FOREST SCIENCE (CIÊNCIAS FLORESTAIS)

Grape rachis in composites manufacturing

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ABSTRACT: The study aims to verify the possibility of using grape rachises in the production of composites. Three different proportions of pine wood (*Pinus eliottii*) and grape rachis, glued with urea-formaldehyde adhesive, were used. Assessment of composite quality consisted of the determination of physical properties: moisture content; specific mass; water absorption and thickness swelling after 2 and 24 h of immersion; and mechanical properties: static bending; screw withdrawal and Janka hardness. Histological slides were made for anatomical visualization of particles and analysis in scanning electron microscopy (SEM) to verify adhesion in the composites. Thephysical and mechanical properties were negatively affected by the addition of grape rachis particles. This may be due to a large part of the anatomical constitution of this material to be medullary cells, different from wood particles. General assessments of the results indicate the technical feasibility of using grape rachis for the production of composites.

Key words: physical-mechanical properties; residues; urea-formaldehyde

Engaço da uva na fabricação de compósitos

RESUMO: O presente estudo tem como objetivo verificar a possibilidade de utilização do engaço da uva na fabricação de compósitos. Foram utilizadas três diferentes proporções de madeira de pinus (*Pinus eliottii*) e engaço da uva, colados com adesivo ureia-formaldeído. A avaliação da qualidade dos compósitos compreendeu na determinação das propriedades físicas: teor de umidade; densidade; absorção de água e inchamento em espessura, após 2 e 24 h de imersão e das propriedades mecânicas: flexão estática; arrancamento de parafusos e dureza Janka. Foram confeccionadas lâminas histológicas para visualização anatômica das partículas e análises em microscopia eletrônica de varredura (MEV) para verificação da colagem nos compósitos. As propriedades físicas e mecânicas foram afetadas negativamente pela adição de partículas de engaço da uva. Os resultados podem ser atribuídos ao engaço possuirem sua constituição anatômica grande parte de células medulares, quando comparado às partículas de madeira. Do modo que a pesquisa foi conduzida é inviável o uso de engaço da uva para produção de compósitos.

Palavras-chave: propriedades físico-mecânicas; resíduos; ureia-formaldeído

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Introduction

The wine sector is one of the most important for the Brazilian agricultural economy, providing livelihood for a number of small grape farms. Although present in various states and regions, it is especially significant in Southern Brazil Region, where much of the production is intended for agribusiness of juice and wine. Its activity encompasses an area of approximately 79,000 hectares, with an average annual production of 703,271,388 kg of grapes and 247,457,541 L of wine, which is about 58% of the national production, making it one of the most important crops in the country (Mello, 2015).

A bunch of grapes consists of two parts, an herbaceous part, called the rachis and another fleshy part, called the berry or grain. The rachis is the first by-product obtained in the winemaking process. It is rich in water, woody matter, resins, minerals and tannins and considered a very important constituent of the grapevine, because it determines the structure of the bunch, making up about 3 to 9% of its total weight. It consists of a main shaft that is connected to the stalk and smaller branches, which support the berries and are responsible for providing them with water and mineral salts (Mendes et al., 2014). The rachis is separated in the process of winemaking by a device coupled to the crusher, called the stemmer (Prozil et al., 2012).

The grape industry is widely diversified for the processing of different products, including wine, juices, jellies, jams and raisins, as well as applications in other areas, such as in the production of beauty products. However, residues are generated during processing which mostly end up being discarded, without an appropriate destination, wasting resources that could have a use in the development of value added by-products. Residues from wineries are not considered dangerous, but due to the high content of organic matter and the seasonal production, they may contribute to pollution problems (Spigno et al., 2008).

According to what has already been exposed, the treatment of wine-making by-products needs attention, be it with a view to their subsequent use, or exclusively due to environmental concerns. Currently, grape rachis applications are limited, essentially, to use as a fertilizer or animal food. However, when used in animal feed, its digestibility is low (Schurg et al., 1980). Chemical composition studies have shown that the rachis presents strong potential in applications in the paper and biocomposites industries (Ping et al., 2011). Thus, the aim of this study is to assess the potential use of grape rachis, as a residue to replace raw wood material in the production of composites.

Material and Methods

Pine wood particles (*Pinus eliottii*) and grape rachises were used to make the composites. The wood came from homogeneous stands, located at coordinates 20° 43 ' 04 '' S and 53 43 ' 35 '' W. Logs (1.30 m) were transformed into chips, while grape rachises, derived from *Vitis* spp. varieties, known as "common grapes", were collected after stemming at a small rural property, located at coordinates 26° 40 ' 19.18 '' S and 53 ' 41 '' W 35.37.

The raw materials were dried at room temperature and ground in a hammer mill equipped with a sieve with 5.0 mm holes. For the determination of particle geometry, 100 samples were randomly selected to measure parameters such as length, width and thickness.

Then, wood particles and grape rachis were placed in an acclimatized chamber, with constant temperature of 20± 2°C and relative humidity of 65 ± 3%. After conditioning, the samples were removed for determination of the moisture content. Before starting the manufacture of composites, the raw material was placed in an oven at 60°± 2°C, for 24 hours, to reach a moisture content of approximately 3%.

A urea-formaldehyde-based (UF) commercial adhesive was used to make the composites. The treatments consisted of three different ratios of wood and rachis: 100% wood; 50% wood and 50% rachis; and 100% rachis, with three replicates each, totaling nine composites, with dimensions of 40 x 40 x 0.95 cm as described in Table 1.

The composition of all treatments presented 89% particles, 9% adhesive, 1% catalyst and 1% paraffin, with a specific mass of 0.7 g cm^{-3}. The predetermination of the specific mass was based on the dry weight of the particles and the content of solids in the adhesive, catalyst and paraffin employed. The parameters used in the production were: pressing force 30 kgf cm^{-2}; temperature of 180 °C; moisture content of 8%; pressing time of 10 minutes and 40-second press lock.

The particles were mixed with adhesive, catalyst and paraffin in a rotating drum. These constituents were applied using a gun powered by air compressor, with 8 kgf cm^{-2} of pressure and flow of 50 g min^{-1}. The composites were formed manually in a wooden box with dimensions of 40 x 40 x 20 cm and pre-pressed in a manual press at room temperature for approximately one minute. After cold pressing, the composites were removed from the box and placed in a hydraulic press with flat horizontal plates and electric heating.

Assessment of composite quality consisted of the determination of physical properties: moisture content; specific mass; water absorption and thickness swelling after 2 and 24 hours of immersion; and the mechanical properties: static bending; screw withdrawal and Janka hardness,

Table 1. Composites by treatment, replicate and proportion of wood and grape rachis.

Treatments	Type of adhesive	Replicates	Particles (%)	
			Wood	Grape rachis
Wood		3	100	0
Wood and grape rachis	urea-formaldehyde-based	3	50	50
Grape rachis		3	0	100

adopting the recommendations of the American Standard Testing and Materials - ASTM D1037-12 (ASTM, 2012).

Histological slides were created for anatomical visualization of wood and grape rachis. Anatomical cuts were performed on the cross section of samples and slides were assembled according to the standard technique, recommended by Burger & Richter (1991). The photomicrographs of the anatomical structure were taken in a microscope, with 4 x/0.10 flat lenses, equipped with an digital camera.

For a more accurate evaluation of differences among treatments, scanning electron microscope (SEM) analyses were performed. The samples, removed manually in the transverse direction and on the inner surface of composites, were covered by a thin layer of gold. The micrographs were taken in a scanning microscope.

The data of the physical-mechanical properties were evaluated using a completely randomized design with three treatments and three replicates. The data presented a normal distribution according to the Shapiro-Wilk test. The analysis of variance of the means was performed and the null hypothesis was rejected, showing that there was a significant difference between the treatments. The mean values of the properties were verified using the Tukey test at 5% probability of error.

Results and Discussion

The wood and rachis particle size distribution showed significant differences for the variables length, width, thickness and volume. There were similarities only for the slenderness ratio, as can be seen in Table 2.

The different dimensional characteristics of particles indicate difficulty to control moisture during formation of the composites and, consequently, the amount of adhesive. When particle dimensions are different, the specific surface area and the availability of adhesive per unit of area undergo alterations.

In relation to slenderness ratio values available in the literature for composites of wood particles, Hua et al. (2015) obtained 72 for rubberwood and 122 oil palm trunkaverage and Baldin et al. (2016) obtained 7.64 and 7.54 for capim-annoni (*Eragrostis plana*) and *Pinus* sp., respectively. The discrepancy in the values was attributed to use of different species, types of equipment for particle generation, desired thickness, and compatibility with the final product.

Considering the mean values of water absorption and thickness swelling (Table 3) at 2 hours of immersion, the grape

Table 3. Mean values of water absorption and thickness swelling in different treatments.

Treatments	Water absorption (%)		Thickness swelling (%)	
	2 h	24 h	2 h	24 h
Wood	18.87 [b]	47.17 [a]	12.50 [b]	31.15 [a]
Wood and grape rachis	18.36 [b]	43.42 [b]	13.12 [b]	29.98 [ab]
Grape rachis	32.61 [a]	42.08 [b]	17.71 [a]	27.73 [b]

Means followed by the same letter in the column do not differ by Tukey's test (p > 0.05).

rachis composites presented greater dimensional instability. Later, there was a stabilization and, after 24 of immersion, the wood composites presented greater absorption values.

This may be due to the lignocellulose content of the rachis, with 30-31% cellulose, 21% hemicellulose, 17-18% lignin, 15-16% tannins and about 6.0% protein (Prozil et al., 2013). The low percentage of holocellulose in the rachis when compared to pine wood, of approximately 70% (Protásio et al., 2015), confers to it hydrophilic characteristics.

The standards commonly used to asses composite quality, such as Brazilian Regulatory Standard -NBR 14810, Brazilian Association of Technical Standards – ABNT (2013), Commercial Standard - CS 236-66 (1968), American National Standards Institute - ANSI A280.1 (2009) and European Norm - EN 312 (2003) do not establish a benchmark for water absorption. However, the mean value obtained in this study is lower than the reported by Ntalos & Grigoriou (2002) which was 71.4% and 79.5%, for composites of grape prunings and wood in proportions of 50:50 and 100:0, respectively, after 24 hours of immersion (Figure 1).

All composites met the requirements of the CS-236-66 (1968) for thickness swelling after 24 hours, which establishes maximum values of 35%, indicating that their
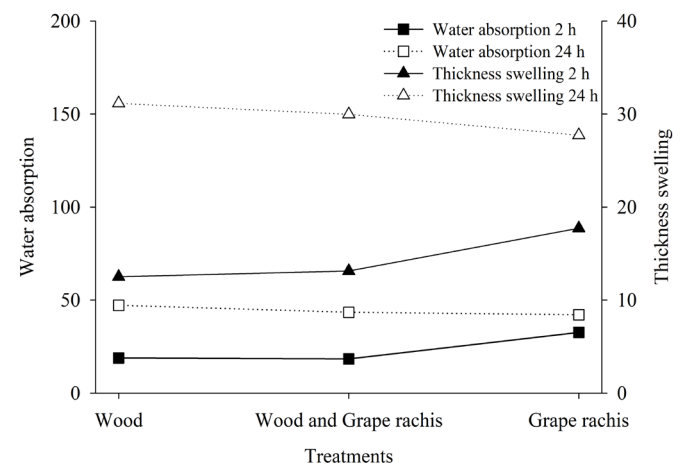

Figure 1. Comparison of the values of absorption and thickness swelling in different treatments.

Table 2. Mean values of characteristics of wood and grape rachis particles.

Type of particle	Len. (mm)	Thick. (mm)	Wid. (mm)	Vol. (mm³)	Slen.	Flat.
Wood	14.62 [a]	2.15 [b]	0.53 [b]	17.72 [b]	7.58 [a]	1.23 [b]
Grape rachis	8.92 [b]	1.46 [a]	1.20 [a]	16.36 [a]	10.70 [a]	1.80 [a]

Len. – length,Thick. – thickness, Wid. – Width, Vol. – Volume, Slen. – slenderness ratio, Flat. – flatness (relation between width/:thickness).Means followed by the same letter in the column do not differ by Tukey's test (p > 0.05).

constituent particles are adhered well enough to withstand immersion in water.

For variable specific mass (Table 4), differences between the means of the experimental composites were identified, with higher values in the wood and rachis treatment and lower values in the rachis treatment. The results did not coincide with the pre-established specific mass value for the composites (0.7 g cm⁻³). This behavior may be related to the differences in particle geometry, to the moisture content and to the lack of particle distribution uniformity in the composites.

The heterogeneity in the specific mass values is undesirable and indicates possible weaknesses in the subsequent assessments of mechanical properties of the composites, since Melo et al. (2010) reported high correlation coefficients between specific mass and mechanical properties.

The NBR 14810, ABNT (2013) specifies that the mean density can vary from 0.551 to 0.750 g m⁻³.Thus, it can be seen that only the rachis composite did not meet the regulatory requirements regarding specific mass.

Moisture content (Table 4) presented significant differences between means, with higher means for rachis and lower values in the other treatments. The percentages of moisture content were outside the range proposed by NBR 14810, ABNT (2013), of between 5 to 11%. The values of moisture found in this study are unacceptable for industrial use of the composites, despite having incorporated paraffin, which should have made the material less reactive to water.

For the mechanical property of Janka hardness (Table 4), there was no statistical difference between the three treatments. In contrast, hardness did not change when residues of yerba mate (*Ilex paraguariensis*) were added to *Pinus caribaea* var. *hondurensis* panels (Carvalho et al., 2015) and in Soratto et al. (2013) when the addition of up to 6% of chips with bark in the composition of the panel does not cause impairment of quality. The CS-236-66 (1968) establishes 22.7 MPa as the minimum Janka hardness. In this context, the mean value obtained from the composite made solely of wood was the only one to meet this requirement.

For the modulus of rupture (MOR) (Table 4) the best value was observed in the wood composite (71.52 MPa). However, it can be seen that addition of 50% of rachis did not affect the quality of the composites. Ntalos & Grigoriou (2002) using vine prunings for the production of composites, found MOR values to be much lower, with panels with 100%, 50% and 0% of vine residues obtaining values of 8.5 MPa, 14 MPa and 19.1 MPa, respectively. Trianoski et al. (2016)

studies the potential use *Grevillea robusta* and *Pinus taeda* for the particleboards production, the values MOR were lower than found in this study.

The mean values of modulus of elasticity (MOE) (Table 4) in the treatments containing rachis (677.7 and 448.3 MPa) were much lower than those found in the wood composite (1234.4 MPa). This may be due to the constitution of rachis, which presents low mechanical resistance and bonding strength between the particles, due to its geometry. It also presents low density, resulting in the need for a greater number of particles to achieve the pre-established composite density and, consequently, a reduction in the quantity of adhesive per particle, leading to a reduction in the mechanical properties. Similar MOE results have been reported with other agricultural residues, such as peanut husks (Gatani et al., 2013) , sugarcane bagasse (Liao et al., 2016) and coconut residues (Machado et al., 2017).

For the MOR static bending tests, all treatments presented commercialization values in accordance with ANSI A280.1 (2009) and NBR 14810, ABNT(2013), of 12.8 and 11 MPa. respectively. However, for the MOE bending tests, no treatment met the standard requirements, with minimum values of 1600 MPa and 1943.8 MPa.

In the screw withdrawal test (Table 4), there was no significant difference between treatments. Greater resistance was observed in the composite with solely wood in its composition. Ntalos & Grigoriou (2002) using residues of vine pruning for the production of composites, found values higher than those of this study for screw withdrawal force, of 922 N, 920 N and 837 N, in wood, wood and vine and only vine, respectively. Screw withdrawal resistance was very low, with all treatments presenting values below the minimum set by NBR 14810, ABNT(2013), which requires values above 1,020 N. Although the wood composite gave the best results, it also did not meet commercialization specifications.

MOR and MOE mechanical properties were the most negatively affected by the addition of grape rachis particles. This may be due to the fact that a large part of the anatomical constitution of this material is medullary cells, different from wood particles (Figure 2). The detrimental influence of medulla on composite properties has been reported in similar studies (Grigoriou & Ntalos, 2001).

The scanning electron microscopy (Figure 3) confirmed the physical and mechanical behavior of the treatments. The composites made from wood presented better particle distribution, while those made of wood and rachis or only

Table 4. Mean values for physical and mechanical properties of composites.

Treatments	SM (g cm⁻³)	MC (%)	JH	MOR	MOE	SW (N)
				(MPa)		
Wood	0.61 ab*	11.90 b	26.57 a	71.52 a	1234.4 a	321.95 a
Wood and grape rachis	0.66 a	12.35 b	20.83 a	57.11 ab	677.7 b	218.98 ab
Grape rachis	0.52 b	13.27 a	19.10 a	48.12 b	448.3 b	176.52 b

SM - Specific mass, MC - Moisture content, JH - Janka hardness, MOR - Modulus of rupture, MOE - Modulus of elasticity, SW - Screw withdrawal. Means followed by the same letter in the column do not differ by Tukey's test (p ≥ 0.05).

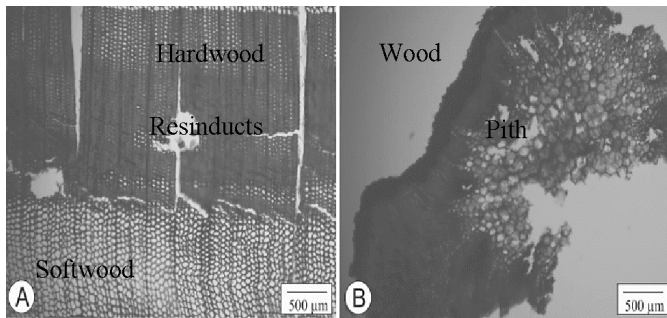**Figure 2.** Cross section of particles used in the manufacture of composites. (A) Springwood (*Pinus eliottii*); (B) Grape rachis.
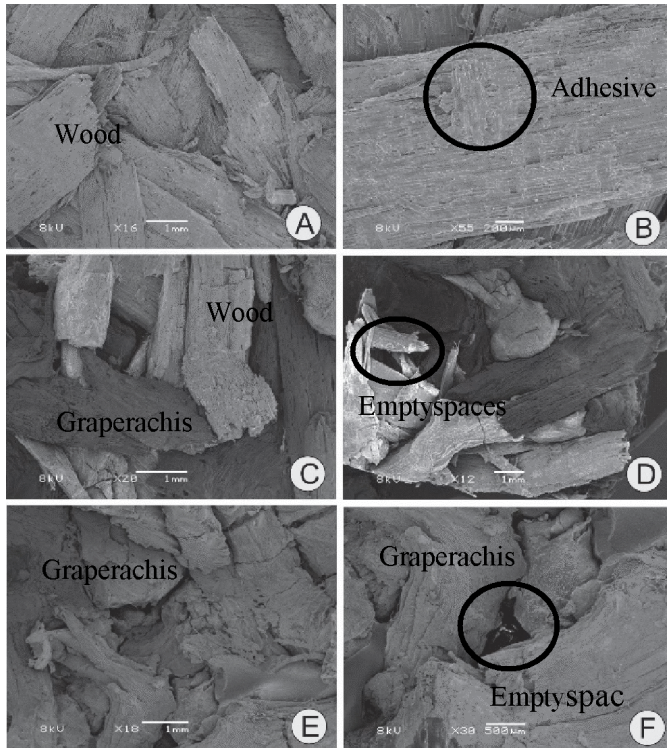

Figure 3. The scanning electron microscopy of composites. (A) wood particles; (B) wood composite showing de adhesive region; (C) composites of wood and grape rachis with heterogeneous matrix; (D) empty spaces in composites of wood and grape rachis; (E) thickness of grape rachis particles resulting in deficiencies in composites; (F) empty spaces in composites of grape rachis.

rachis presented gaps in certain regions, which partially explains the deficiency of these treatments.

The geometry of the rachis and wood particles, when grouped together, provided poor adhesion, resulting in composites with heterogeneous matrices (Figure 3 C, D). A greater incidence of gaps can be seen in composites with rachis (Figure 3 D, F), due to the lack of particle organization, caused mainly by its thickness.

In future studies, to produce composites with improved features, it is recommended that the particles be reduced in size and that the composite present increased specific mass, with the incorporation of small amounts of grape rachis, in proportions of 5%, 10% and 15%, for example.

Conclusions

The addition of grape rachis to *Pinus eliottii* wood composites led to a significant increase in the properties of water absorption and thickness swelling at 24 hours, moisture content and specific mass.

The incorporation of up to 50% of grape rachis particles provided improvement in the properties of Janka hardness and screw withdrawal, however, the incorporation of 100% rachis led to a decrease in these properties.

The autors recommend in future test to use others proportions, for example 25% rachis and 75% wood, which could provide better results.

General assessments of the results indicate the technical feasibility of using grape rachis for the production of composites. However, based on this study, these composites are not recommended as a structural material for the furniture industry.

Acknowledgments

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