Characterization of Composite Formed by Cement and Wheat Straw Treated with Sodium Hydroxide

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Cement-based composites with lignocellulosic residues (*e.g.* wheat straw, rice husk, and bagasse) as fillers have reduced density and cost. Pretreatments have been applied in these raw materials to improve their compatibility with cement. This work aimed to characterize composites made from cement and wheat straw particles that had been chemically treated (2% NaOH solution) at two different temperatures (30 °C and 60 °C). Values of bulk density (209 kg·m^{-3}) and specific density (679 kg·m^{-3}) were determined for wheat straw at 12% moisture content. Straw yield was 71.9% and the particle size distribution showed large concentrations of wheat straw particulate material between 5- to 10-mesh. Composites made with wheat straw particles, in comparison to the control treatment, showed half the density, *i.e.*, from 1800 to 900 kg·m^{-3}. Also, the stress x strain curve presented a higher deformation before failure. NaOH pretreatment did not affect the mechanical properties of wheat straw composites.

Keywords: Biomass; Portland cement; Agricultural residues; NaOH; Particle pretreatment

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INTRODUCTION

The use of agricultural residues in the production of cement-based particleboards is an alternative to traditional construction materials. These composites have resistance to biological spoilage and insect infestation, as well as flame retardance properties and formaldehyde-free content (Qiao *et al.* 2016; Davies and Davies 2017). On the other hand, these materials have a limited mechanical performance and are sensitive to moisture (Calegari and Oliveira 2016).

Cobreros *et al.* (2014) and Naik and Kiran (2018) affirm that cereal straw can replace and improve the quality of conventional materials, such as wood products and asbestos cement, thus increasing their resistances, but little progress has been made in applying these residues. Research on cement wheat-straw particle composite is rare, even though these boards have the potential to replace current wood-based panels in the production of furniture and indoor and outdoor decoration (Qiao *et al.* 2016).

According to Elbashiry *et al.* (2017), the possibility of replacing conventional building materials with natural fiber-reinforced composites has been widely investigated. Straw is a convenient reinforcement in brittle cement matrix, especially in developing countries, such as China, where this material is widely available (Khorami and Ganjian 2011; Chen *et al.* 2018). More studies are needed related to the application of locally available lignocellulosic residues in order to increase its use in Brazil, although papers have been focused on lightened earth blocks production (Calegari and Oliveira 2016). This paper has a different approach, as locally available wheat straw was used to produce cement-straw composites.

Straw is used extensively in construction, in roof solutions, as fibrous reinforcement in adobe, in lightened earth blocs, and in continuous earth wall, presenting advantages as good thermal insulation, energy savings, earthquake resistance, fire resistance, and noise insulation (Cobreros *et al.* 2014; Elbashiry *et al.* 2017). From an economic perspective, the utilization of agricultural residuals increases the value of crop straw, which benefits local farmers (Qiao *et al.* 2016).

To improve the properties of the composite, chemical or mechanical pretreatments may be applied to lignocellulosic waste. Pretreatments and chemical additives influence the density and compressive strength of the composite materials, as well as other properties such as bending strength and internal bonding (Davies and Davies 2017). The most common pretreatment methods for natural fibers are steam cooking, steam explosion, physical radiation, liquid hot water, and chemical treatments (Elbashiry *et al.* 2017).

The main goal of straw pretreatments is to improve its surface compatibility with the cement matrix, which happens by the dissolution of low-molecular-weight impurities and the roughening of the surfaces (Cai *et al.* 2017; Yu *et al.* 2017).

Alkaline pretreatment also causes several changes in the wheat straw, such as increasing the surface area, modifying the lignin structure, and swelling the cellulose (Elbashiry *et al.* 2017). After pretreatment the wax layer of straw fiber is eroded, and the surface of the straw fiber becomes coarse with a large number of irregular holes, enhancing the fibers roughness and increasing the friction between cement paste and the straw fiber (Cai *et al.* 2017). Most studies present pretreatments applied to the straw fibers instead of straw particles (Basta *et al*. 2011; Khorami and Ganjian 2011; Qiao *et al.* 2016; Elbashiry *et al.* 2017; Naik and Kiran 2018).

To determine the effect of straw particles pretreatment in composite cement, this work characterized composites based on cement and wheat straw particles that had been chemically treated (solution of 2% NaOH) at two different temperatures (30 °C and 60 °C).

EXPERIMENTAL

The wheat straw was provided by farmers from the Itapeva region in the state of São Paulo, Brazil. Portland cement employed in the production of wheat straw-cement composite was CPII – E32 type. Wheat straw was characterized physically to determine its bulk, specific density, moisture content, and straw yield, as described in the following subsections.

Bulk Density and Specific Density Determination

Biomass density was determined as described by Lam *et al*. (2007). Each element of the wheat straw had its length measured with a ruler (0.1 mm precision), diameter and wall thickness measured with a caliper (0.01 mm precision), and mass measured with a precision scale (0.01 g precision).

Two types of densities were determined: bulk density and specific density. Bulk density considered the wheat straw as a filled cylinder (Eq. 1), and specific density used the wall thickness and considered the wheat straw as a hollow cylinder (Eq. 2).

$$V_f = [(\pi^*D^2{*}L)/4] * 10^{-9} \quad (1)$$

where V_f is the filled cylinder volume (m^3), D is the average straw diameter (mm), and L is the straw length (mm). The hollow cylinder volume (m^3) is given by,

$$V_h = [(D^*t - t^2)^*\pi^*L] * 10^{-9} \quad (2)$$

where t is the average wall thickness (mm).

A total of 69 samples of wheat straw were used to determine bulk and specific density. Equation 3 generally describes how density was determined. The straw density ($kg \cdot m^{-3}$) is given by,

$$D_s = (m/V) * 10^{-3} \quad (3)$$

where m is the mass (g), and V is the volume (m^3).

Moisture Content Determination

To determine the moisture content of wheat straw, 7 samples of at least 4.0 g were used. Sample mass was measured in a laboratory scale with 0.0001g of precision, and subsequently dried in a laboratory furnace at 103 (± 2) °C until achieving a constant mass (less than 0.2% variation between two consecutive measurements). The moisture content was determined using Eq. 4, according to ABNT NBR 7190 (1997) standard,

$$M = [(mi - md)/(md)] * 100 \quad (4)$$

where M is moisture content (%), mi is the initial mass of the sample (g), and md is the dried mass of the sample (g).

Straw Yield Determination

Straw yield was determined using 6 samples of at least 5.0 g, which were withdrawn from 6 to 13 plants. The ratio between straw mass and grain mass was considered in obtaining straw yield (Eq. 5), which was measured with a laboratory scale with 0.01 g of precision,

$$Y = (ms/mg) * 100 \quad (5)$$

where Y is the straw yield (%), ms is the straw mass (g), and mg is the grains mass (g).

Wheat-straw Cement Composite Production

Straw was reduced to particles with a hammer mill, and the particle size proportion distribution was determined using an electromagnetic sieve with several openings according to the recommendations of Qiao *et al*. (2016). Particles were dried in a laboratory drying oven at 103 ± 2 °C for 24 h, reaching 3% of moisture content before the production of composites.

Two pretreatments on the particles using 2% NaOH solution for 2 h were tested. Treatments were performed at a water bath (30 °C and 60 °C), where the particles were soaked in weight ratio of 1:0.25 (*i.e*., reagent solution: wheat particles). After the treatment, the particles were washed repeatedly using distilled pure water as described by Morsy (2011) and Elbashiry *et al*. (2017).

Four treatments were performed: “T1” was the control, produced with cement and water only; “T2” was the composite produced with untreated wheat straw particles; “T3” was the composite produced with wheat straw particles treated at 30 °C; and “T4” was the composite produced with wheat straw particles treated at 60 °C. Portland cement, from

Cauê InterCement®, type CP II E32 was used in the composite production.

The straw particles/cement ratio used in the production of the composites was 0.15, as specified by Cai *et al*. (2017) as the optimal amount of wheat in cement composites. The water-to-cement ratio used was 0.80, unlike Cai *et al*. (2017), for which the optimal value was 0.60 for wheat fibers, due to a higher water absorption observed on the particles.

Wheat straw cement composite specimens were produced and tested according to ABNT NBR 7215 (1996) standard. The following properties and parameters were determined: treatments density, compressive stiffness, and compressive strength after 28 days.

Detailed descriptions of the characterization methods are given in the following subsections. Treatments were produced by mixing wheat straw particles, cement, and water until achieving a homogenous mix, and then each sample was molded in a short PVC tube with a cylindrical shape (50 mm diameter and 100 mm height). The material was compacted inside the tube with a wooden compactor.

Compressive Stiffness Determination

Compressive stiffness was determined for the linear stretch of the stress *versus* strain curve obtained for the test specimens. These tests were also based on the ABNT NBR 7215 (1996) standard and performed on a universal testing machine (EMIC, INSTRON, Itapeva, SP, Brazil) with a capacity of 300 kN. The compressive stiffness calculation is shown on Eq. 8,

$$Cs = (4*h/ \pi*d^2) * [(P_{50} - P_{10})/(di_{50} - di_{10})] \quad (8)$$

where Cs is the compressive stiffness (MPa), h is the initial height of the specimen (mm), d is the specimen initial diameter (mm), $(P_{50} - P_{10})$ is the increase of load, from 10% to 50% of P_{max} (N), and $(di_{50} - di_{10})$ is the increase of displacement when $(P_{50} - P_{10})$ is applied (mm).

Statistical Analysis

Tukey tests (with 5% significance) were performed to determine if the treatments showed significant differences. Average confidence intervals (with 5% significance) were determined with raw material tests results. All statistical analyses were performed using R software, version 3.5.1 (R Core Team 2018).

RESULTS AND DISCUSSION

Table 1 shows the results obtained after the tests performed with the wheat straw. The statistical parameters and the number of samples considered in each case are also presented.

Wheat straw used to produce wheat-straw cement composite had a density higher than usual, which was attributed to the moisture content of the particles. Zhang *et al*. (2012) determined lower values of moisture content, ranging from 5% to 8%.

Lam *et al*. (2007) determined lower bulk density (ranging from 93 to 106 $kg \cdot m^{-3}$) and specific density (ranging from 395 to 557 $kg \cdot m^{-3}$) of wheat straw in comparison to results shown in Table 1. Zhang *et al*. (2012) verified the wheat straw bulk density from different regions, with values that ranged from 97.5 to 177.2 $kg \cdot m^{-3}$.

Table 1. Physical Properties of Wheat Straw

Tests	Average	Average confidence interval	Standard deviation	Coefficient of variation (%)	Number of samples
Bulk Density (*B Ds*) (kg·m^{-3})	209	202 – 217	33	15.08	69
Specific Density (*S Ds*) (kg·m^{-3})	679	610 – 748	288	42.34	69
Moisture content (*M*) (%)	12.01	10.81 – 13.20	1.14	9.47	6
Straw yield (*Y*) (%)	71.89	64.83 – 78.95	6.73	9.36	6

Lee and Grove (2005) studied six varieties of wheat straw and found that yields ranged from 68% to 83%. These values are inside the average confidence interval shown in Table 1.

The particle size distribution is shown graphically in Fig. 1. Most of the milled straw had dimensions that ranged from 5- to 10-mesh, which are considered large particles. According to Zhang *et al*. (2012), 6 to 43% of the particles were larger than 20 mesh.

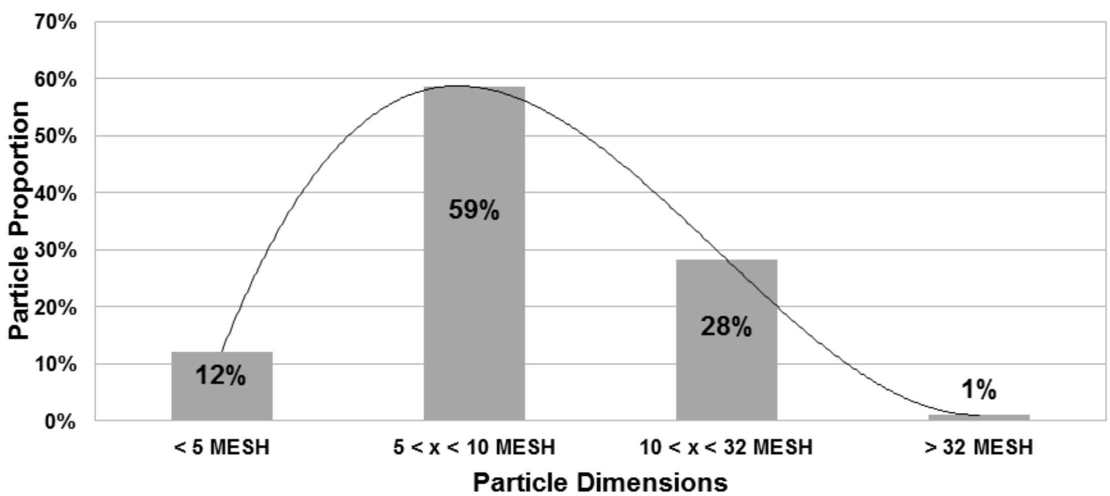

Fig. 1. Particle size distribution according to the opening sizes of three sieves (5 mesh, 10 mesh and 32 mesh)

Table 2 shows the results of the physical and mechanical tests performed in the wheat straw cement composites. The standard deviation values were placed in parentheses, and analyses of variance are shown as letters in each row.

The density of treatment T1 was twice that of wheat straw composites, indicating a significant reduction of material density when straw particles were introduced and cement was replaced. The values obtained were inferior to those obtained by Qiao *et al.* (2016), which were close to 1000 kg·m^{-3}.

The composite density was not reduced when the pretreatment temperature increased (*e.g.*, treatment T4 where produced with particles treated at the highest temperature, and still showed average density values when compared to other treatments).

The compressive strength and the stiffness of wheat straw particle composite were inferior to the control treatment. A reduction of compressive strength was expected, due to the lower density of the composite. According to Chen *et al.* (2018), an increase of percentage of straw in the composite decreases its strength. For this reason, no more than 5% of straw should be added for its use in non-load-bearing structures.

Table 2. Physical and Mechanical Properties of the Wheat Straw Composites

Tests*	T1	T2	T3	T4
Density (*Cd*) (kg·m^{-3})	1799 a (51)**	880 c (26)	936 b (31)	889 bc (28)
Compressive strength (*fc*) (MPa)	18.56 a (9.08)	0.42 b (0.07)	0.27 b (0.01)	0.44 b (0.04)
Compressive stiffness (*Cs*) (MPa)	1470 a (673)	12 b (3)	46 b (17)	51 b (13)

* Seven specimens were used for each test
** The same letters in a row means no statistical difference (5% level of significance)
T1: Control; T2: Untreated straw; T3: 30 °C straw; T4 60 °C straw

Wheat straw particles absorbed water during the mixing process with cement, forming particle agglomerations inside the composite. Cai *et al.* (2017) described that straw fibers easily absorb water as well, resulting in straw fiber agglomeration, thus reducing the mechanical properties of the composite material.

Morsy (2011) produced rice straw-cement composite (0.075:1 mass ratio) treated with 1.0% of NaOH at room temperature for a period of 24 h in a 10:1 soaking solution. These composites were produced with particles of 10- and 20-mesh sizes and achieved compressive strength close to the values in Table 1, varying from 0.42 MPa to 6.63 MPa.

Compressive stress x strain behavior of the wheat-straw cement composites are shown in Figure 2. Note that treatments T2, T3, and T4 are represented by the same dotted line, due to the same mechanical behavior.

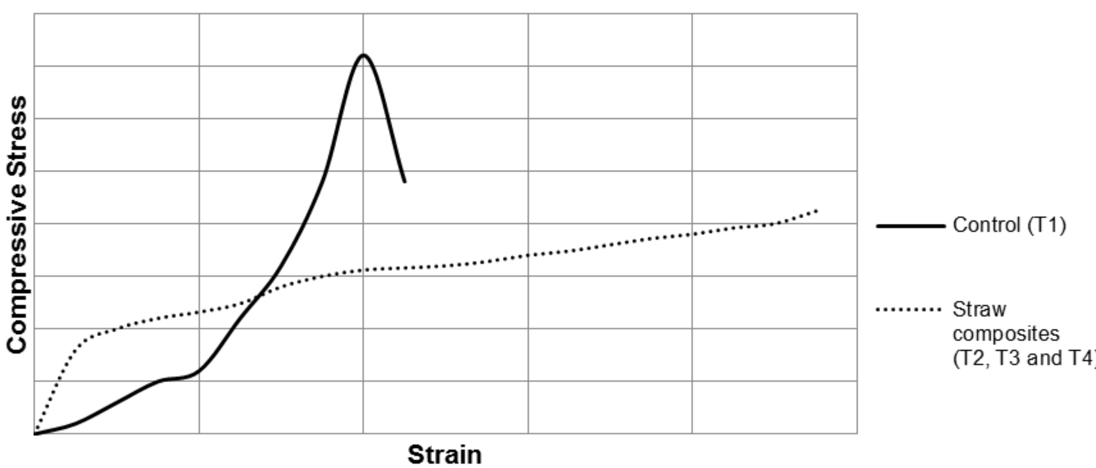

Fig. 2. Compressive behavior of wheat straw particle composite (compressive stress axis is out-of-scale)

In contrast to Morsy (2011), who reported that chemical treatment improved the mechanical performance of the composite, there was no improvement in the compressive strength performance after the chemical treatment with NaOH at both temperatures 30 °C and 60 °C compared with the untreated material. Thus, a longer treatment of agro-industrial residue with a higher amount of soaking solution presented better results, improving the interaction between composite matrix and filler.

It should be noted that the control treatment (T1) curve presented a fragile behavior, unlike the average curve presented for the straw cement composites (T2, T3, and T4) that shows large plastic deformation. The same trend was observed in the case of wood cement composite materials, which is an advantage for some specific applications due to higher energy dissipation (Frybort *et al.* 2008).

No significant difference was observed among treated and untreated straw. Basta *et al.* (2011) verified an improvement in the compressive strength and an increase of the density in composites with rice straw fibers pre-treated with linseed oil. Although Cai *et al*. (2017) affirm that high values of compressive strength of straw-cement composites cannot be reached, because straw decompose into sugars in an alkaline environment, the results shown in Table 2 suggest that alkaline treatment did not affect the particles.

CONCLUSIONS

1. NaOH pretreatment of wheat straw did not affect the strength and stiffness of the composites. Composites produced with straw particles showed half the density of the control treatment without straw.
2. Wheat straw had higher bulk and specific densities as well as higher moisture content than the values previously reported. Particle size distribution showed a concentration of large particles between 5- to 10-mesh. The straw yield value was similar to results previously published.

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