

Potential of Jerusalem Artichoke (*Helianthus tuberosus* L.) stalks to produce cement-bonded particleboards



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ABSTRACT

Jerusalem Artichoke (*Helianthus tuberosus* L.) stalk particles were studied as an alternative raw material for cement composites. Cement-bonded particleboards measuring 300 × 300 × 10 mm with nominal density of 1250 kg/m³ were produced and assessed. Particleboards were subjected to two curing conditions: (1) – 48 h in a climatic chamber, followed by 25 days in an air saturated environment; and (2) – 48 h in a climatic chamber, and then in a dioxide carbon (15% concentration) environment (24 h), followed by 24 days in an air saturated environment. Physical and mechanical characterizations were carried out for both particleboard samples at 28 days. The particleboard tested in this study may be applied for industrial purposes, since cement-bonded Jerusalem Artichoke particleboards meet the mechanical requirements of the ISO 8335 Standard for building applications. Furthermore, thickness swelling and water absorption of the particleboards were lower than values reported in the literature and Standards requirements. In view of the results, Jerusalem Artichoke stalk particles are suitable to be used to produce cement-bonded particleboards for construction applications.

1. Introduction

The cement-bonded particleboards have been used in Austria since the 1920s and Germany since the 1940s. After World War II, there was an expansion of their use in North America and Asia. Nowadays, the acceptance of this product is reported as an appealing technology, because particleboards made of a polymer matrix are vulnerable to environmental weathering, heat and fungal attack, while the use of a Portland cement (PC) as a matrix, significantly provides the strength performance and structure durability (Wang et al., 2016).

However, currently wood is basically the most used raw material to produce cement-bonded particleboards, a fact which has been increasing the demand and consequently the wood price (International Energy Agency, 2016). On the other hand, this increase in demand and higher wood prices can stimulate the companies to look for new alternative raw materials.

Thinking of a solution for all the above-mentioned issues, the search for alternative lignocellulosic raw materials that can dampen the pressure on slow growing forests is crucial. As such, several vegetal materials could have great potential as alternative raw material for the cement-bonded particleboard production, showing acceptable chemical

composition and morphological structures (Ayrlimis et al., 2017; Cabral et al., 2017; Cavdar et al., 2017; Dong et al., 2016).

In recent years, many studies have been conducted in order to apply stalks of vegetal materials to produce building elements, especially sunflower (Mati-Baouche et al., 2014; Nozahic and Amziane, 2012), eggplant (Guntekin and Beyhan, 2008) maize (Babatunde, 2011), cotton (Zhou et al., 2010) and achar (Aggarwal et al., 2008).

Jerusalem Artichoke (*Helianthus tuberosus* L.) is an herbaceous and tuberoscous vegetal of the *Asteraceae* family which has several advantages over other vegetal materials, such as high growth rates (over 6–8 t/ha), good frost and drought tolerance and growth in soils with minimal fertilizer requirements (Slimstad et al., 2010). The chemical composition of Jerusalem Artichoke (JA) stalks is similar to that of softwoods usually used to produce cement-bonded particleboards. The JA stalk presents an average content of 41% cellulose, 22% hemicellulose and 20% lignin (Wróblewska et al., 2009), while softwood presents a content of 45% cellulose, 20% hemicellulose and 25% lignin (Fengel and Wegener, 2003).

However, stalks from vegetal materials, when agglomerated with cement, have low durability due to the alkaline medium (pH ~ 12), as there is the mineralization of these vegetal materials by the

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precipitation process of the PC hydration products inside (lumen), and around these vegetal materials (Cabral et al., 2017). To provide a more durable cement-bonded particleboard, an alternative shown by Cabral et al. (2017) is the adoption of curing by accelerated carbonation, which provides cement-bonded particleboards with lower water absorption, thickness swelling and the preservation of the mechanical properties. Additionally, accelerated carbonation can provide a longer durability of the vegetal stalk material inside the PC due to the pH decreasing with consequently less chemical attack on the vegetal matter (Almeida et al., 2013; Santos et al., 2015).

Hitherto, there is a lack of information regarding the potential of JA stalk particles to produce PC composites. Therefore, the goal of this study was to evaluate the potential of JA stalks to produce cement-bonded Jerusalem Artichoke particleboard (CBJAP) cured by accelerated the carbonation process.

2. Materials and methods

This research was conducted in three steps: in the first step the collection and preparation of the raw material (JA stalks), physical, chemical and morphological characterization, as well the hydration test of JA in a PC matrix was conducted. In the second step, the manufacturing as well as the curing process of CBJAP was carried out. Finally, the products underwent analytical, thermal, physical and mechanical characterizations in order to determine the feasibility of producing CBJAP cured by accelerated carbonation process.

2.1. Raw material and preparation

JA stalks collected and processed in Acton Vale, Quebec, Canada were dried in a forced ventilation oven (Model MA 035, Marconi, Brazil) at 60 °C for 72 h. After the drying process, JA stalks were ground using a knife mill (Model DPC-1, Cremasco, Brazil) and then separated on an automatic shaker (Model G, Produtest, São Paulo, Brazil) to obtain particles with an eight-mm length. Brazilian Portland cement type CPV-ARI (high early strength) was used in this experimental work, referring to previous studies on cement-based composites (Tonoli et al., 2009). This PC is equivalent to Type III ASTM C150 (2011).

2.2. Jerusalem Artichoke (JA) characterization

2.2.1. Chemical characterization

To perform the chemical characterization, JA particles were ground and sieved to reduce particle size to below 1 mm in a Wiley mill (Model 4, Thomas Scientific, USA). The cellulose, hemicellulose and lignin contents were analyzed according to the French Standards XPU 44–162 (AFNOR, 2005) using the Van Soest method. ASTM D1110 Standard (2007) was used to analyze the extractives content.

2.2.2. X-ray diffraction

JA particles were ground and sieved in a Wiley mill (Model 4, Thomas Scientific, USA) to reduce particle size below 1 mm, the particles were oven-dried (60 °C, 24 h), and subsequently characterized with the aid of AXS Analytical X-Ray Diffractometer (Model, AXS D5005, Siemens, Germany), operated at 1600 W of power: 40 kV x 40 mA. Cu-K alpha radiation, wavelength λ : 1.54056 Å inherent in the copper tube. Standardized test: 2 θ angle range from 5° to 70° in reflection mode scanned at 2°/min.

The crystallinity index (CI) of JA particles was calculated according to the Buschle-Diller and Zeronian (1992) method. To compare the JA CI value, softwood XRD analysis was also carried out using the same conditions of the JA experimental analysis of this work. The CI is defined from Eq. (1):

$$CI = 1 - \frac{I_1}{I_2} \quad (1)$$

Where I_1 is the intensity at the minimum 2 θ value between 18° and 19° and I_2 is the intensity associated with the crystalline region of cellulose (2 θ values between 22° and 23°).

2.2.3. Morphological characterization

The morphological characterization of the JA particles (oven-dried at 60 °C, 24 h) without a metallic coating and without epoxy resin impregnation were evaluated in a Scanning Electron Microscope (SEM), model TM-3000, Hitachi, Japan. The particles were fixed to metallic holders (“stubs”) and images were generated by the acquisition of backscattered electrons in different fields and magnifications (50, 100 and 500x) with a working distance of 6.60 mm. Therefore, the main cell types from the pith and bark of the JA particles were evaluated. Around 50 SEM images were obtained from twenty different samples for each magnification. However, just some of the representative images of each magnification were used in this manuscript.

2.3. Hydration test

Vegetal materials can mitigate the temperature increment that occurs during the PC setting process. To assess the compatibility of the JA particles within the PC matrix, a preliminary hydration test was conducted using a 500-mL stainless steel vessel insulated by a vacuum double wall. For each repetition ($n = 5$), 15 g of JA particles were mixed with 200 g of PC and a specific quantity of water for each sample was calculated using Eq. (2) (Hachmi et al., 1990).

$$W = C \left[JA \left(0.3 - \frac{MC}{100} \right) \right] \quad (2)$$

Where W is the water volume (mL), C is the amount of PC (g), JA is the mass of JA (g) and MC is the moisture content (%) of JA particles.

PC-JA-water samples were mixed during 5 min and immediately dropped into plastic bags. A J Type Thermocouple (Omega, Stamford, Connecticut, USA) was inserted into each fresh mixture and connected to a data acquisition system (Brand Campbell Scientific Data 21 X) which ensured temperature data recording every minute for a period of 24 h. Additionally, a PC-water (control samples) were prepared without JA particles. Each sample was put into a 500-mL stainless steel vessel isolated by a vacuum double wall. All adiabatic systems were put into a 45.4 L thermal box (Coleman, Wichita, Kansas, USA) filled with fiberglass insulation (R-40 EcoTouch, Toledo, Ohio, USA) to avoid heat exchange. The thermal box was stored in a room kept at a temperature ranging from 23 to 29 °C. The hydration data were obtained and the inhibitory index was calculated according to the Okino et al. (2004) method.

2.4. Manufacturing and characterization of CBJAP

The CBJAP production parameters are shown in Table 1. CBJAP were manufactured with a target density of 1250 kg/m³ (air-dry density), and a thickness of 10 mm. Initially, the JA particles were inserted in a planetary mixer, and subsequently the water was added using a spray nozzle. PC was added into the mixer and the mixture was homogenized for five min to prevent agglomeration.

After the homogenization, the mixture was manually placed in a

Table 1
Production parameters of CBJAP.

Particleboard dimensions		Particleboard constituents	
Length (mm)	300	Jerusalem Artichoke particle length (mm)	8
Width (mm)	300	Jerusalem Artichoke moisture (%)	8
Thickness (mm)	10	Jerusalem Artichoke ratio (g)	216
Target density (kg/m ³)	1250	PC ratio (g)	683
		Water (g)	360

wooden mold (300 mm × 300 mm) to provide a density as uniform as possible and pre-pressed to a thickness of approximately 40 mm. The pre-pressed sample was then placed in a hydraulic press (Model PHH100T, Hidral-Mac[®], Brazil) and subjected to 3 MPa pressure for 24 h at room temperature. The final thickness of CBJAP was (10 ± 0.1) mm and a total of 20 particleboards, 10 per each curing process were manufactured.

2.4.1. Curing process

The CBJAP were subjected to two curing processes. The main reason to use two curing conditions in this study is based on the improvement of the physical and mechanical properties of the composites in the initial ages as well as the improvement of the cement-bonded composites durability, as previously reported by Cabral et al. (2017).

The first process was the control curing. Control curing of CBJAP consisted of storing the materials in a controlled environment (60 °C; 90% relative humidity) for 48 h (Model EPL4H, Espec Corporation, USA), and subsequently maintaining them in a saturated environment (i.e. sealed in plastic bags), at 23 °C for 25 days.

The second process was the carbonation curing. The accelerated carbonation procedure described by Cabral et al. (2017) was adopted using a climatic chamber (Model EPL4H, Espec Corporation, USA). After maintaining the CBJAP for 48 h in a controlled environment at 60 °C and 90% relative humidity, carbon dioxide (15% concentration by volume) was added during 24 h. The completion of the cure was carried out in a saturated environment at 23 °C (in sealed plastic bags) for 24 days.

2.5. Evaluation of the curing procedure

2.5.1. Analytical methods

To evaluate the curing treatments, three methods were used: visual assessment (phenolphthalein indicator); thermogravimetry (TG) and X-ray diffraction (XRD).

When sprayed, the phenolphthalein indicator measures the depth of the colorless zone of PC composites. The pH of the colorless zone is then less than 9 and corresponds to the carbonated region of the PC composite (Metalssi and Ait-Mokhtar, 2009).

To perform visual assessment with the phenolphthalein indicator, a solution was prepared with 2% of phenolphthalein diluted in ethanol. This solution was sprayed on the CBJAP (28-day age) for both curing procedures adopted in this study.

TG tests were performed using a TG/DSC analyzer (Model STA449 F3 Jupiter[®], Netzch, Germany). A nitrogen gas dynamic atmosphere (50 mL/min) was used at a constant heating rate of 10 °C/min from 25 to 1000 °C.

Portlandite and calcium carbonate amounts were estimated using the molar mass balance, considering that 74 g/mol, 44 g/mol, 100 g/mol and 18 g/mol are the molar masses of portlandite, dioxide carbon, calcium carbonate and, H₂O, respectively. Mass loss was obtained from the TG curve and the derivative thermogravimetry (DTG) results were used to inspect the nature of hydration products formed in the cement-based systems as proposed by Borges et al. (2010) and Santos et al. (2015).

The XRD analysis was used to identify variations due to different curing processes in the main crystalline phases present in the CBJAP samples. XRD was conducted in an Analytical X-Ray Diffractometer (Model, AXS D5005, Siemens, Germany), operated at 1600 W of power (40 kV × 40 mA, Cu-K alpha radiation, wavelength λ 1.54056 Å inherent in the copper tube).

Standardized test: 2θ angle range from 5° to 60° in reflection mode scanned at 2°/min. International Centre Diffraction Data (ICDD) was applied to identify crystalline phases. To produce the powder specimens to conduct the TG and XRD tests, we extracted small pieces from the control and carbonated CBJAP (28 days old) and prepared the specimens as described by Mohr et al. (2007).

2.6. Microstructural characterization of the CBJAP

Microstructural characterization of the CBJAP was performed by SEM analysis, in an electron microscope (Model TM-3000, Hitachi, Japan) with 15 kV (acceleration voltage). The images were generated via acquisition of backscattered electrons at 500 X magnification with a working distance of 6.60 mm.

Samples of the control and carbonated CBJAP (28-day age) analyzed by SEM were initially soaked in isopropyl alcohol for 10 h, followed by drying in an oven at 60 °C for 24 h (Mohr et al., 2007). Subsequently, samples were embedded in epoxy resin (Akasel) and cured for 24 h without the use of vacuum. Polishing of the samples was conducted in an automatic polishing machine (Model TegraPol-1, Struers, Denmark), using silicon carbide abrasive paper with sequential grit sizes of 320, 600, and 1200 for 2 min each at an applied load of 10 N using isopropyl alcohol as a lubricant. A final polishing was carried out with diamond paste composed of 6, 4, and 1 μm (Struers) for 4, 2, and 1 min, respectively, at 10 N of applied load (Cabral et al., 2018a,b). Around 50 SEM micrographs were obtained for each CBJAP curing process and only typical images of the observed microstructure of each treatment were used in this work.

2.7. Physical and mechanical characterization of the CBJAP

Physical testing of the CBJAP followed the procedures established by the Standards for Wood-based panels: Determination of moisture content, EN322 (European Committee for Standardization, 1993a) Wood Based Panels; and Determination of bulk density, EN 323 (European Committee for Standardization, 1993b). For the physical tests, 40 specimens (50 mm × 50 × 10 mm) were used for each curing procedure.

Mechanical tests were performed on the CBJAP specimens (28 days old) in equilibrium with the temperature and air humidity of the laboratory using the universal testing machine (Emic Model DL 30000, Illinois Tool Works, USA). The specimens were prepared with a diamond saw blade, with nominal dimensions of 250 mm × 50 mm × 10 mm to determine the flexural performance, and samples with 50 mm × 50 mm × 10 mm to determine the internal bond (IB).

A three-point bending test configuration with a span of 200 mm was used to calculate the mechanical properties for the 40 specimens of each experimental treatment: modulus of rupture (MOR) and modulus of elasticity (MOE), Eq. (3) and Eq. (4) respectively. A crosshead speed of 7 mm/min was applied during the tests according to recommendations of the Standards for Wood-based panels. Determination of MOE and MOR in bending followed the recommendations of the EN 310 (European Committee for Standardization, 1993c).

$$\text{MOR} = \frac{3F_{\text{max}} l_1}{2bt^2} \quad (3)$$

Where: MOR = modulus of rupture, in MPa; F_{max} = maximum load, in N; l₁ = length of the major span, in mm; b = width of the specimens, in mm; t = thickness of the specimens, in mm.

$$\text{MOE} = \frac{l_1^3(F_2 - F_1)}{4bt^3(a_2 - a_1)} \quad (4)$$

Where: MOE = modulus of elasticity, in MPa; l₁ = length of span, in mm; F₂-F₁ = is the increment of load on the load-deflection curve, where F₁ was approximately 10% and F₂ 40% of the maximum load; b = width of the specimens, in mm; t = thickness of the specimens, in mm; a₂-a₁ = the increment of deflection corresponding to (F₂-F₁) in load-deflection curve.

The IB specimens (50 mm × 50 mm × 10 mm) were bonded to steel braces using a fast curing epoxy adhesive. The determination of the IB strength followed the procedures established by the Standards for Particleboards and Fiberboards: Determination of tensile strength perpendicular to the plane of the board (European Committee for

Standardization, 1993d). The specimens were tested until ultimate failure with a continuous crosshead speed of 0.5 mm/min.

$$IB = \frac{F_{max}}{a \times b} \quad (5)$$

Where: IB = Internal bond; Fmax = maximum load, in N; a = width of the specimens, in mm; b = length of the specimens, in mm.

2.8. Statistical analysis

The analysis of the data was performed in an arranged completely randomized design, where the factor studied corresponds to the CBJAP with two levels (control and carbonated), totaling two treatments. The Tukey test was performed by ANOVA analysis, being both tested with $p < 0.05$. The software used to interpret the results was SAS version 2.5.1. (2011).

3. Results and discussion

3.1. Jerusalem Artichoke (JA) analysis

3.1.1. Chemical composition

Table 2 shows the mean values of the chemical constituents of the JA particles used for the CBJAP production and the literature results for the JA as well as softwood chemical constituents.

Chemical constituents of vegetal materials vary with harvest time and soil and environmental issues, which result in differences in the experimental values of the JA particles as displayed in Table 2. Based on the chemical constituents of the JA particles of this research, the chemical constituents of these samples are closer to those softwoods (Table 2), usually applied to produce the cement-bonded particleboards.

3.1.2. Crystallinity analysis

The CI value of the cellulose can be a parameter to evaluate the amount of the crystalline portion in the cellulose in relation to the total amount of cellulose of a vegetal material. Higher CI values can point to higher material hardness and specific gravity (Gurunathan et al., 2015; Methacanon et al., 2010; John and Thomas, 2010). Additionally, higher CI values can represent a decrease in water absorption, wet expansion and chemical reaction activity. Therefore, the CI value of the JA particles helps to better understand the relationship between the internal structure of particles and their properties, and thus provide a scientific basis for JA particles usage. The typical XRD pattern of the JA particles is shown in Fig. 1.

The mean value, as well as the standard deviation for the crystallinity index (CI) of the JA (Fig. 1), is listed in Table 3. As shown in

Table 2

Chemical composition of Jerusalem Artichoke compared to other previous studies.

	Jerusalem Artichoke			Softwood
	Present research	Gunnarsson et al. (2014)	Klímek et al. (2016)	Fengel and Wengel (2003)
Cellulose (%)	49.4 * (0.3)	31.0	30.9	45
Hemicellulose (%)	13.3 * (0.5)	13.0	–	20
Lignin (%)	21.8 * (0.2)	17.0	16.3	25
Extractives (%)	12.1 * (0.9)	–	–	8
Humidity content (%)	8.2 * (0.3)	–	–	–

*Each value represents the mean of three replicates followed by standard deviation.

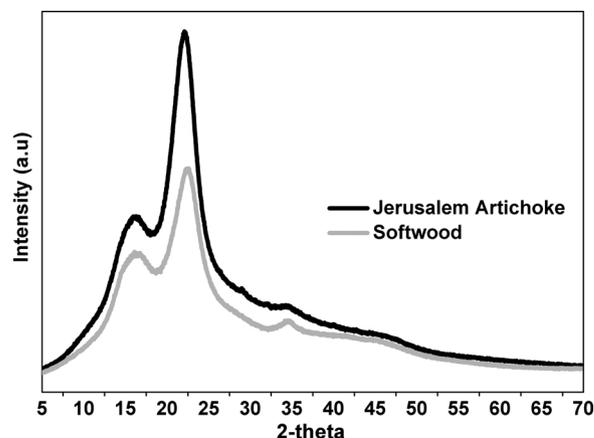


Fig. 1. XRD pattern obtained for the JA particles and softwood.

Table 3

Crystallinity index (CI) value of the Jerusalem Artichoke used in the production of CBJAP in comparison to the softwood species.

	Jerusalem Artichoke	Softwood
CI	0.56 *(0.01)	0.46 *(0.01)

*Each value represents the mean of three replicates followed by standard deviation.

Fig. 1, the XRD pattern of JA showed a major peak intensity at the diffraction angle of 2θ at around 22° . JA particles presented a CI value of 0.56 while softwood presented CI equal to 0.46 (Table 3). Thus, JA particles are potentially suitable to produce CBJAP, since the higher crystallinity of the vegetal material represents improved chemical resistance, higher density and greater mechanical performance in Portland cement (PC) composites (Gurunathan et al., 2015; Methacanon et al., 2010; John and Thomas, 2010; Hu and Hsieh, 2001).

3.1.3. Morphological analysis

Fig. 2 shows typical images of the JA particles. JA particles are formed by two kinds of constituent elements, pith and bark that present a morphological structure as a honeycomb and that can be associated to the porosity of the JA particles.

According to Fiorelli et al. (2017), these characteristics in the vegetal material morphology influence the bonding of the particles during the particleboard production process, as they have a close correlation with the impregnation of the matrix into the vegetal material structure, allowing proper anchorage among particles. Thus, the morphological structure of the JA particles can present an advantageous performance in the production of CBJAP. The cementitious matrix (PC) during composite production in its fresh phase can fill in the porosity of the JA particles and create proper anchorage between the reinforcement phase (JA particles) and the matrix (PC) (Bentur and Mindess, 2007).

3.2. Inhibitory index

Inhibitory index of JA particles into PC was 6% (Table 4). According to Okino et al. (2004), vegetal materials with inhibitory index values up to or below 10% may be considered as low inhibitors of the PC setting time. Therefore, due to the low inhibitory index, JA particles present compatibility with PC, allowing its utilization as raw material to produce CBJAP.

Cabral et al. (2018a,b) conducted inhibitory index tests using sugarcane bagasse fiber in a PC matrix and achieved a 67.30% index. Papadopoulos (2008) evaluated the inhibitory index of hornbeam wood (*Carpinus betulus* L.) particles to produce cement-bonded particleboards and the index found was 39.15%. The red pine (*Pinus brutia*) wood

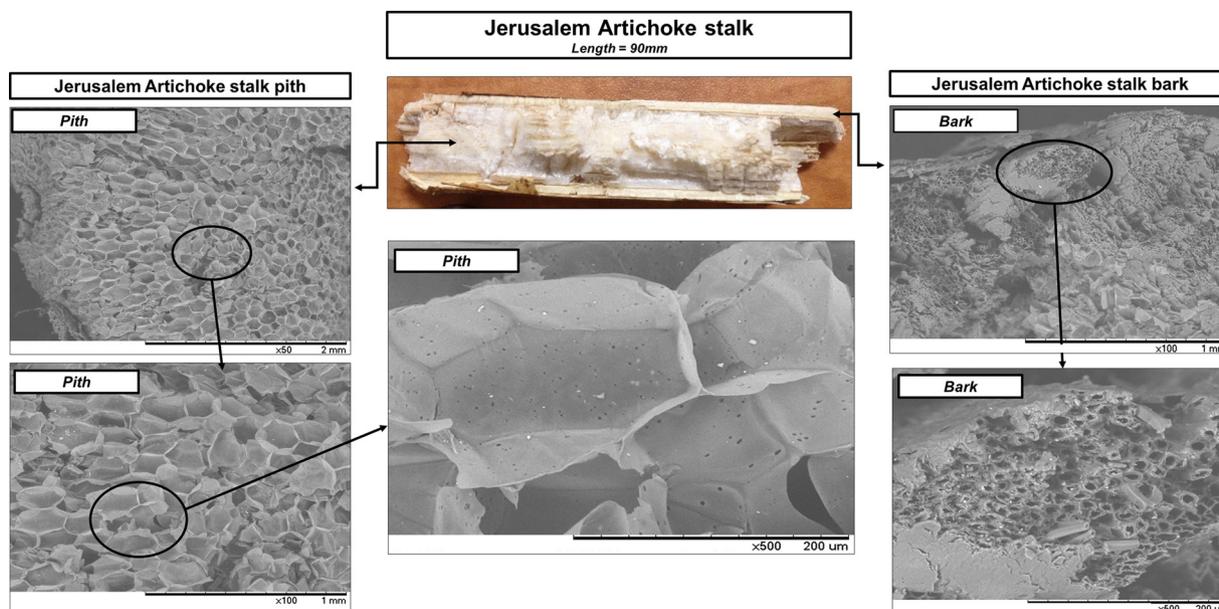


Fig. 2. SEM images of pith and bark of the Jerusalem Artichoke particles.

Table 4
Inhibitory index of Jerusalem Artichoke particles into the PC paste.

Sample	Inhibitory index (%)
PC-Jerusalem Artichoke	6 ^a (0.06)

^aEach value represents the mean of five replicates followed by standard deviation.

inhibitory index was also determined by Papadopoulou (2008) and the hydration test showed that red pine has an inhibitory index of 25.65%. However, the studies with JA particles presented much lower inhibitory index values of around 6%.

The initial setting time for both curves (Fig. 3) begins at the same point (around 6 h). However, the JA particles caused the peak temperature to decrease with a delay in the final setting time due to the interference caused in the C₃A (tricalcium aluminate) hydration process (Taylor, 1997). There was a reduction in the maximum temperature of the PC curve from 85.4 °C in 9 h to 66.2 °C in 12 h for the PC-JA particles. Studies conducted by Dong et al. (2016) found that the dissolution of the extractives from the vegetal materials in the mixing water, even at low contents (1.01% by mass), postponed the clinker hydration process when compared to the plain water.

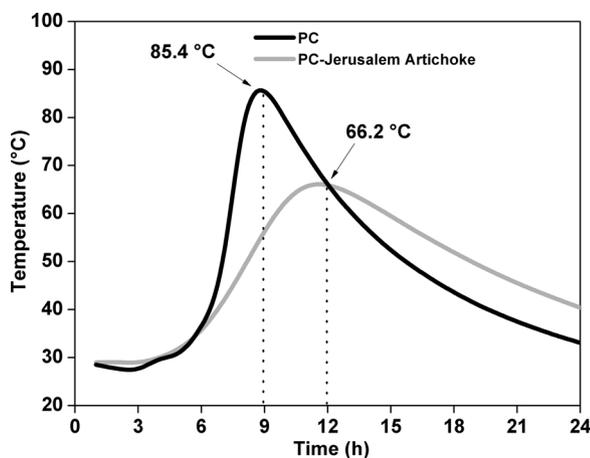


Fig. 3. PC hydration colorimetric test curve of PC and PC-Jerusalem Artichoke.

According to Chakraborty et al. (2013), the delay of the PC setting time occurs due to the dissolution of the extractives present in the vegetal materials. When dissolved, these compounds may form a protective layer on the partially hydrated PC grains, forming a temporary barrier on the cement grains to further hydration (Garci and Jennings, 2002), which leads to a disequilibrium in the formation of portlandite and calcium silicate hydrate (C-S-H). This fact can be observed in the curve of the PC with JA particles (Fig. 3) and contrarily it is not so evidenced in the curve with PC only.

Based on the results achieved for the JA particles characterization by means of the chemical composition, XRD pattern, morphologic analysis and hydration test, it is possible to consider its utilization to produce PC composites, thus indicating its viability as a constituent for CBJAP.

3.3. CBJAP evaluation

3.3.1. Analytical analysis

The phenolphthalein indicator of the control and carbonated CBJAP with 2% phenolphthalein solution is presented in Fig. 4. Control CBJAP (Fig. 4A) shown violet coloration after the visual assessment with 2%. The high color intensity of the PC materials after analysis by phenolphthalein (Fig. 4A) is given as a result of matrix pH between 10 and 14, this fact can be explained by the presence of portlandite dissolved in the aqueous medium (Hewlett, 2004).

On the other hand, carbonated CBJAP did not show a violet color after spraying the phenolphthalein indicator (Fig. 4B), demonstrating that the carbonation process reduces the amount of free hydroxyl groups of the portlandite which is the alkaline source in PC environment. This occurred because the carbon dioxide reaction with the portlandite dissolved in the solution resulted in the formation of calcium carbonate. Regarding particle degradation, this chemical modification may result in an improvement of JA particles durability, since the alkaline environment of the PC degrades the vegetal materials and causes loss of mechanical properties over time.

In addition, in the CBJAP the porosity of the composites caused by high JA particles concentration contributed to the improvement of the accelerated carbonation curing procedure, making the process more efficient and homogeneous throughout the CBJAP composite (Fig. 4.B).

Fig. 5 plots TG-DTG of the CBJAP specimens of control curing and after accelerated carbonation (28 days age).

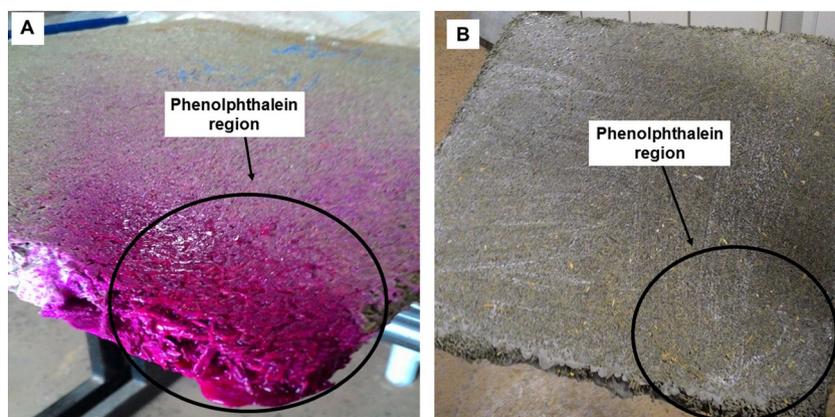


Fig. 4. Phenolphthalein titration of the CBJAP: (A) control (B) carbonated.

The changes in mass loss between 90 °C and 200 °C (Fig. 5a) corresponds to the calcium silicate hydrate (CSH), ettringite (Aft), monosulfoaluminate (AFm) and monocarboaluminate (Mc) (Taylor, 1997; Pipilikaki et al., 2008). Bentur and Mindess (2007) states that the thermal decomposition of vegetal materials in PC composites can be detected in the temperature interval between 295 °C and 370 °C. However, for the JA particles in cement-bonded particleboards specimens, the thermal decomposition was detected in the temperature interval between 250 °C and 350 °C (Fig. 5a), corroborating with the TG-DTG results for JA particles of Fig. 5b. As shown in Fig. 5b for the JA particles, two main peaks are presented, around 100 °C is attributed mainly to the JA particles' moisture and in the temperature range from 200 °C to 450 °C there is the thermal decomposition of hemicelluloses, cellulose and lignin components of the JA particles (Megiatto et al., 2008).

As mentioned, the vegetal material has low durability when exposed in the alkaline medium of the cement (pH ~ 12), i.e. the vegetal particles are decomposed by alkaline attack. According to Sedan et al. (2007) alkaline solutions lead to the removal of amorphous compounds from the vegetal particles surface like hemicelluloses and lignin.

Accelerated carbonation promotes a decreasing of the cement pH (ranging from 12 to 8), and consequently reducing the alkaline attack on the vegetal particles (Almeida et al., 2013; Santos et al., 2015). Such effect is seen by the peak around 300 °C in the DTG graph presented in

Fig. 5a. Compared to control, the mass loss in this peak for the carbonated CBJAP is higher, which means that the accelerated carbonation arrested the alkaline deterioration of JA particles.

In the temperature range from 400 °C to 500 °C (Fig. 5) there is the thermal decomposition of the portlandite. The peak from 650 °C to 850 °C (Fig. 5) refers to the thermal decomposition of calcium carbonate. The thermal decomposition results obtained from the samples extracted from the CBJAP are in line with the results obtained from studies conducted by Almeida et al. (2013), Borges et al. (2010), Rostami et al. (2012) and Taylor (1997), regarding the PC hydration products.

The portlandite and calcium carbonate content of the CBJAP was estimated using the mass loss rates (Fig. 5) obtained from the TG curves, as proposed by Borges et al. (2010) and Santos et al. (2015). The carbonated CBJAP presented 29.3% calcium carbonate and 6.2% portlandite, on the other hand the control CBJAP showed 19.7% calcium carbonate and 10.7% portlandite.

The carbonation process occurs naturally in PC materials, this reaction will occur over the lifetime of PC materials (Neves et al., 2013; Rozière et al., 2009; Sanjuan et al., 2003). Nevertheless, the natural carbonation process can be facilitated because of the voids and porosity present in the CBJAP as observed by the SEM analysis (Fig. 7). Additionally, studies conducted by Govin et al. (2006) showed that the alkaline degradation of the vegetal materials may induce the internal

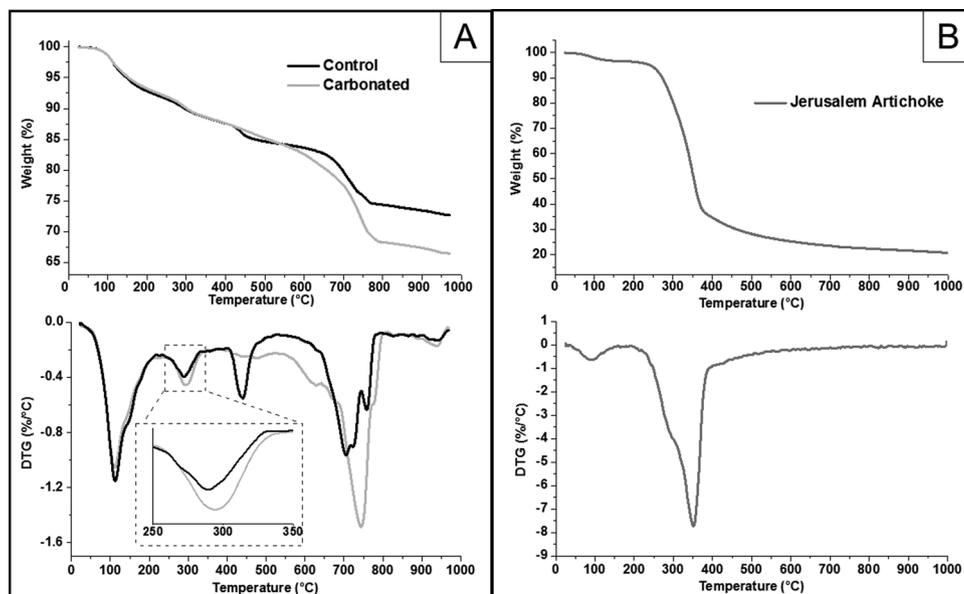


Fig. 5. TG-DTG of the CBJAP specimens of control curing and after fast carbonation (A) and JA particles (B).

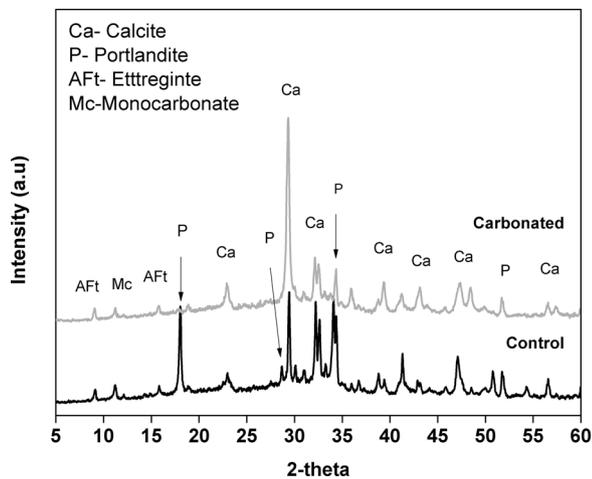


Fig. 6. XRD of CBJAP.

release of carbon dioxide, facilitating the natural carbonation of PC composites.

Fig. 6. presents the XRD patterns of samples taken from the control and carbonated CBJAP (28 days). In both CBJAP, the presence of portlandite and carbonates can be identified. In the samples from the carbonated CBJAP, carbonate peaks are noted and the absence of portlandite peaks can be verified when compared with the PC. These results indicate absorption of dioxide carbon by the PC, a fact also noted by Almeida et al. (2013) and Tonoli et al. (2013).

Based on the results obtained with the phenolphthalein indicator, TG-DTG and XRD, the effectiveness of the accelerated carbonation process in the PC was confirmed, as was the transformation of portlandite into calcium carbonate, reducing the pH of the PC and making the medium less aggressive to the JA particles.

3.3.2. Microstructural analysis

Fig. 7 presents SEM images of polished samples of control and carbonated CBJAP. From the image obtained from the control sample (Fig. 7A), voids between the matrix and the interface are observed.

The voids in the interface (Fig. 7A) explain the significantly lower values ($p < 0.05$) of MOR and MOE for the control CBJAP. However, in carbonated samples (Fig. 7B), the matrix is more compact and denser, which possibly favored the adhesion between the vegetal particles and the PC matrix. This fact resulted in higher physical and mechanical performance of the composite.

According to Bertos et al. (2004), the main reaction in the accelerated carbonation process corresponds to the consumption of portlandite with carbon dioxide, forming calcium carbonate, which is a more stable and denser product when compared with the portlandite. Such a reaction provides an increase in the volume of material inside

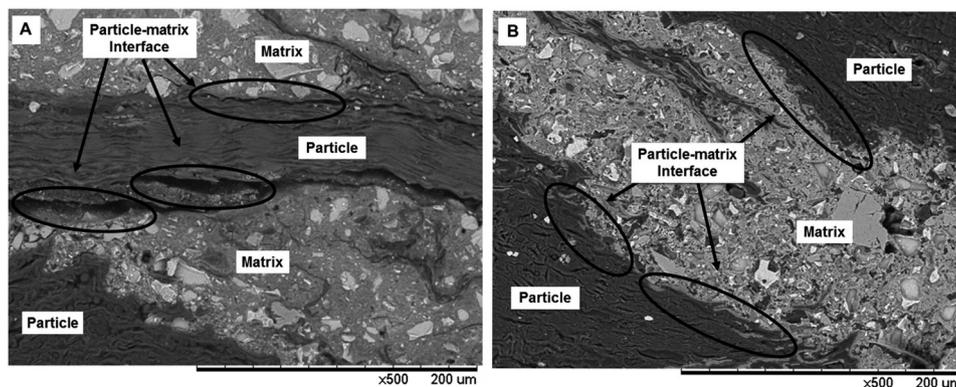


Fig. 7. CBJAP SEM analysis: (A) control; and (B) carbonated.

Table 5

Average values of physical and mechanical properties of CBJAP.

	Physical properties			Mechanical properties		
	WA 24 h (%)	TS 24 h (%)	BD (kg/m ³)	MOR (MPa)	MOE (GPa)	IB (MPa)
Control*	14.80 ^a	1.38 ^a	1163 ^a	9.09 ^a	3.39 ^a	0.4 ^a
Carbonated*	8.86 ^b	0.35 ^b	1256 ^b	11.01 ^b	4.71 ^b	0.6 ^b
ISO 8335 (1987)	–	1.8	1200	9.00	3.00	0.4

*Means with same letter in the column do not differ by the Tukey Test ($p < 0.05$).

the composite, that is, for each mole of portlandite (density 2.21 g/cm³) consumed one mole of calcium carbonate is formatted (density 2.71 g/cm³), which corresponds to an increase of 11.8% volume solids (Bertos et al., 2004). In addition, Bertos et al. (2004) states that carbonate deposition within the pores of the matrix modifies and improves the physical and mechanical properties of the composite.

3.3.3. Physical and mechanical properties

Table 5 presents average results for physical properties of water absorption (WA); thickness swelling (TS) after 24 h of immersion in water, bulk density (BD) and mechanical properties of MOR, MOE and IB of CBJAP evaluated in this research.

The results indicated that WA and TS values for carbonated CBJAP were statistically lower ($p < 0.05$) compared with the values obtained for control CBJAP. Lower values for WA and TS of the carbonated CBJAP were verified when compared to control CBJAP, (40.1% less WA and 74.6% less TS). This behavior can be explained by the lower capillary porosity in the CBJAP after accelerated carbonation curing, which caused the densification of the PC due to the formation of calcium carbonate and resulted in less dimensional variation, reflecting in the values of these physical properties.

The physical properties under study (Table 5) are very close to those for cement-bonded wood particleboards described in the literature (Okino et al., 2004; Ferraz et al., 2011). In addition, the TS results indicated that the dimensional variation of the CBJAP for both curing procedures were lower than that required by ISO 8335 (ISO, 1987), which set standardized values of 1.8% for the TS after immersion in water for 24 h.

The average values of MOR obtained in this study ranged between 9.09 MPa (for control CBJAP) and 11.01 MPa (for carbonated CBJAP). For the MOE result, there was a variation from 3.39 GPa (control CBJAP) to 4.71 GPa (carbonated CBJAP).

The results for the IB ranged between 0.4 MPa (control CBJAP) and 0.6 MPa (carbonated). In fact, it was observed that the control and carbonated CBJAP reached the minimum requirements established by the ISO 8335 (ISO, 1987) to MOR, MOE and IB.

It is important to note that higher mechanical results of MOR, MOE

and IB indicate a better adherence between the PC and JA particles, and this fact may be related to the morphology of the JA particles that have provided the PC filler in the voids of the JA particles and created an adhesiveness between the reinforcement phase (JA particles) and the matrix (PC).

4. Conclusions

The results of this study indicate the potential of Jerusalem Artichoke (*Helianthus tuberosus* L.) stalks to produce cement-bonded Jerusalem Artichoke particleboards (CBJAP) with optimal physical and mechanical performance. The CBJAP met the requirements established by the ISO 8335 Standard for building purposes, what opens perspectives for future industrial utilization. Under both curing conditions (conventional saturated air x CO₂ enriched environment), the values of thickness swelling and water absorption of the particleboards were lower than those reported in the literature and required by the Standards.

Alternative vegetal resources are confirmed to be efficient in the production of low cost and versatile materials for construction applications, especially in regions with local agriculture crop availability. The following conclusions can also be drawn from the results reported herein:

- (1) Accelerated carbonation curing process, after 48 h in a controlled environment, was efficient to favor the formation of carbonate compounds performing as additional binder in the composite. The carbonated CBJAP presented 29.3% calcium carbonate and 6.2% portlandite (by mass). On the other hand, the control CBJAP under conventional saturated air curing showed 19.7% calcium carbonate and 10.7% portlandite.
- (2) Water absorption (WA) and thickness swelling (TS) values decreased by 40% and 75% respectively when the CBJAP passed through the fast carbonation curing.
- (3) The accelerated carbonation increased the modulus of rupture (MOR) from 9.09 MPa to 11.01 MPa, modulus of elasticity (MOE) from 3.38 GPa to 4.71 GPa and internal bond (IB) from 0.4 to 0.6 MPa of the studied CBJAP under flexural solicitation (in comparison to the counterpart cured in saturated air conditions).

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