



Lignocellulosic Materials for Production of Cement Composites: Valorization of the Alkali Treated Soybean Pod and Eucalyptus Wood Particles to Obtain Higher Value-Added Products

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Abstract

Brazil, as a predominantly agricultural country is the second largest world producer of soybean. Consequently, these plantations are responsible for high production of agricultural wastes. For reusing the large volume of one type of this material generated, the soybean pods, one type of waste generated by this crop, could be applied as a raw material for production of higher value-added products. Good examples of value-added product are those applied in the field of construction. The building construction is an intensive activity and it requires a great variety of materials. A great ideal in this field would be the obtainment of a material that relates good mechanical strength and reuse of wastes. In this context, the aim of this study was to produce cement composites with soybean pods associated with eucalyptus wood, both treated with alkaline solution and evaluate their physical properties and mechanical strength. The wood and the wastes particles were submitted to alkaline treatment in NaOH solution (5%) during 48 h in order to removing chemical compounds that could harm the cement solidification. For the composites production, the soybean pods addition was performed in proportions of 0, 25, 50, 75 and 100% and mixed with the complementary percentage of *Eucalyptus grandis* wood particles until achieve the amount of 100% of lignocellulosic material which constitutes the composite. The lignocellulosic materials were evaluated by basic density and chemical composition. The physical properties of water absorption (WA) (2 and 24 h) and thickness swelling (TS) (2 and 24 h) and the mechanical properties modulus of elasticity (MOE), modulus of rupture (MOR), compression strength and janka hardness of the cement composites were evaluated. Scanning electron microscopy (SEM) images showed the occurrence of cavities in the composites produced with higher proportions of soybean pods. All the properties were analyzed by ANOVA and in case of significance, the regression equation was adjusted to correlate the property with the percentage of soybean waste added. It was noted an increase of WA 24 h in the samples with high insertion of soybean pods. The TS was not affected by the different compositions of the composites. The mechanical properties decreased with the increase of soybean pod in the cement composites. However, the cement composites could be produced with up to 48% of soybean pods associated to eucalyptus wood and applied, for example, as partition walls or thermal and acoustic insulation walls.

Keywords Agricultural wastes · Cement composites · Wood particles · Mechanical strength

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Statement of Novelty

It is known that the use of wastes for high value-added materials production is the subject of several researches worldwide. This research focuses on the agricultural wastes produced in Brazilian lands which have potential for being destined for other purposes. Unfortunately, our reality is still poor when it comes to the use of waste for purposes of value products generation. Therefore, studies in this field are extremely important for sectors such as construction and engineering materials, for example. Producing cement

composites from the agricultural wastes associated with wood allows, in addition to the reuse of the waste, the possibility of maintaining suitable properties of the material, since the cement-wood panels are very well applied for construction. The possibility of reuse of soybean pods in construction applications can mean a great achievement for this sector.

Introduction

The high diversity of agricultural species cultivated in Brazilian soil combined with the high productivity are the main responsible for generate a large volume of agricultural wastes. Among the lignocellulosic wastes widely available in Brazil, those originated from soybean cultivation deserve attention, since soybeans are one of the main agricultural products of the world, with their main production destined for the extraction of oil and vegetal protein [1]. Brazil is the second main soybean producer of the world, with area of 33 million ha planted and production of 96 million t harvested in 2016 [2]. Soybean is also responsible for the generation of great amounts of wastes. It can be produced between 1.2 and 1.5 t of soybean straw per ton of grain produced and 31.78% of the straw is constituted of pod [3]. As secondary products, soybean pods have not necessary attention, for example, for production of materials with higher value-added [4]. Presently, these wastes are either burnt or land filled, which can cause several problems such as the air pollution, emission of greenhouse gases and occupation of useful land [5]. In the last decades, there has been increasing research interests towards the utilization of lignocellulosic materials and by-products as composite materials, in particular, for thermal insulation, false ceilings, fiberboards and packaging materials [6]. Another interesting way to use these wastes is in the production of cement composites. They are special types of structural materials which have in their composition: fibers, mineral cementing agent, water and additives, with the use of any conventional adhesive [7]. The main application of these composites is for production of prefabricated house walls, counter tops, floors, cladding, partition walls, thermal and acoustic insulation walls and house linings, etc [8]. Some lignocellulosic materials have already been studied in order to improve the properties of cement composites, in addition to allow the use of waste materials as pine fibers [9], eucalyptus fibers [10], malva [11], curauá fiber [12], wheat straw [13], rice straw [14], coir [15], cork granules [6] and hazelnut shell [16].

The inclusion of lignocellulosic materials for cement composites production is a great challenge due to some chemical properties of the lignocellulosic material which may cause a negative effect during the cement solidification. In most of cases the extractives are the main responsible

for this inhibition. They are resins and other chemicals that may migrate to the surface of the particle during the drying of the wood resulting in formation of a hydrophobic layer, which reduces the hydrogen bonds between the lignocellulosic material and cement, resulting in a reduction of the bonding strength between both [17].

In order to become feasible the use of certain species with high content of extractives, some treatment methods are performed in order to remove some polar components present in the raw material. A significant number of physical or chemical pretreatment approaches has been tested. These techniques include acid, alkali, ionic liquid treatment, organosolv, steam treatment, wet oxidation and microwave irradiation [18] as well as enzymatic treatments [19–21]. One of the most used methods is the treatment of the lignocellulosic material with alkaline solution, which aims to “clean” the fiber surface of waxes and greases, besides partially removing the hemicelluloses and lignin [22]. The literature reports alkaline treatments in vegetal fibers, including agricultural wastes such as kenaf, proving that the treatment was efficient for the removal of surface impurities [23]. In this work, the alkaline treatment was performed with a solution of 5% NaOH, which is a low concentration when compared to the mercerization, for example, which uses contents of 12% NaOH or more. Regarding the cementitious matrix, a frequent problem found by the researchers is the fiber mineralization. Mineralization of the cellulose fibers occurs when the solution diffusion penetrates the interior of the fiber. This penetration into the fiber voids (both cell wall and lumen) induces the embrittlement of the cellulose fibers. Secondary, occurs degradation mechanisms of the fiber into cement matrix [24, 25]. This effect is hardly studied when producing fiber-cement composites, in which the mineralization effect can occur even more intensely, as each individual fiber comes into contact with the cement matrix. In this context, the aim of this study was to evaluate the physical properties and mechanical strength of cement composites (cement matrix) produced with addition of several proportions of soybean pods mixed to eucalyptus wood, including the composites produced only with eucalyptus wood and only with soybean wastes, both lignocellulosic materials treated with alkaline solution. The goal of the present work is to report soybean pod composites to overcome all the challenges and mechanical behavior, posteriorly demonstrating the utility of soybean pod particles for application in construction field.

Materials and Methods

Obtainment and Treatment of the Material

The soybean waste (Fig. 1a) was obtained from a commercial planting located in Jataí-GO. The soybean pods were

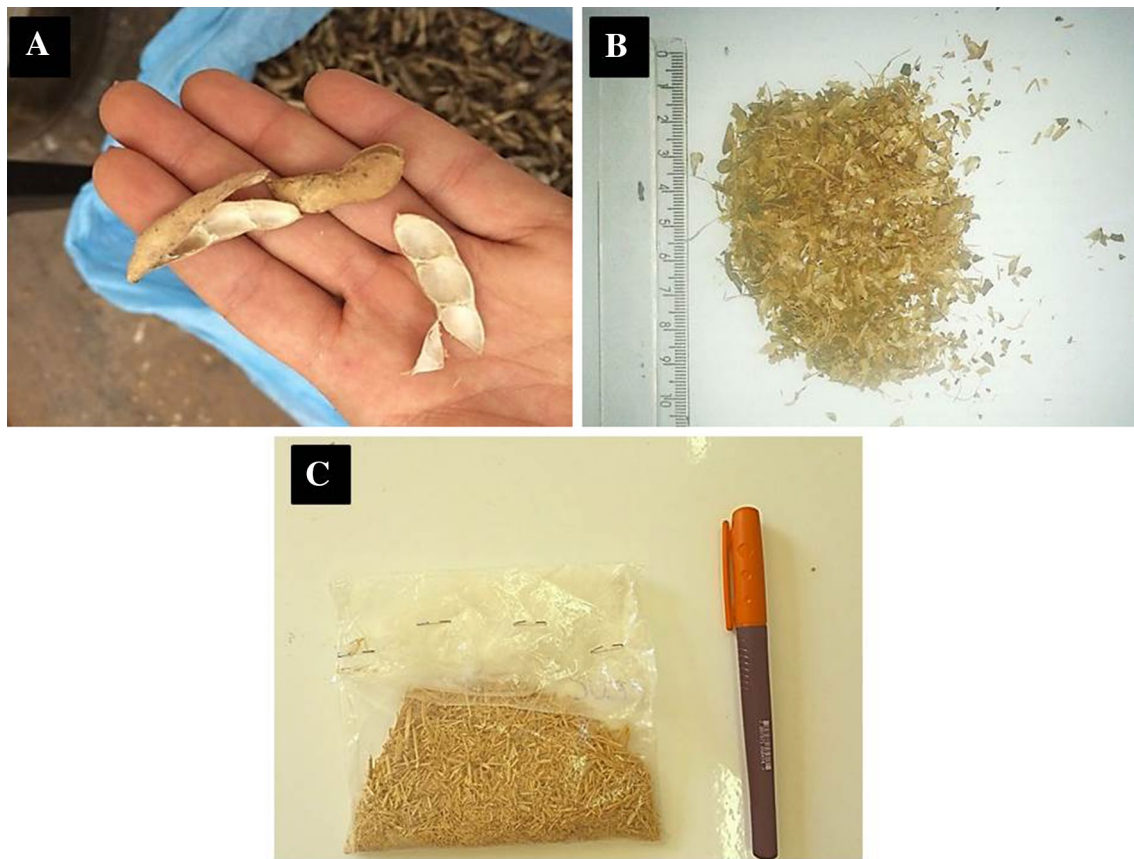


Fig. 1 Soybean wastes used for production of cement composites; **a** whole soybean pods; **b** soybeans pods with the correct particle size for the composites production; **c** eucalyptus wood particles

separated from the rest of the waste and processed in the Laboratory of Forest Products of Federal University of Goiás. The *Eucalyptus grandis* obtained from the experimental planting of Federal University of Lavras was forwarded to the Experimental Unit of Wood Panels (UEPAM), where it was milled to generate particles. The particles were selected through a sieve and the fraction retained between 20 (0.841 mm) and 40 (0.420 mm) mesh (Fig. 1b) was used to produce the composites. The particles passed through sieves with the purpose of remove fine particles, as well as excessively large particles resulting from the milling process and which could impair the bonding. The particles of the soybean pod were submitted to treatment with alkaline solution for removing the compounds that could harm the cement solidification. The alkaline treatment followed the procedures described in Ferraz [26] and Asgher [27], in which the particles were immersed in NaOH solution (5%) for a period of 48 h. The eucalyptus particles (Fig. 1c) were submitted to the same treatment. Posteriorly, the particles were washed with water in abundance to remove the excess of reagent. The washing was performed until obtain neutral pH. The residual water resulted from the alkaline treatment of the particles was sent to the Waste Treatment Division of

the Federal University of Lavras in intention to find a correct destination. In this sector, the reagent wastes used can be distilled and, when possible, recovered and redistributed to the university laboratories.

Characterization of the Lignocellulosic Material

The basic density of the lignocellulosic materials was carried out according to the procedures described in NBR 11941 [28]. For the chemical analysis, the material was milled to facilitate the action of the chemical reagents. The particles were selected in sieves of 40 (0.420 mm) and 60 (0.250 mm) mesh, been the fraction which was retained in the sieve of 60 mesh used for the analysis. The quantification of the components was based on different standards: total extractives—NBR 14853 [29], insoluble lignin—NBR 7989 [30], minerals content—NBR 13999 [31].

Production of the Cement Composites

The compounds: cement, water, lignocellulosic material particles and additive were mixed in a concrete mixer. Posteriorly, the mixture of compounds was homogeneously

distributed on aluminum plates so that the composite obtained uniform thickness. The aluminum plates were greased with diesel oil Toyama LUB DG15 (Texsa do Brazil, Umuarama, Paraná) to facilitate post-press demolding. Five proportions of soybean pod particles were determined in relation to eucalyptus wood. The total mass of each composite was obtained by multiplying the volume by the pre-established density. The calcium chloride additive was added in percentage of 6% (based on cement mass) to accelerate the cement solidification. The mass of each composite compound was determined according to the proportions of (lignocellulosic material)/(cement) and (water)/(cement) was exposed in Table 1.

The mixture was pressed with specific pressure of 2.45 MPa at room temperature for a period of 24 h. The resulting composites were conditioned in an environment at 20 ± 2 °C and relative humidity $65 \pm 3\%$ for 28 days. Posteriorly, the samples were obtained and tested for evaluation of their strength and water absorption properties.

Determination of the Cement Composites Properties

The procedures to determinate the WA and TS outlined in the ASTM D1037 [32] were followed. Test samples of $150 \times 150 \times 15$ mm were soaked in water for 2 and 24 h. At the end of immersion period, the specimens were removed from the water and then measured (thickness) and weighed. The Janka hardness and the compression strength of the composites were determined by ASTM D1037 [32] procedures. Janka hardness was determined by using a Janka ball with 11.28 mm in diameter with a uniform

rate of motion applied by a universal testing machine of 5 mm/min. The properties MOR and MOE were evaluated by DIN 52363 [33] standard (distance between supports: 200 mm; loading: 5.0 mm/min).

Morphological Analysis

The samples were visualized by scanning electron microscopy (SEM) in order to show the structure and possible defects in the composites produced. The micrographs were obtained by a scanning electron microscope model JMS 6510 (JEOL®) with a voltage of 10 kV.

Experimental Design

The experiment was carried out in a completely randomized design with five treatments (combinations of lignocellulosic materials). Three composites were produced for each treatment (see Table 1). The data generated by the physical and mechanical properties of the cement composites were evaluated through variance analysis (ANOVA) ($p \leq 0.05$) and regression analysis. The ANOVA was performed for all properties. In case of statistical differences between the treatments, the regression equation was adjusted. The equations were adjusted aiming to correlate the maximum amount of soybean waste that could be mixed to the eucalyptus particles so that the minimum value required by the standard was achieved. If no statistical differences were observed between the treatments, the regression adjustment was not performed.

Table 1 Parameters of the cement composites production

Composite density	1.2 g/cm ³
Dimensions	20 × 20 × 1.5 cm
Total volume	600 cm ³
Lignocellulosic material/cement	1/2.5
Water/cement	0.25
Additive CaCl ₂	6% of cement mass
Mass of each composite component	
Cement	436 g
Lignocellulosic material	175 g (soybean pod + eucalyptus wood)
Water	109 g
Total composite mass	720 g
Additive CaCl ₂	26 g
Proportions of the lignocellulosic material mixture	
Eucalyptus 100%	175 g eucalyptus wood
Eucalyptus 75%—soybean pod 25%	131 g eucalyptus wood + 44 g soybean pod
Eucalyptus 50%—soybean pod 50%	87.5 g eucalyptus wood + 87.5 g soybean pod
Eucalyptus 25%—soybean pod 75%	44 g eucalyptus wood + 131 g soybean pod
Soybean pod 100%	175 g soybean pod

Results and Discussion

Characteristics of the Lignocellulosic Material

The mean value obtained for basic density of the eucalyptus wood in this study was 0.554 g/cm³ (Table 2). This value is between 0.500 and 0.720 g/cm³, which indicates medium density wood according to the classification proposed by Institute of Technological Research [34]. The density of the lignocellulosic material influences the compaction ratio of the composites. As lower is the density of the material, greater the compression and strength of the composite tend to be [35]. Low or medium density lignocellulosic materials could also be perfectly suited for generation of other engineering materials used in domestic furniture, such as medium density particleboards (MDP).

The extractives content of the lignocellulosic materials evaluated were similar. These substances are the most limiting factor for production of cement composites because their action may results in a lower resistance of the composites [14]. Lignocellulosic materials such as husks and pods usually show high levels of extractives, which act as a barrier avoiding the satisfactory linkage between the particles and cement, being necessary the performance of treatment for the achievement of better properties of the composites. In addition, Chakraborty et al. [36] states that dissolved extractive may form a protective layer on

the partially hydrated cement grains. This layer forms a temporary barrier on the cement grains for further hydration. According to Miller and Moslemi [17], materials with high content of components such as tannins are not desirable for production of cement composites without a suitable pretreatment. The amount of lignin found for the eucalyptus wood was slightly higher when compared to the amount found for the soybean pods. Lignin is a three-dimensional polymer which serves as a chemical adhesive within and between fibres [37–39]. Lignin and pectin are two compounds which work as bonding agents [39, 40]. The soybean pods showed higher ashes content in comparison to eucalyptus wood. The values of ashes content found for the soybean pods were similar to those found by Silva et al. [41] for the same material, which was 8.90%. The use of siliceous materials such as ashes for production of these type of composites is common [42–44], therefore high ashes content in the biomass will not impair the composite properties.

Properties of the Cement Composites

No significant statistical difference for WA after 2 h (Fig. 2a) was observed. On the other hand, when evaluated the WA 24 h, the values were statistically different (Fig. 2b). This was assumed to be due to the insufficient time for the water to reach the totality of the lignocellulosic material when evaluated after 2 h. After the period of 24 h the water had a

Table 2 Chemical composition of the lignocellulosic materials

Lignocellulosic material	Basic density (g/cm ³)	Total extractives (%)	Insoluble lignin (%)	Ashes (%)
Soybean pod	0.203 ± 0.003*	8.27 ± 0.632	23.00 ± 0.637	8.77 ± 0.049
Eucalyptus wood	0.554 ± 0.011	8.14 ± 1.257	29.03 ± 2.084	0.26 ± 0.030

*Standard deviation

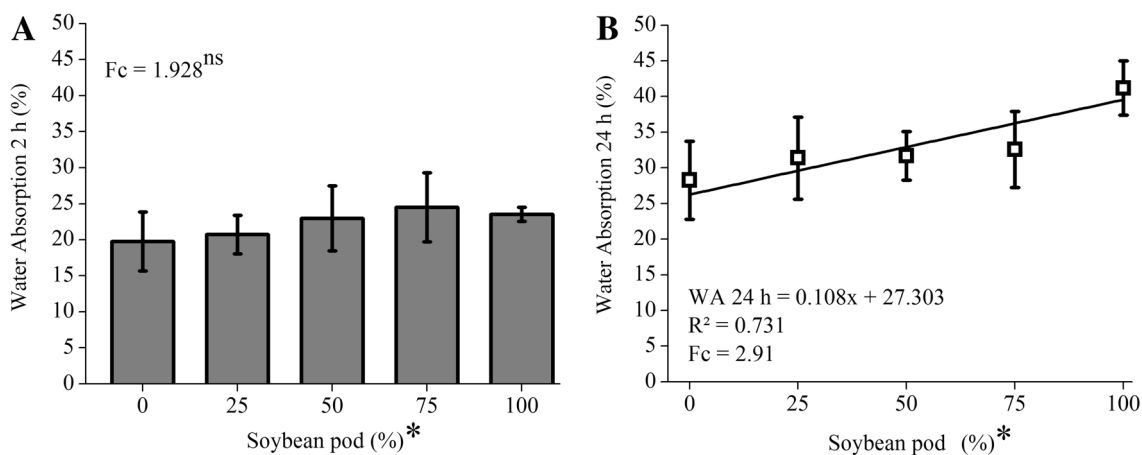


Fig. 2 WA of the composites evaluated; **a** after 2 h of immersion in water; **b** after 24 h of immersion in water; ns = F_c value not statistically significant; *the x-axis shows the percentage of soybean wastes associated to eucalyptus particles

sufficient time to penetrate in the cementitious matrix and reach the particles of wood and soybean pods. The characteristics of composites with natural fibres immersed in water are influenced by the nature of the fibre and matrix materials, by the relative humidity and manufacturing technique, which determines factors such as porosity and volume fraction of fibres [45].

Cement composites present low WA due to the absence of hydrophilic groups in the cement. The hydrophilic zones of the lignocellulosic components are the main responsible by the fraction of water absorbed, resulting in a porous and weak interfacial zone between the lignocellulosic material and the matrix [46, 47]. The highest values for WA of the composites with higher percentages of soybean pods may be due to the lower basic density of the wastes (see Table 2), which results in a higher volume of particles and consequently more hydrophilic zones for connection to water molecules in relation to eucalyptus wood. The literature reports a mean value of 10.7% for WA in molded cement composites produced with Portland cement with no addition of lignocellulosic material [48], which clearly indicates that the matrix shows high resistance to water. Fernandez and Taja-on [14] produced cement bonded rice straw boards using different cement:rice-straw ratios. The WA of these boards was rather high. The variation found (24–42%) was close to that found in this present study.

Another important physical property to be evaluated in cement composites is the TS. In opposition to WA, no differences were observed between the compositions of lignocellulosic particles (Fig. 3). Although it shows relatively low values, the knowledge of this property allows give a better destination to the final product according to the standardized values.

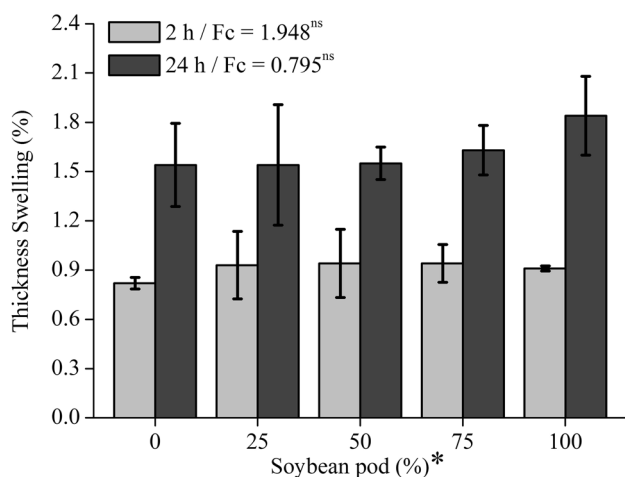


Fig. 3 TS after 2 and 24 h immersion in water of the composites produced; ns=Fc value not statistically significant. *The x-axis shows the percentage of soybean wastes associated to eucalyptus particles

The low values found for TS of the cement composites may be due to the protection layer that the cement provides around the particles, decreasing its expansion [49]. Compared with other types of panels, such as conventional particleboard, cement composites, on average, show extremely low results of dimensional variation. Latorraca and Iwakiri [35] found values for TS similar to those found in this study for cement composites produced with the species *Eucalyptus urophylla* and *Eucalyptus citriodora*, with mean values of 1.64 and 1.73%, respectively. In a similar study, Nasser et al. [50] found values close to 4.73% for TS when evaluating cement composites with density of 1.200 g/cm³. The Bison Wood-Cement Board [51] process establishes a value of 1.8% for TS after 24 h of immersion. Therefore only the composites produced with 100% of soybean pods did not meet the requirements of the standard. In comparison to the resin bonded composites, the cement composites have as advantage a better resistance to water, fire and biodegradation [5]. The apparent density of the cement composites may cause influences on their properties. There was no statistical difference between the density of the composites evaluated (Fig. 4).

The statistical equality for apparent density of the different treatments allows more accurate comparison between the composites produced. Composites with higher densities tend to have higher mechanical properties. The MOE and MOR values showed a decreasing trend according to the higher percentages of soybean pod associated to eucalyptus wood (Fig. 5a, b).

This fact may have occurred due to the lower basic density of soybean pods when compared to eucalyptus wood (see Table 2). Lower basic density results in a larger volume of particles of the waste, which leads to greater quantity of

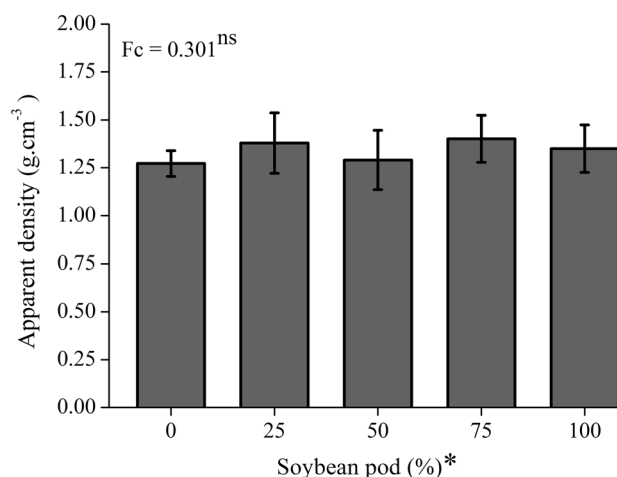


Fig. 4 Apparent density of the cement composites; ns=Fc value not statistically significant. *The x-axis shows the percentage of soybean wastes associated to eucalyptus particles

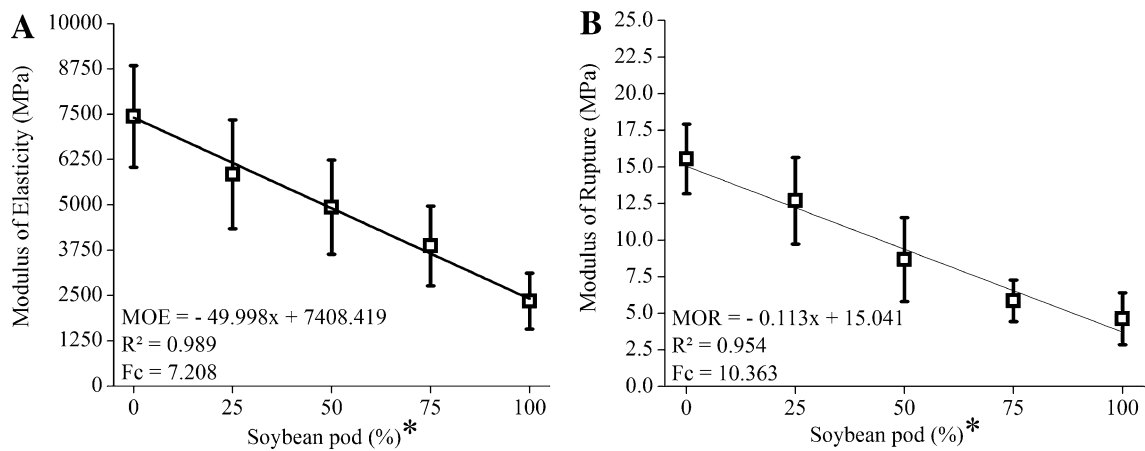


Fig. 5 **a** MOE of the cementitious composites; **b** MOR of the cementitious composites. *The x-axis shows the percentage of soybean wastes associated to eucalyptus particles

particles for being covered by the cement matrix. As a result, the matrix-fiber interface may be impaired due to the appearance of cracks and empty spaces. The composites produced with 50 and 100% of soybean pods showed a greater number of regions with empty spaces in the interface, which may have impaired the mechanical properties (Fig. 6). In addition, this higher quantity of cavities may explain, besides the basic density previously discussed, the higher values of WA for the composites with higher amounts of soybean pods (see Fig. 2). For each 1% of soybean pod added, there was a decrease of 44.26 MPa in MOE value. The Bison Wood-Cement Board [51] process establishes 3000 MPa as a minimum for MOE considering the commercial cement composite. Replacing this value (dependent variable) in the equation generated for MOE, it is noted that the percentage up to 77.62% of soybean pods could be used in combination to eucalyptus wood (22.38%) for production of cement composites that reach the value established by the standard.

For the property MOR, it was observed by the adjusted equation that for each 1% of soybean pods added to the composites, there was a decrease of 0.11 MPa in their strength. Iwakiri et al. [52] found value of 6.59 MPa when studying cement composites produced with paricá wood particles treated with NaOH solution. This value is close to the obtained for the composite produced with 25% of soybean pods. According to the process Bison Wood-Cement Board [51], the minimum value for MOR when considering cement composites is 8.92 MPa. Replacing this value in the regression generated for MOR, it is noted that a percentage up to 47.63% of soybean pods could be combined to eucalyptus wood so that the composites produced achieve the minimum mentioned by the standard. Another factor that may have caused the decrease of mechanical properties is the possible inhibition of the cement solidification caused by the high extractive content of the lignocellulosic material,

causing a lower bond between the particles and the matrix. It is well known that alkali treatment breaks the linkages (benzyl ether, glycosidic ether, benzyl ester, acetal groups) present in lignin–cellulose complexes of the lignocellulosic materials [53]. However, even after the alkaline treatment, extractive waste may remain in the material, impairing the bonding properties. Scatolino et al. [54] performing the alkaline treatment on natural fibers of the Amazonian paricá, verified a reduction of 50% in the extractives content of the material. That is, a good part of the extractives still remained in the fiber. Regarding the compression strength of cement composites, an evident decrease of the values can be noted (Fig. 7).

Since strength is the principal property required by engineer, the pore structure is the most important factor affecting the strength of porous material. A good understanding of the pore structure (see Fig. 6) of the composites will be indispensable to help understand the mechanical performance [12]. The literature reports mean value of 37.3 MPa for compression strength in molded cement composites produced with Portland cement with no addition of lignocellulosic material [48]. The decrease in compression strength may occur due to extractives and sugars released by the lignocellulosic particles after the contact with water, since these components can impair the matrix hardening. In addition, the presence of inclusions in the still plastic matrix creates the “wall effect”, which increases the porosity in the transition zone allowing the calcium accumulation in this zone, becoming more fragile and susceptible to cracks [55]. The values obtained were lower in comparison to that found by Pomarico [56] when evaluating cement composites with addition of eucalyptus wood, which found values ranging from 9.3 to 12.0 MPa. As for the previous mechanical properties evaluated, higher percentages of soybean pods caused also a negative effect in janka hardness (Fig. 8).

Fig. 6 Typical scanning electron microscopy images of the cement composites produced; **a** and **b** micrograph of the composites composed by 100% of eucalyptus wood; **c** and **d** micrograph of the composites composed by 50% of eucalyptus wood and 50% of soybean pods; **e** and **f** micrograph of the composites composed by 100% of soybean pods

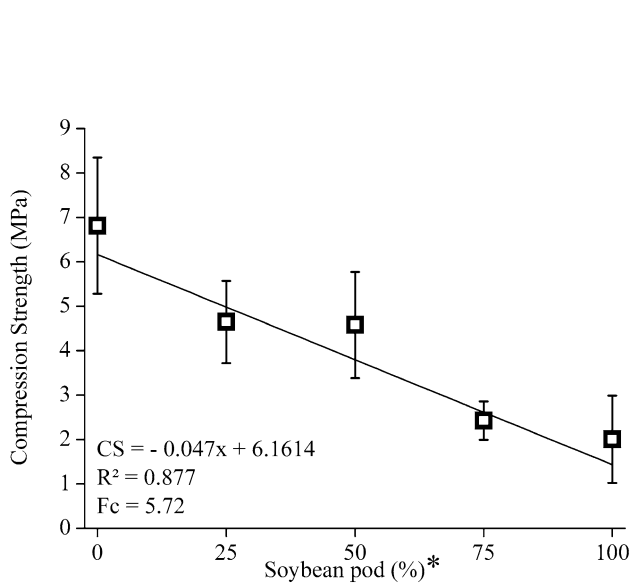
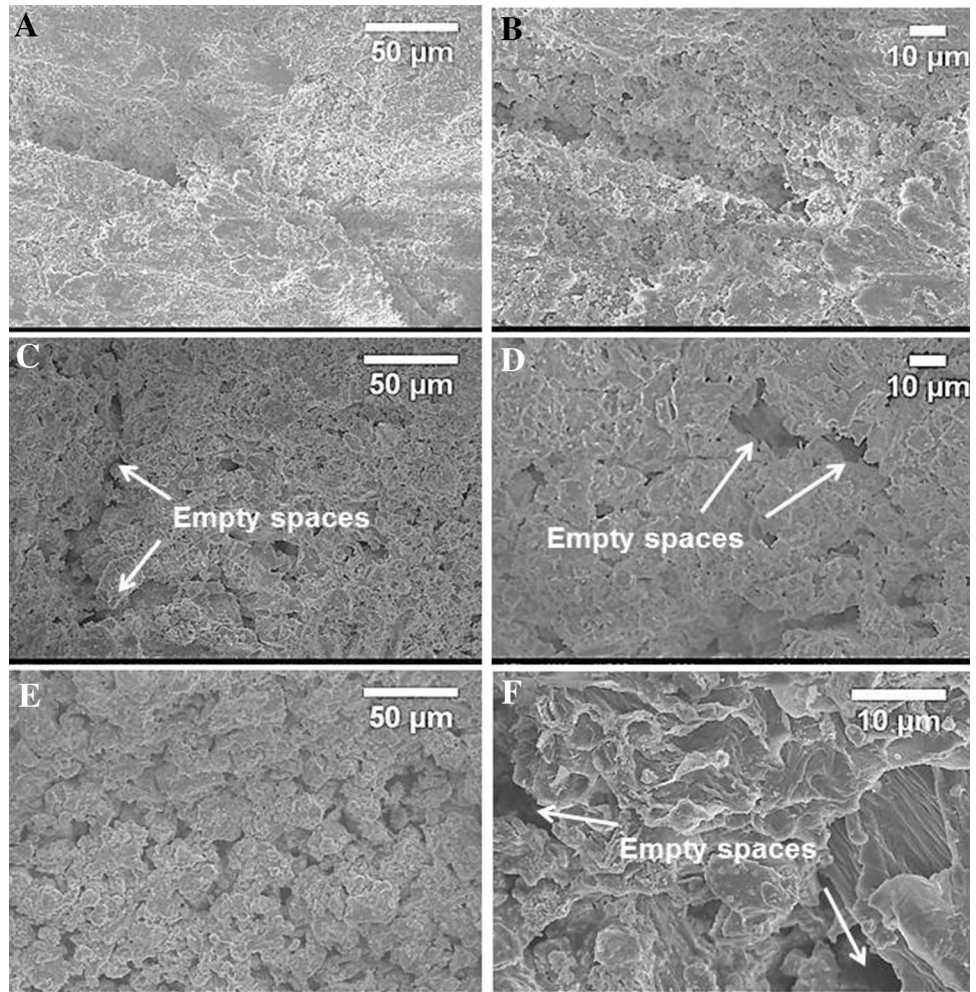


Fig. 7 Compression strength of cement composites. *The x-axis shows the percentage of soybean wastes associated to eucalyptus particles

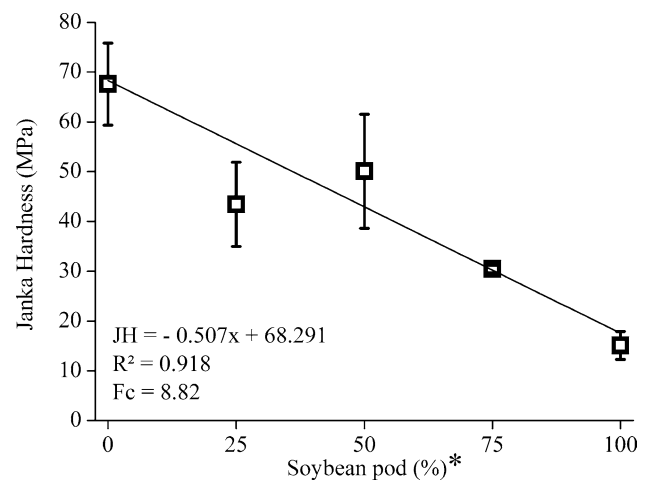


Fig. 8 Janka hardness of the cement composites. *The x-axis shows the percentage of soybean wastes associated to eucalyptus particles

It is noted that there was a reduction of 0.51 MPa for each 1% of soybean pods added. The lowest Janka hardness values were observed in the composites produced with 100% of soybean pods with mean values close to 15 MPa. Since the janka hardness evaluates the composites strength to the sphere penetration, when it comes to lignocellulosic materials, the cellular wall is an important factor. The low basic density of the lignocellulosic material directly influences in cell wall thickness and therefore in strength and stiffness of the material [57]. In addition, Filho et al. [58] found a positive correlation to basic density of the specie and the janka hardness. The resulting value of hardness is well known in the wood engineering literature for its effects on the performance such as flooring [59]. The low density of the soybean pods (see Table 2) may have caused this low strength in comparison to composites with eucalyptus. The formation of a more porous structure in the treatments which it was carried out greater addition of soybean pods may also have contributed to lower results for this property.

Final Evaluation

The final evaluation of the results obtained by the tests performed on the cement composites was done based on the regression equations generated for MOE and MOR. The process Bison Wood-Cement Board [51] requires the minimum of 8.92 MPa for MOR. Replacing this value into the adjusted equation $MOR = -0.113x + 15.041$ (Fig. 5b), where x is the minimum percentage of soybean waste that can be combined with eucalyptus for production of the composite. The result obtained for x was 47.63% of soybean pods so that the composites produced achieve the established value mentioned by the standard. The same process was done for the evaluation of the MOE. The adjusted equation was $MOE = -49.998x + 7408.419$ (Fig. 5a). The minimum established for MOE by the Bison Wood-Cement Board [51] process is 3000 MPa, considering commercial cement composites. The value obtained to the dependent variable in the equation was 77.62%. Therefore the value of up to 48% of soybean waste added to the composites reach the minimum required for MOE and MOR. The Bison Wood-Cement Board [51] standard does not present exact minimum value for compression strength, however Gong et al. [60] reports that the compression strength values required for material to be used as pavements ranged from 20 to 25 MPa. Therefore, this panel would not be applicable for this purpose, being restricted to the use in partition walls or thermal and acoustic insulation walls.

Conclusion

Cement composites were produced aiming the use of soybean pods wastes in combination to eucalyptus wood. According to the obtained results, the characteristics of the material chemical composition cause great influence on its properties. The insertion of different proportions of soybean pods significantly affected the physical property of WA, but did not affect the swelling of the composites produced. The addition of soybean pod resulted in a decrease in the mechanical properties of the cement composites produced. The microscopy images showed the existence of regions with empty spaces in the composites produced with the higher insertion of soybean waste. The adjusted linear regression equations indicate that the maximum percentage of soybean pod insertion in cement composites to meet the established for mechanical properties by the Bison Wood-Cement Board process is approximately 48%. Further studies are required in order to evaluate the thermal and acoustic insulation characteristics of these composites. A good application for this product could be as a material where dimensional stability is required.

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