to the sealing of the fibers provided by polymer treatments Rocha Ferreira et al. (2020). The main components of jute fibers (cellulose, hemicellulose and lignin) produce other characteristic peaks, such as the two peaks at 2919 and 2850 cm^{-1} , relative to C–H bonds stretching; a peak at 1735 cm^{-1} due to C=O bonds stretching Ferreira, Silva, Lima and Toledo Filho (2017); as well as other characteristic peaks in the fingerprint region.

XSBR treated jute samples present weak peaks between 3100 and 3000 cm^{-1} , relative to the stretching of –C=H bonds present in butadiene, as well as stronger peaks at 2919 and 2850 cm^{-1} compared to raw jute, due to the presence of C–H bonds in both monomers that comprise this copolymer. Other characteristic peaks can be observed at 1492 cm^{-1} , relative to C=C bonds in aromatic rings, and sharp peaks at 760 and 698 cm^{-1} , indicating a monosubstituted benzene derivative, due to styrene; as well as two sharp peaks at 966 and 910 cm^{-1} , relative to C=C bonds present in butadiene. Between 1600 and 1500 cm^{-1} , the appearance of low-intensity peaks can be observed in samples treated with XSBR/ZnO 5%, indicating the presence of zinc oxide.

XNBR treated jute samples present similar spectra to XSBR treated ones, with the main difference being the absence of peaks that are characteristic to styrene. At wave number 2236 cm^{-1} weak peaks are observed in samples containing XNBR. These peaks are related to the stretching of the C≡N bonds, characteristic of nitrile groups.

Although the PCM did not produce easily identifiable peaks due to the number of components in the samples, lower intensity peaks were observed at 1735 cm^{-1} and between 1220 and 1133 cm^{-1} in samples containing PCM. This suggests an interaction between the PCM and –OH or C–O groups present in the polymers or jute components' structure.

3.5. Thermogravimetry (TG)

TG curves can be observed in Figure 7. Samples were kept at 40°C for 30 minutes, in order to remove the free water present in the materials, before being subject to a constant heating of 10°C/min. In untreated jute fiber samples, this procedure led to a mass loss of about 3.8%, while in treated samples mass loss was negligible, due to the sealing of the fibers provided by the polymer coating, reducing water absorption Rocha Ferreira et al. (2020).

Thermal stability of samples was evaluated through the temperature at which 3% mass loss occurs, as proposed by Poletto, Ornaghi Júnior and Zattera (2014). These results can be observed in Table 3. Polymer coating treatments have increased fibers' thermal stability, increasing 3% mass loss temperature by 80–108°C and shifting the main DTG main peak from 367 (cellulose degradation Poletto et al. (2014)) to 440–452°C (pyrolysis of the polymers Yang, Yu, Zhong, Liu, Yao, Zhang, Zou and Du (2021)). Addition of 5% ZnO to XSBR slightly increased thermal stability and residue at 1000°C. XNBR, on the other hand, provided slightly lower thermal stability than XSBR. Finally, addition of PCM to treated fibers has decreased thermal stability, lowering 3% mass loss temperature by 107–118°C. Residue at 1000°C was increased by PCM addition, however.

The PCM used is an organic ester, which typically begin thermal degradation at relatively low temperatures Poletto et al. (2014), leading to the lower thermal stability observed. DTG results further confirm this result, as shown in Figure

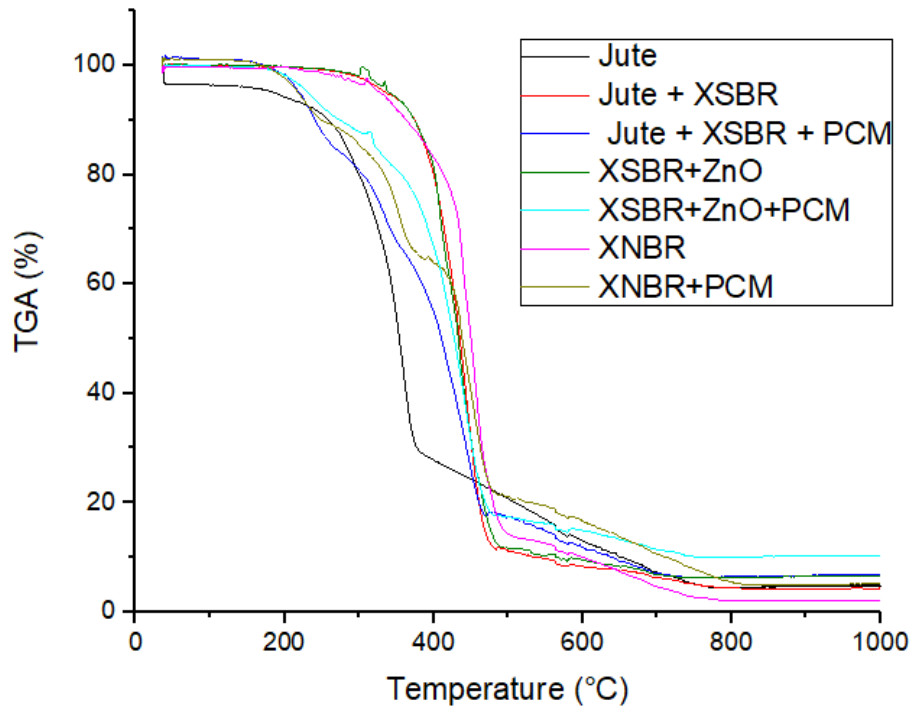


Figure 7: TG curves of jute textile samples.

8. Samples containing PCM presented an additional peak with centers at 220-240°C, corresponding to the degradation of the PCM. Two other large peaks can be observed in DTG curves, correspondent to the degradation of cellulose and the polymers. DTG results confirm the presence of the PCM in all samples, atesting to the method's ability to contain the PCM, in accordance with Guimarães et al. (2023).

Table 3
TG tests analysis.

Treatment	T (°C) 3% mass loss	DTG Peak (°C)	Residue at 1000°C (%)
Raw jute	214	367	4.8
XSBR	313	452	4.3
XSBR + PCM	195	447	6.7
XSBR/ZnO	322	440	6.5
XSBR/ZnO + PCM	215	442	10.3
XNBR	294	441	2.1
XNBR + PCM	181	436	5.1

3.6. SEM and micro-CT scan images

SEM images of XSBR/ZnO 5% and XNBR-coated jute yarns' cross section can be observed in Figure 9. Guimarães et al. (2023) investigated cross section images of untreated and XSBR-coated jute yarns, observing that XSBR polymer treatment had effectively completely coated jute fibers, reducing chances of liquid PCM leakage. The same can be verified in these images, indicating that these polymer treatments can also be used to prevent liquid PCM leakage and corroborating the results discussed in section 3.1.

Figure 10 shows the micro-CT scan images obtained from microcentrifuge tubes with jute yarns embedded in a cement matrix. It can be observed that, while the interface between untreated jute yarns and the matrix presents numerous voids, the interface between treated jute yarns and the cement matrix has a more continuous aspect, with significantly fewer voids. This is due to the chemical interaction between the polymers and cement, and confirms the results from the pullout tests showing a stronger adhesion, as discussed in section 3.3.

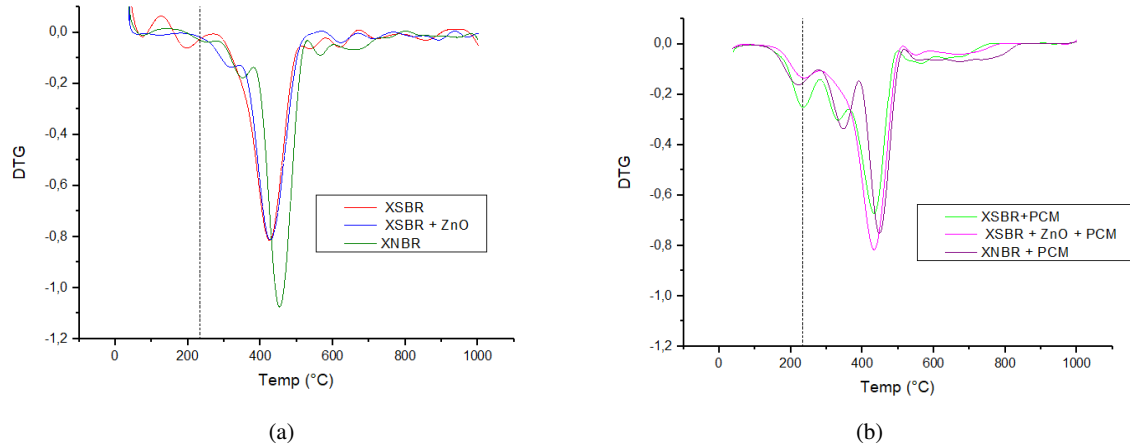


Figure 8: DTG of treated jute without (a) and with PCM (b).

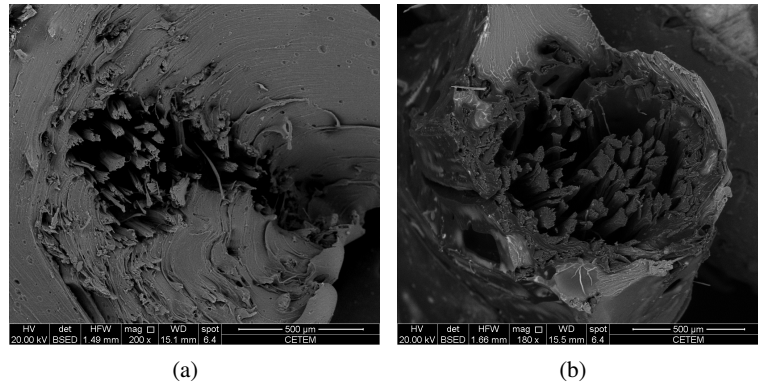


Figure 9: SEM images of XSBR/ZnO (a) and XNBR (b) coated jute yarns.

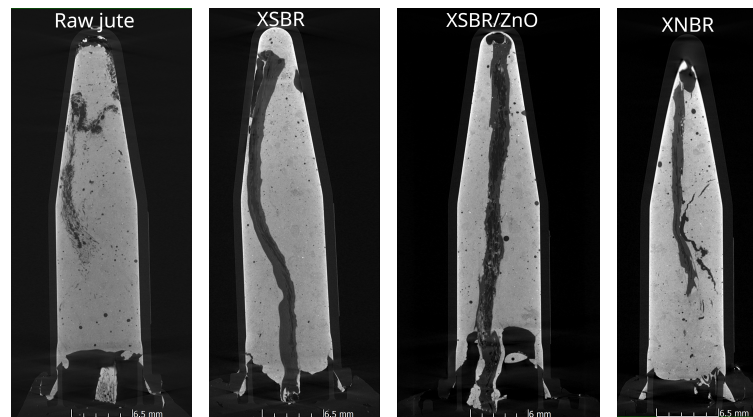


Figure 10: Micro-CT scan images of embedded jute yarns.

4. Conclusions

Further investigation of the method of containing a PCM within jute fibers' pores through an immersion method followed by a polymer coating treatment has corroborated results from a previous study, indicating that this method is effective to produce jute textiles with thermal energy storage properties. Evaluation of effect of the PCM's physical

state on the mechanical properties of jute yarns have shown no significant losses when the PCM is in its liquid state compared to the solid one.

XSBR treatment had already been pointed out as a suitable polymer for the coating of jute fibers in a previous work. While the addition of 5% zinc oxide did not significantly alter the properties of the fibers, its use could still be justified by a better workability obtained. Tests with XNBR have shown that it can provide higher tensile strength to jute yarns and stronger adhesion between yarns and a cement matrix. Furthermore, XNBR treatment was able to successfully completely coat jute fibers, preventing liquid PCM leakage, while forming a thinner and lighter coating layer. These results indicate that XNBR can potentially ensure better heat exchange between the PCM and its surroundings, leading to improved thermal behavior. However, further studies about the thermal properties of XNBR-coated jute textile composites are required to confirm this.

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