INFLUENCE OF CHEMICAL ADMIXTURES AND OF PRE-TREATMENT IN WOOD-CEMENT COMPOSITES

ABSTRACT

The purpose of this study was to verify the influence of two chemical admixtures on the properties of composites produced with Portland cement and *Pinus* spp. sawdust pretreated or not with calcium hydroxide suspension. The composites were produced with cement:wood ratio of 1:0.75 and a water/cement ratio of 0.50, containing superplasticizer admixture or air-entraining agent. Three mixtures were prepared with sawdust pretreated with 1:6 ratio of lime:water suspension (by mass). The bulk density of fresh composite was determined and prismatic specimens were produced. The results indicated that the composites produced in this study can be considered as lightweight composites (720 to 880 kg/m^3) with flexural strength between 0.5 and 3.2 MPa and compressive strength between 1.4 and 7.0 MPa. From the mechanical results, it can be concluded that wood-cement composites, except those produced with sawdust pretreated and containing air-entraining agent, presented viability for production.

Keywords: Sawdust. Portland cement. Waste. Calcium hydroxide. Admixtures.

1. INTRODUCTION

Wood is considered a renewable, versatile material with a high potential for impact mitigation (HÖGLMEIER *et al.*, 2013). In addition, waste from this material can be reused for the production of wood components and for the production of energy (KIM *et al.*, 2014; RATAJCZAK *et al.*, 2015). In this way, virgin raw materials can be replaced by recycled wood, contributing to the reduction of costs for extraction, transport and disposal processes in a sustainable way (MADRID *et al.*, 2017). According Höglmeier *et al.* (2013), about 26% of the recovered wood is suitable for reuse. Among the types of wood wastes, sawdust has been accumulated around the world creating problems in relation to environmental conservation and health (MORALES-CONDE *et al.*, 2016; PEDRENO-ROJAS *et al.*, 2017). Researchers claim that there are alternatives for the destination of various residues, which previously had no solution. Quality materials can be produced with various applications in civil construction (BREMER *et al.*, 2013).

Sawdust is generated from cutting, milling and drilling operations or during the manufacture of finished wood products, and consists of very fine particles being generally collected in filter bags or dust collectors (CORINALDESI *et al.*, 2016). Wood composites are generally obtained from mixing a binder with the wood particles. However, the use of plant material with a cement matrix also has deleterious effects on the binder, such as a strong handle retardation effect (MEHTA *et al.*, 2014).

In order to improve the compatibility of some species of wood for the production of wood-cement composites have been added chemicals or accelerators in diluted suspension - such as Sodium Hydroxide (NaOH) or Calcium Chloride ($CaCl_2$) - to accelerate the cure of the cement, carried out different pretreatments in the wood, carried out the extraction of the soluble sugars in hot and cold water (MOSLEMI *et al.*, 1983; BERALDO *et al.*, 2009). As an alternative, Fan *et al*. (2012) demonstrated that the use of pretreatment particle suspension of $Ca(OH)_2$ in water significantly improved the bond between cement and wood.

In order to improve the properties of the composites, some admixtures have been used, among them, air-entraining agents and superplasticizers. The first contributes to the production of lightweight materials (ROMANO *et al.*, 2009) because it is able to introduce small air bubbles (or voids) dispersed in the matrix that contribute to a better cohesion and the functionality of cementitious compounds preventing the penetration of the water and reducing the tendency of segregation and exudation in the fresh state (MEHTA *et al*., 2014). The second one consists of chemical compounds able to remain adsorbed on the surface of the cement particles, producing repulsive forces that contribute to the dispersion of the particles, improving their properties (UCHIKAWA *et al.*, 1995; OUATTARA *et al*., 2018).

In this sense, the objective of this study was to verify the influence of two chemical admixtures on the properties of composites produced with Portland cement and *Pinus* spp. sawdust pretreated or not with calcium hydroxide suspension.

2. EXPERIMENTAL PROGRAM

2.1 Materials

Brazilian cement CP V-ARI (Table 1), similar to Portland cement Type III ASTM-C150/2018, was used as binder.

Table 1: Chemical and physical characteristics of Portland Cement

Composition (% by mass)								**Parameter**		
Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO	SO_3	Free CaO	Na_2O_{eq}	L.O.I.	Insoluble r	Specific gravity (g

									esidue	/cm³)
4.31	18.95	2.98	60.17	3.9	3.26	0.58	0.69	3.34	0.83	3.09

Source: Manufacturer (2017).

The two adxmitures used were an air-entraining agent - AEA (ASTM-C260/2016) with pH 11.7 and density 1.05 g/cm³; and a modify polycarboxylate ether-base type superplasticizer - SUP (ASTM-C494/2014) with pH 5.0 and density 1.07 g/cm³.

The pine wood sawdust used is a waste/by-product from a logging company located in the Curitiba/Brazil. Sawdust (Figure 1) was characterized in terms of particle size composition (ASTM-C136/2014) and moisture content (ASTM-D4442/2016), which resulted in 40.86%.

Figure 1: *In natura* pine wood sawdust particles

Source: Authors, 2020.

2.2 Methods

Mix proportions (Table 2) were defined, based on cement:wood ratio of 1:0,75, in mass, and water/cement ratio of 0.50. Superplasticizer (SUP) and air-entraining (AEA) contents were set at 0.40% by weight of binder, as indicated by the manufacturers. Also, composite mixtures with no admixture were prepared as reference samples (REF). Sawdust particules were immersed in a saturated lime suspension, prepared with calcium hydroxide type CH II (NBR 7175/2003) with a lime:water ratio of 1:6 in mass, for 15 minutes.

Table 2: Identification and composition of wood-cement composites

Binder	**Pretreatment**	**Sample identification**	**b/w ratio**	**Admixture**		**w/c ratio**
				Type	**Dosage (%)**	
Portland cement	No Treatment	NTREF	1:0.75	-	-	0.50
		NTSUP		SUP	0.40%	
		NTAEA		AEA	0.40%	
	Lime Suspension	LSREF		-	-	
		LSSUP		SUP	0.40%	
		LSAEA		AEA	0.40%	

Legend: b/w is the binder/wood ratio; w/b is the water/cement ratio.

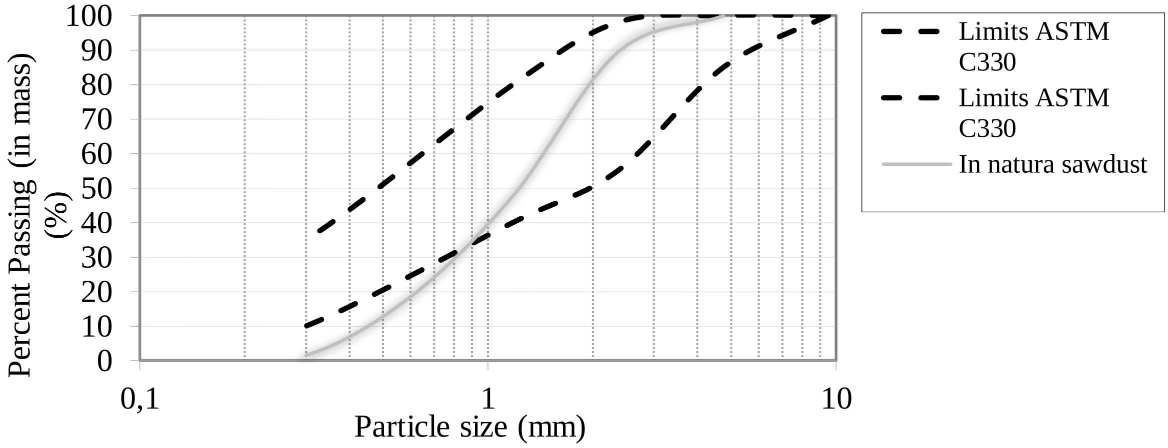
The bulk density of fresh composite was performed (ASTM-C270/2014). Six prismatic specimens of 4x4x16 cm^3 were casting for each composite mixture. At 28 days, the bulk density of hardened composite, the flexural strength ASTM-C348/2014 and the compressive strength ASTM-C349/2014 were determined. The Minitab 18 software was used for the statistical analysis. All statistical tests were carried out at 95% confidence level. The Pearson correlation coefficient was used to evaluate the relationship between the investigated properties.

3. RESULTS AND DISCUSSION

3.1 Particle size analysis of wood sawdust

The particle size composition of pine wood sawdust is presented in Figure 2 and compared with the limits according to the ASTM-C330/2017. Thus, due to its shape and small size (0.15 to 2.36 mm), the sawdust can be considered as a fine aggregate.

Figure 2: Particle size composition of pine wood sawdust compared with the limits according to the ASTM-C330/2017



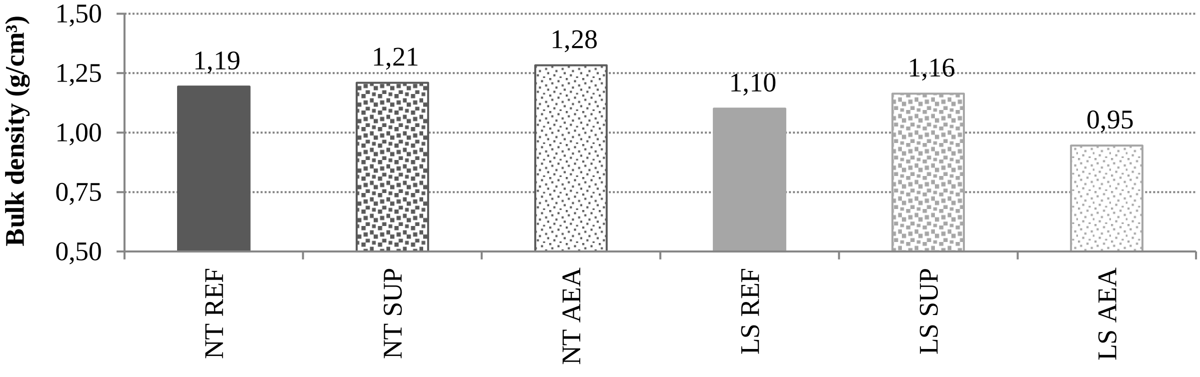
Source: Authors, 2020.

Also, it is important to highlight the geometry of sawdust particles may be related to the structure of the composite, i.e., because larger particles tend to limit the compaction procedure for the production of composite (AIGBOMIAN *et al*., 2013).

3.2 Bulk Density of Fresh Composite

The results of bulk density of fresh composite are shown in Figure 3.

Figure 3: Results of bulk density of fresh composites



Source: Authors, 2020.

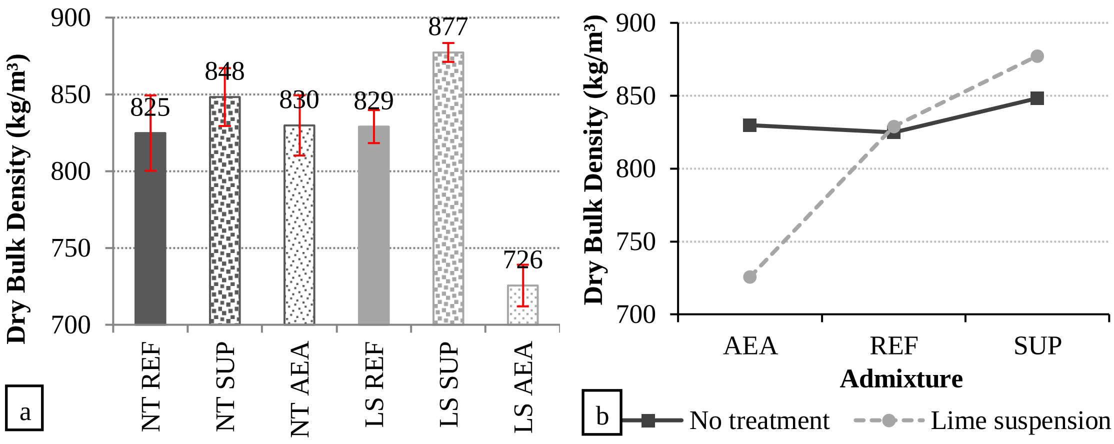
It is observed that the composites produced with pretreated sawdust in lime suspension presented lower bulk density when compared to composites with sawdust without pretreatment, different from that observed by Azambuja *et al*. (2017). With respect to the admixture used in the production of the composites, it is verified that regardless of whether or not the pre-treatment was carried out, the samples containing superplasticizer presented a higher bulk density when compared to their respective reference samples. This is because, superplasticizers admixtures are able to change the rheology and behavior of the (FLATT *et al.*, 2012; NKINAMUBANZI *et al.*, 2016) as, for example, contributing to the reduction of the pores in the cement matrix (ZHANG *et al.*, 2014) and, consequently, increasing the density of the composite (BOŁTRYK *et al.*, 2018).

However, the composites produced with the air-entraining agent had different behaviors, because the composites produced with the particles without pre-treatment obtained a bulk density higher than the reference sample. This phenomenon may occurred due to the fact that the presence of the air-entraining agent triggers an extremely complex process and can be affected by numerous factors, such as the mixing process, the presence of aggregates, the physicochemical properties of the cement, among others (OKINO *et al.*, 2005), while the results of composites produced with air-entraining agent and containing pretreated sawdust in lime suspension decreased when compared to the reference sample (BENAZZOUK *et al.*, 2006).

3.3 Dry Bulk Density of Hardened Composite

Figure 4 presented the results of dry bulk density of hardened composites at 28 days.

Figure 4: Results (a) and the interactions (b) of the dry bulk density of hardened composites



Source: Authors, 2020.

From the statistical analysis performed by Fisher's LSD method with a 95% confidence level, it can be observed that the reference composites (no admixtures) did not presented a significant difference in relation to the pretreatment adopted. Even as, considering the pre-treatment used, it is verified that the composites produced without lime suspension presented statistical equivalence between the composites produced (REF, SUP e AEA). However, for the composites produced with the lime suspension, a significant difference was observed among the dry bulk density of hardened composites produced with the different types of admixtures used.

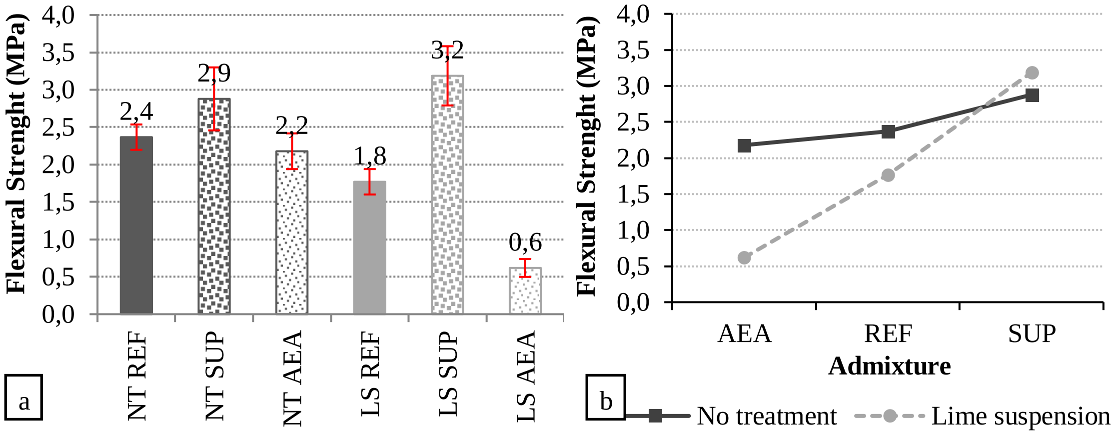
It is important to note that the higher dry bulk densities were observed for the composites produced with the pretreatment and with the SUP admixture. This is due to the fact that, as previously reported, superplasticizers contribute to the reduction of pores in the cementitious matrix (ZHANG *et al.*, 2014) and, consequently, for increasing the composite density (BOŁTRYK *et al.*, 2018), while the lower values were obtained by the composites produced with the pretreatment and the AEA admixture (AAMR-DAYA *et al.*, 2008; USMAN *et al.*, 2018). Thus, it is understood that the use of the admixtures (AEA and SUP) when combined with the pretreatment in lime suspension significantly change the density of the composite in the hardened state, when compared with the other compositions.

In general, the results of dry bulk density of hardened composites were between 720 and 880 kg/m³ indicating that the composites produced can be considered as lightweight materials (CORINALDESI *et al.*, 2016).

3.4 Flexural Strength

The results of flexural strength of composites at 28 days are shown in Figure 5.

Figure 5: Results (a) and interactions (b) of the tensile strength of the composites



Source: Authors, 2020.

From the statistical analysis performed by Fisher's LSD method with a 95% confidence level, it can be observed that, when considering the pretreatment used, the composites produced with the lime suspension presented a significant reduction of the flexural tensile strength when used without admixture (REF) and with AEA admixture. This may have occurred because of the greater availability of water provided by the pretreatment it contributes to increase porosity and consequently to reduce the mechanical performance (KUMAR *et al.*, 2003; CAPRAI *et al.*, 2018). Other researchers also suggest that softened particles tend to weaken their bond with the cementitious matrix (ONUAGULUCHI *et al.*, 2016). In addition, the different hydration processes of the two binders present in the composites - when carried out the pretreatment (lime and Portland cement) - can influence the performance of the composites (AIGBOMIAN *et al.*, 2013). However, when used with SUP admixture the effect of pretreatment is not significant.

It is important to note that the higher flexural strengths were observed for composites produced with SUP admixtures, regardless of whether the pretreatment was performed, while the smaller results were obtained by the composites produced with the admixture AEA and the pretreatment with lime suspension. This is because, due to the presence of air voids, in addition to presenting lower density, the composites tend to develop inferior mechanical properties (NARAYANAN *et al*., 2000; AAMR-DAYA *et al*., 2008; WONG *et al*., 2011; TORRES *et al*., 2014). It is verified that the use of admixtures (AEA and SUP) significantly changes the flexural strength of the composites when compared to the mixtures without admixtures (REF). The SUP admixture, used to increase workability, helped to improve the flexural strength of the wood-cement composites, since it allowed cement particles dispersion and, consequently allowed cement hydration (GARCEZ *et al*., 2017).

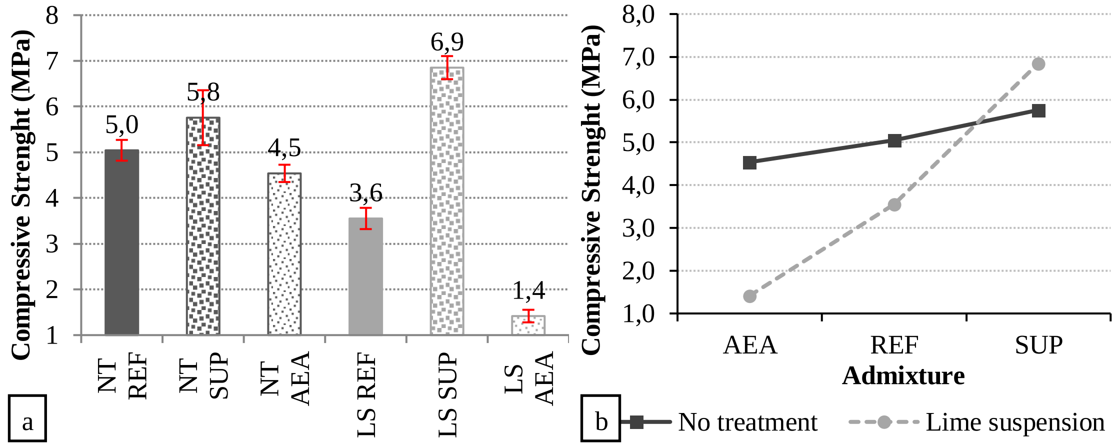
However, it's important to emphasize that the dispersion of sawdust may influence the mechanical performance of the specimens during the placement of the composites in the molds, especially in the property of flexural strength (ÇOMAK *et al*., 2018) and therefore, the standard deviation of the samples tends to be higher.

In general, the results of flexural strength were between 0.5 and 3.2 MPa indicating that the composites, except those produced with pretreatment and with AEA admixture, demonstrate the feasibility of wood-cement composites manufacture.

3.5 Compressive Strength

Figure 6 shows the results of compressive strength of composites at 28 days.

Figure 6: Results (a) and the interactions (b) of the compressive strength of composites



Source: Authors, 2020.

From the statistical analysis performed by Fisher's LSD method with a 95% confidence level, it can be observed that when considering the pretreatment used, the composites produced with the lime suspension presented a significant reduction of the compressive strength when used without admixtures (REF) and with admixture AEA.

As previously explained, the decrease in compressive strength observed for the composites produced with the pretreated sawdust may have been influenced by the greater availability of water that contributed to the increase of the porosity and to the decrease of the mechanical performance (KUMAR *et al*., 2003; CAPRAI *et al.*, 2018; USMAN *et al*., 2018). It's also considered that in addition to the softened particles favoring the weakening of the bond with the cementitious matrix (ONUAGULUCHI *et al*., 2016), and the different hydration processes of the two binders present in the composites can influence the performance (AIGBOMIAN *et al*., 2013).

It's important to note that the higher compressive strengths were observed for composites produced with SUP admixture, regardless of whether the pretreatment was performed (BOŁTRYK *et al.*, 2018; OUATTARA *et al.*, 2018), while lower strengths were obtained by the composites produced with the AEA admixture in both pretreatments. This is because, due to the presence of air voids, in addition to presenting lower density, the composites tend to develop inferior mechanical properties (NARAYANAN *et al*., 2000; PUNURAI *et al*., 2006; AAMR-DAYA *et al*., 2008; WONG *et al*., 2011; TORRES *et al.*, 2014). It's understood, then, that the use of the admixtures (AEA and SUP) significantly changes the compressive strength of the composites when compared with the mixtures without admixtures (REF). The SUP admixture, used to increase workability, helped to improve the compressive strength of the wood-cement composites, since it allowed cement particles dispersion and, consequently allowed cement hydration (GARCEZ *et al*., 2017).

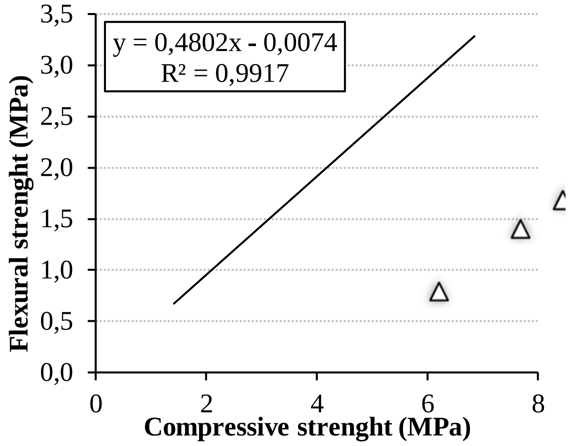
In general, the results of compressive strength were between 1.4 and 7.0 MPa indicating that the composites, except those produced with pretreatment and with AEA admitures, can be considered light materials, similar to mortars that have values of about 3.5 and 4.5 MPa (COATANLEM *et al.*, 2006; AAMR-DAYA *et al*., 2008; BOŁTRYK *et al.*, 2014).

3.6 Correlation of hardened state properties of composites

Figures 7 and 8 show the correlations of hardened state properties of wood-cement composite: dry bulk density, flexural strenght and compressive strenght of the composites at 28 days. There is a positive correlation (r = 0.996) which indicates the tendency of increasing flexural strength with increasing compressive strength (Figure 7). According to the adjusted equation for the linear regression model, with R^2 = 99.2%, the relationship between flexural strength and compressive strength is approximately 0.48 (i.e., 48%).

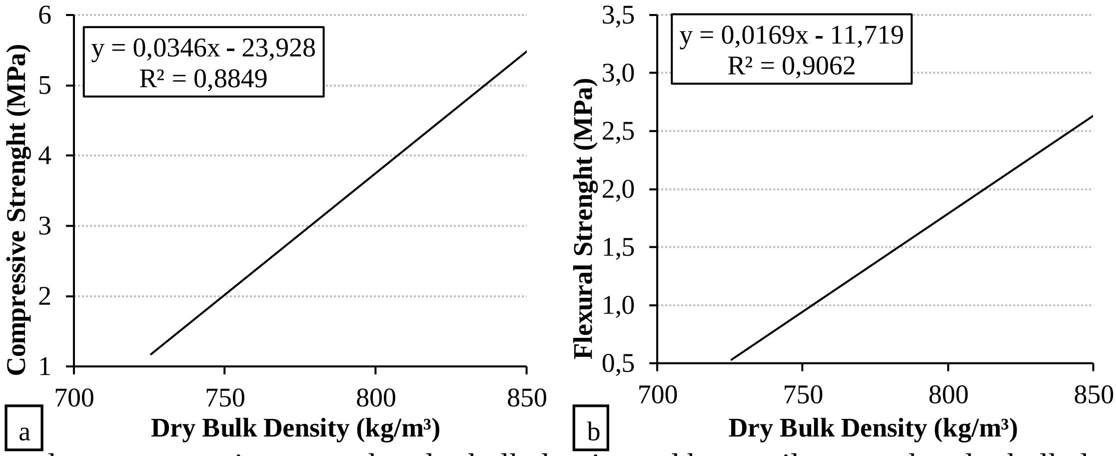
A positive correlation (r = 0.941) indicating the trend of increasing compressive strength with increasing dry bulk density of hardened composite (Figure 8a) was also observed. The adjusted equation for the linear regression model explains 88.5% of the variation in compressive strength. Finally, a positive correlation (r = 0.952) indicated the tendency of increasing flexural strenght with increasing dry bulk density of hardened composite (Figure 8b). The adjusted equation for the linear regression model explains 90.6% of the variation in flexural strength.

Figure 7: Correlation between flexural strength and compressive strength



Source: Authors, 2020.

Figure 8: Correlation between the mechanical strengths and the density of composites



Legend: a - compressive strength x dry bulk density and b - tensile strength x dry bulk density

Source: Authors, 2020.

4. CONCLUSIONS

In this work, several wood-cement composites were produced and, based on the experimental results, the following can be concluded particle pretreatment and admixtures might influence the physical and mechanical properties of wood-cement composites. *Pinus* spp. sawdust used in the development of this research can be considered as a fine aggregate from the granulometric analysis performed.

All the composites produced in this study with Portland cement and *Pinus* spp. sawdust can be considered as lightweight composites. Wood-cement composites produced with superplasticizer admixture had the highest dry bulk density in the hardened state among the evaluated composites. Pretreatment with saturated lime suspension was not able to significantly change the density of the reference composites in the hardened state.

Highest values of flexural and compressive strengths were observed for composites produced with SUP admixtures, regardless of whether the pretreatment was performed. Composites produced with sawdust pretreated with saturated lime suspension showed a significant reduction of the flexural and compressive strength when used without admixture (REF) or with the AEA admixture. Use of admixtures (AEA and SUP) significantly altered the compressive strength of the composites when compared to the mixtures without admixture (REF). The superplasticizer, used to increase workability, helped to improve the mechanical properties of the wood-cement composites, since it allowed cement particles dispersion and, consequently allowed cement hydration.

All composites produced with the AEA admixture presented lower mechanical performance when compared to their respective reference composites REF. Compressive strength results indicate that composites, except those produced with pretreatment and with AEA admixture, can be considered lightweight materials and resemble mortars having values of around 3.5 and 4.5 MPa. Composites made with superplasticizer (SUP) or without admixtures (REF) show results of density, flexural strength and compressive strength that demonstrate the feasibility of wood-cement composites manufacture.

From the correlations of the properties in the hardened state, it was observed that dry bulk density is directly proportional to compressive and flexural strength, i.e., the decrease in the dry bulk density contributed to the reduction of the mechanical performance of the composites. The correlation test showed a high positive correlation (r=0.996) between flexural and compressive strength. According to the linear regression model (R^2 = 99.2%) flexural/compressive strength ratio is approximately 0.48.

5. ACKNOWLEDGMENT

The authors would like to thank the Materials and Structures Laboratory (LAME/DCC) of the UFPR and the Post-Graduation Program in Civil Construction Engineering (PPGECC) of the UFPR; the Coordination for the Improvement of Higher Education Personnel (CAPES) and the Araucária Foundation for financial assistance.

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