PANELS MADE FROM GIANT REED BONDED WITH NON-MODIFIED STARCHES

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Panels were made from *Arundo donax* L. particles bonded with different non-modified starches as adhesive without chemical additives by hot-pressing at a low temperature (110 ºC) and pressure (2.6 N/mm^2). The experimental panels were tested for their physical and mechanical properties according to the procedures defined by the European Union (EN) Standard. The microstructure of samples was observed by scanning electron microscopy (SEM). Panels manufactured with potato starch had the highest modulus of rupture and modulus of elasticity, meeting the standard for load bearing (grade P4 for indoor use in dry ambient) (EN 312: 2003). Panels made with corn starch and wheat flour, at a 10% level and three pressing cycles met the standard for general uses (grade P1). Panel bonded with rye bran flour achieved the best internal bond strentgh. The water resistance was poor and needs to be improved.

Keywords: Giant reed; SEM; Eco-friendly particleboard; Formaldehyde-free; Starch; Microstructure; Silica bodies

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INTRODUCTION

Particleboard is a composite product manufactured under elevated pressure and temperature from particles of wood or other lignocellulosic fibrous materials and a binder (EN 309, 2005). Particleboard is widely used in furniture, where it is typically overlaid with other materials for decorative purposes. It is the predominant material used in ready-to-assemble furniture, flooring systems, manufactured houses, and underlayment. Since most applications are interior, particleboard is usually bonded with a urea-formaldehyde (UF) resin (Stark *et al.* 2010). However, UF adhesive can release low concentrations of formaldehyde gas from bonded wood-based products. When the products are new, high indoor temperatures or humidity can cause increased release of formaldehyde. In the European Union, formaldehyde is considered a high-priority pollutant. Therefore, there is much interest in developing more environmentally friendly adhesives. Many researchers have investigated the use of natural polymers obtained from plants and animals such as starch, proteins, lignins, tanins, *etc*. (Imam *et al.* 2001; El-Wakil *et al.* 2007; Ciannamea *et al.* 2010; Moubarik *et al.* 2010; Wang *et al.* 2011; Treusch and Petutschnigg 2012).

Starch is a relatively inexpensive and renewable product from plants (Kennedy 1989). Annual starch production from cereals is approximately 2050 million tonnes, and from roots and tubers, approximately 679 million tonnes (Tester and Karkalas 2002; Burrell 2003). In addition to being the main source of energy in the human diet, starch is also used for a wide variety of industrial processes: as an adhesive in paper making, as additive in cement, and as a binder in gypsum plaster and gypsum fiber board (Burrell 2003). It is also used as natural filler in traditional plastics, and more recently, as a main component in polymer blends, and composites in the form of thermoplastic starch (TPS) (Kaseem *et al.* 2012). However, the bonding capacity of native starches has been characterized as not being strong enough to glue wood (Imam *et al.* 2001). To solve this problem, physical, chemical, and/or enzymatic modifications have been considered (Kennedy 1989; Singh *et al.* 2010). The chemical modifications are usually alkali or acid treatments (Stofko 1982; Moubarik *et al.* 2010; Wang *et al.* 2011) or oxidation (Singh *et al.* 2010). Physical modifications are produced by cooking, extrusion, spray drying, and annealing, *etc*.

The two major components of starch are amylose and amylopectin. These two molecules are assembled together to form semi-crystalline granules. The proportions of the two molecules and the size and shape of the granule vary between species. The granules must be opened through processing to obtain adhesive bonding. When native starch granules are heated in water, they are gradually disrupted, resulting in phase transition from an ordered granular structure into a disordered state in water, which is known as "gelatinization" (Lelievre 1974; Atwell *et al*. 1988; Ratnayake and Jackson 2008; Xie *et al*. 2012). Full gelatinization of starch under shearless conditions requires an excess of water (Xie *et al*. 2012). If the water concentration is limited, the complete gelatinization will not occur in the usual temperature range. But if the temperature is increased, the crystalline regions will be destructured and will eventually melt (Donovan 1979).

The gelatinization/melting behavior of starch is different when the granules are subjected to shear treatment. For example, in extrusion processing, shear forces can physically tear apart the starch granules, allowing faster transfer of water into the interior molecules (Burros *et al*. 1987; Xie *et al*. 2012).

The objective of this study was to manufacture particleboards from giant reed (*Arundo donax* L.) particles as a low-cost lignocellulosic substrate and adhesives based on non-modified starches and cereal flours. The hypothesis to be proven is that the adhesive capability of native starch for bonding lignocellulosic materials can be enhanced if gelatinization/melting of starch is produced during the hot-pressing process. The performance of such panels was evaluated following the procedures defined by the European Union (EN) Standard. The bonding mechanism was observed by scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

Giant reed culms (*Arundo donax* L.) were purchased from a commercial factory in Alicante (Spain) and were dried under ambient conditions for 12 months until reaching 8% moisture content before use. The culms were manually cut into slices (*ca.* 40 cm long) and chipped in a laboratory scale ring knife chipper equipped with a screen of 10 mm openings. The particles were then classified using a horizontal screen shaker. The particles used for this study were the fines that passed through a sieve of 0.25 mm. The particles did not undergo a previous treatment.

As the adhesive, different commercial-grade cereal flours (rice flour, bran rye flour, and wheat flour) and unmodified commercial-grade starches (corn and potato) were used at 5 and 10% level based on the weight of particles (with an 8% moisture content). No other additives or chemicals were used.

Methods

Seventeen types of panels were made. Pre-weighed raw material was placed into a laboratory drum glue blender (Model LGB 100; IMAL S.r.l., Modena, Italy). The adhesive was mixed with 20% water (based on the weight of particles) at 20 ºC, obtaining a suspension, and then added to the blender. The mixture was blended for 5 min at ambient temperature to obtain a homogenized mixture. No wax or any other hydrophobic substances were used. The mat configuration was single layer. Every panel was made with 2000 g of chips, 100 g or 200 g of adhesive (5% or 10%, respectively), and 400 g of water.

Boards measuring 600 mm x 400 mm were manually formed in a mold and pressed in a hot-press under 2.6 N/mm^2 at 110 ºC for 15 min. After pressing, the boards stayed in the mold while cooling down for 1 hour under ambient conditions. During the cooling down, the pressure was not maintained. After that, the particleboards were brushed with distilled water at a rate of 12 g /1000 cm^2 on the upper surface and then they were hot pressed for a second time under the same pressing conditions. The panels with a 10% level of adhesive were subjected to a third pressing cycle. Two binderless particle-boards were manufactured following the same procedure for comparison. The experi-mental design is shown in Table 1.

Table 1. Manufacturing Conditions of Particleboards

Type	Adhesive	Adhesive content	Pressing cycles	Pressing Temperature (ºC)	Pressing Pressure (N/mm^2)	Pressing time (min)
A1	corn starch	5%	2	110	2.6	15 + 15
A2	corn starch	10%	2	110	2.6	15 + 15
A3	corn starch	10%	3	110	2.6	15 + 15 + 15
B1	rice flour	5%	2	110	2.6	15 + 15
B2	rice flour	10%	2	110	2.6	15 + 15
B3	rice flour	10%	3	110	2.6	15 + 15 + 15
C1	rye bran flour	5%	2	110	2.6	15 + 15
C2	rye bran flour	10%	2	110	2.6	15 + 15
C3	rye bran flour	10%	3	110	2.6	15 + 15 + 15
D1	potato starch	5%	2	110	2.6	15 + 15
D2	potato starch	10%	2	110	2.6	15 + 15
D3	potato starch	10%	3	110	2.6	15 + 15 + 15
E1	wheat flour	5%	2	110	2.6	15 + 15
E2	wheat flour	10%	2	110	2.6	15 + 15
E3	wheat flour	10%	3	110	2.6	15 + 15 + 15
Reed 1	-	-	2	110	2.6	15 + 15
Reed 2	-	-	3	110	2.6	15 + 15 + 15

Two replicate panels were made for each board type. Once finished, the particleboards were conditioned at a temperature of 20 ºC and 65% relative humidity for four days. The finished particleboards were trimmed to avoid edge effects and then cut into various sizes for property evaluation according to EN 326-1 (1999).

Physical and Mechanical Properties

The physico-mechanical properties of particleboard are an indication of quality and suitability in relation to the proposed use of the boards (García Fernández *et al*. 2008). Some physical properties were determined in accordance with the appropriate EN Standards: density (EN 323, 1993), water absorption (WA), and thickness swelling (TS) after 2 and 24-hour immersion (EN 317, 1993). The mechanical properties determined were: modulus of rupture (MOR), modulus of elasticity (MOE) (EN 310, 1993), and internal bond (IB) (EN 319, 1993). Each panel was cut to get six samples for determining density (50 mm x 50 mm), three samples for determining WA/TS (70 mm x 70 mm), six specimens for the measurement of MOR/MOE (different lengths, depending on the thickness, x 50 mm width), and three specimens for the measurement of IB (50 mm x 50 mm). Tests for mechanical properties, WA, TS, and density were conducted on an Imal universal testing machine (Model IB600, Modena, Italy).

Scanning Electron Microscopy (SEM)

The microstructure of the particleboards of the giant reed and the interfacial bonding of the experimental panels were observed using a Hitachi S3000N microscope equipped with an X-ray detector for microanalysis (EDS), model Bruker XFlash 3001.

Statistical Analyses

Data for each test were statistically analyzed. Analysis of variance (ANOVA) and the *t*-test were used to test ($\alpha = 0.05$) for significant difference between factors and levels. When the ANOVA indicated a significant difference among factors and levels, a comparison of the means was done employing a Tukey HSD and a Duncan's test to identify which groups were significantly different from others at a 95% confidence level.

RESULTS AND DISCUSSION

The panels obtained with only one pressing cycle were not sufficiently bonded, and their edges broke up partially when demolding. These panels were not evaluated. It is obvious that the temperature of the hot press and the water content of the mat did not suffice to disrupt the granules of starch during the first hot-pressing, resulting in particleboards with very poor qualities. The particleboards tested were manufactured with two and three pressing cycles. In these, water was brushed on the surface prior to hot-pressing. According to Kelly (1977), moisture at the mat surface vaporices when the press closes and the resultant steam flows to a cooler region (toward the mat centre) where it condenses. It seems that when this condensed hot water contacts the granules of starch that are under pressure, the gelatinization/melting is produced and the bonding capability is enhanced.

Physical Properties

The results of thickness, density, thickness swelling, and water absorption tests of the produced particleboards are shown in Table 2.

Table 2. Mean Values of Some Physical Properties of Panels

Type	Thickness (mm)	Density (Kg/m^3)	TS (%) 2 h	TS (%) 24 h	WA (%) 24 h
A1	14.24 (1.56)	883.06 (72.88)	25.95 (8.05)	27.03 (8.01)	96.94 (11.97)
A2	13.54 (0.02)	927.94 (17.05)	27.39 (5.88)	38.68 (6.29)	75.39 (0.54)
A3	13.01 (0.92)	929.80 (33.60)	38.18 (1.85)	49.21 (0.10)	71.51 (14.69)
B1	13.56 (1.32)	812.28 (45.20)	25.68 (2.04)	28.23 (1.50)	115.45 (2.44)
B2	13.02 (1.56)	825.66 (26.07)	28.60 (1.65)	42.06 (5.89)	106.75 (14.90)
B3	13.04 (1.96)	830.37 (13.48)	51.46 (0.01)	58.83 (2.40)	98.66 (2.63)
C1	13.78 (0.50)	873.86 (47.79)	27.79 (4.14)	33.12 (7.84)	94.68 (5.23)
C2	13.00 (0.44)	915.30 (11.60)	18.28 (6.63)	18.45 (5.31)	54.92 (6.53)
C3	13.00 (0.02)	932.30 (12.58)	32.69 (12.09)	41.91 (14.45)	78.37 (28.52)
D1	13.04 (0.68)	913.55 (1.77)	42.11 (9.34)	49.22 (8.66)	96.71 (16.15)
D2	13.02 (0.02)	920.76 (7.07)	54.86 (4.98)	62.41 (7.07)	92.53 (7.30)
D3	14.25 (1.56)	914.61 (29.91)	41.24 (0.02)	48.68 (1.25)	108.07 (22.64)
E1	13.58 (1.46)	883.50 (3.53)	39.42 (3.87)	52.80 (13.38)	70.69 (2.34)
E2	14.96 (1.22)	885.55 (76.28)	48.16 (7.65)	66.26 (16.74)	82.37 (3.03)
E3	13.80 (0.76)	897.15 (26.42)	38.72 (12.43)	49.67 (24.53)	86.63 (25.30)
Reed 1	14.72 (0.76)	856.84 (32.99)	57.34 (4.12)	74.54 (6.06)	135.99 (1.68)
Reed 2	14.54 (0.56)	845.69 (16.37)	61.49 (2.43)	80.01 (1.59)	133.73 (4.05)

Values in parentheses are standard deviations.

Thickness swelling

Particleboards should have a maximum thickness swelling value of 15% for 24 h immersion for load bearing (P4 grade EN 312, 2003). Average thickness swelling of the specimens for 2 h immersion ranged from 18.28 to 61.49%. For 24 h immersion, the results lay between 18.45 and 80.01%. There is no minimum value of TS in the standards for general uses and furniture manufacturing in dry ambient (P1 and P2 grades, respectively). None of the panels met the standard value of TS for load bearing (grade P4), but they could be used, if their mechanical properties meet the standards for general uses and indoor fitment. As can be seen in Fig. 1, in general, the TS values increased with increasing the adhesive content. This is due to the high affinity that the starches have for water. The binderless particleboards had the highest TS values. These particleboards were manufactured without adhesives, and the pressure and temperature applied during the compression of the mats were not sufficiently high to produce the self-bonding of the lignocellulosic particles, resulting in particleboards with very poor qualities. The binderless particleboards that have been reported in the literature were produced with steam injection during the hot pressing at pressing temperatures above 175 ºC and pressure around 10 MPa (Panyakaew and Fotios 2011; Umemura *et al.* 2009; Okuda *et al.* 2006; Velásquez *et al.* 2002; Angles *et al.* 1999; Suchsland *et al.* 1987). The panels bonded with bran rye flour had the lowest TS values. This may be due to the presence of lipids in the bran flour. Copeland *et al.* (2009) reported that complexes between amylase and lipids reduce the solubility of starch in water, decreasing the swelling capacity and increasing the gelatinisation temperature.

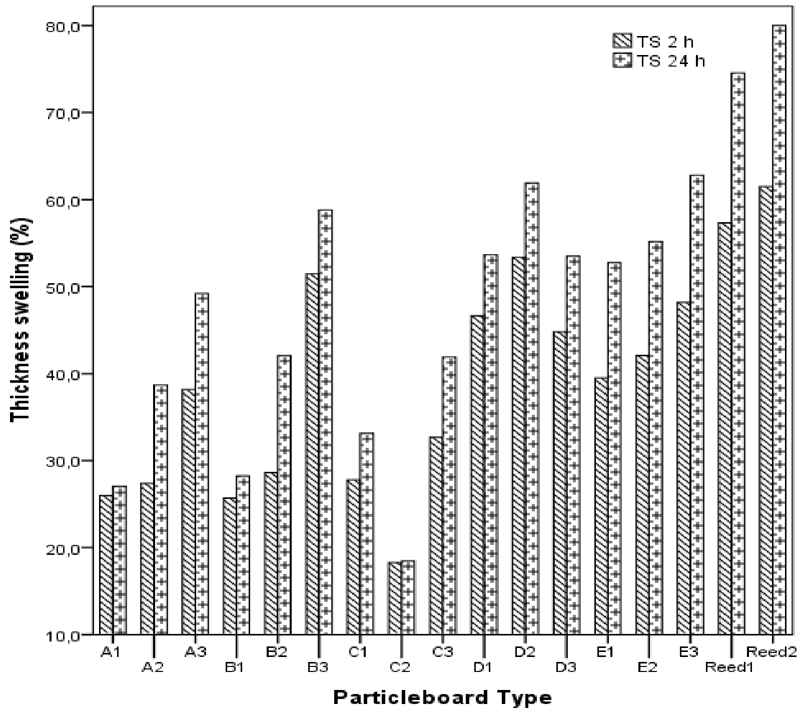


Fig. 1. Average results of thickness swelling (TS) of the produced particleboards.
A: cornstarch; B: rice flour; C: rye bran flour; D: potato starch; E: wheat flour; Reed: binderless.
The minimum TS value for P4 grade (load bearing) is 15%.

Mechanical properties

Based on EN standards (EN 312, 2003), the minimum requirement of MOR for general uses is 11.5 N/mm^2 and an IB value of 0.24 N/mm^2; these are the minimum requirements for general uses in dry ambient (P1 grade). A MOR value of 13 N/mm^2, a MOE value of 1600 N/mm^2, and an IB value of 0.35 N/mm^2 are the minimum requirements for furniture manufacturing (P2 grade). For load bearing (P4 grade), the values of MOR, MOE, and IB are 15 N/mm^2, 2300 N/mm^2, and 0.35 N/mm^2, respectively. The values of MOR ranged from 3.20 to 16.67 N/mm^2.

Samples A3 and E3 (made with 10% corn starch and wheat flour, respectively, and three pressing cycles) had a MOR sufficiently high to meet the requirements for general uses as can be observed in Fig 2. Panel D2 (10% potato starch) exceeded the MOR requirement for indoor fitment (including furniture manufacturing). Panel D3 met the MOR requirement for load bearing. The MOR significantly increased when the adhesive usage was increased from 5% to 10%, independently of the type of adhesive used. The third pressing cycle affected the MOR, improving it for corn starch, potato starch, and wheat flour. The best results were achieved by potato starch, followed by wheat flour and corn starch.

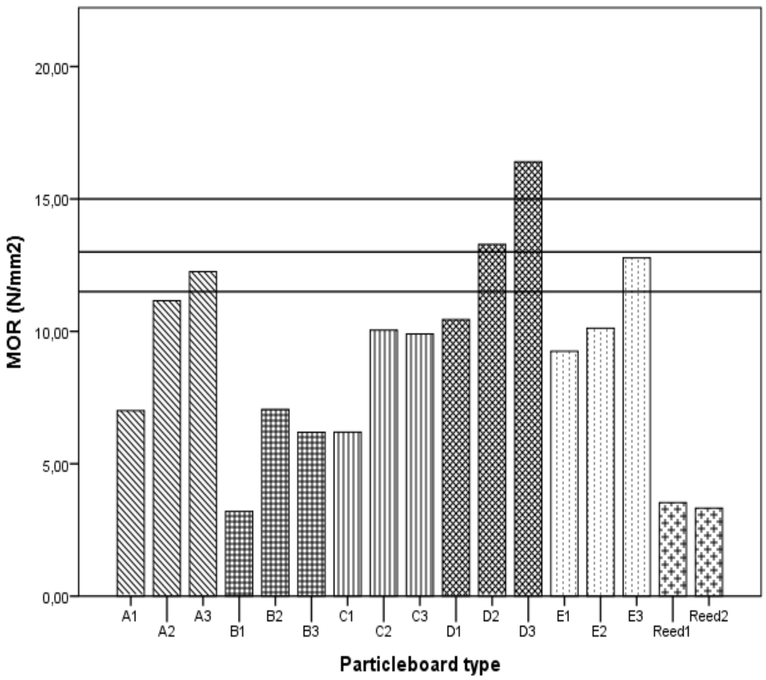


Fig. 2. Average values of modulus of rupture (MOR) of the produced particleboards.
The horizontal lines are the minimum values of MOR: 11.5 N/mm^2 for particleboards for general uses in dry ambient (P1 grade); 13 N/mm^2 for furniture manufacturing (P2 grade); and 15 N/mm^2 for load bearing (P4 grade).
A: corn starch; B: rice flour; C: rye bran flour; D: potato starch; E: wheat flour; Reed: binderless

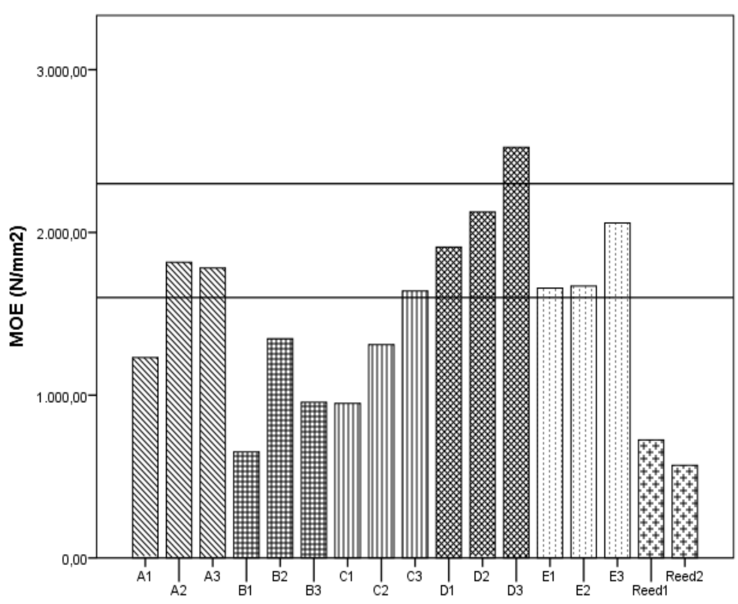


Fig. 3. Average values of modulus of elasticity (MOE) of the particleboards.
The horizontal lines represent the minimum values to meet the standard for P2 and P4 grades: 1600 N/mm^2, indoor applications including furniture manufacture in dry ambients (P2), and 2300 N/mm^2 for load bearing (P4).
A: corn starch; B: rice flour; C: rye bran flour; D: potato starch; E: wheat flour; Reed: binderless

The values of MOE lay between 569.09 and 2520.97 N/mm^2. There is no minimum requirement of MOE for general uses. Particleboards A2, A3, C3, D1, D2, D3, E1, E2, and E3 met the requirements for grade P2 (indoor fitment, including manufacture). Panel D3 exceeded the standard for grade P4 (load bearing in dry ambient). Generally, the MOE significantly increased when the adhesive usage was increased from 5% to 10%, independently of the type of adhesive used. A third pressing cycle influenced the MOE in different ways: improving the panels made with bran rye flour, potato starch, and wheat flour, and decreasing its value for the rest of adhesives and the binderless panels.

The results of IB ranged between 0.04 to 0.40 N/mm^2 (Fig. 4). Panels A2, A3, C2, C3, D1, D2, D3, and E3 met the standard for grade P1 (general uses in dry ambient).

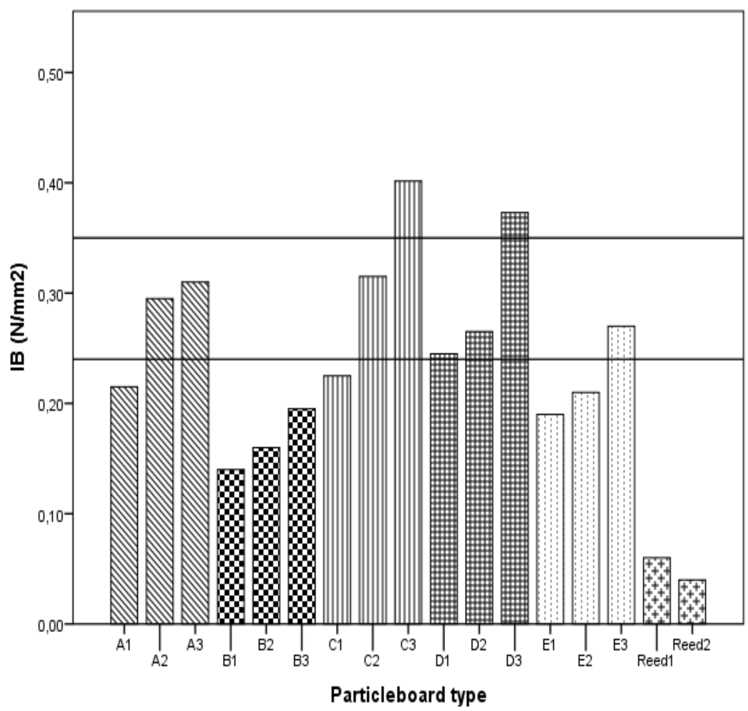


Fig. 4. Average values of internal bond strength (IB) of the particleboards.
The horizontal lines represent the requirements to meet the standard for P1, P2, and P4 grade (P1: 0.24 N/mm^2 for general uses in dry ambient; P2: 0.35 N/mm^2 for indoor fitments including furniture manufacturing; P4: 0.35 N/mm^2 for load bearing).
A: corn starch; B: rice flour; C: rye bran flour; D: potato starch; E: wheat flour; Reed: binderless

Panels C3 and D3 achieved the requirement for grades P2 and P4 (indoor fitment and load bearing, respectively). Panel C3 had the highest IB strength value. The IB was influenced by the level of adhesive used, improving with increasing the level from 5% to 10%. The third pressing cycle had a profound effect on this property, increasing the IB for all the adhesives.

Considering the three mechanical properties studied together, it can be said that particleboards A3 and E3 (made with 10% corn starch and wheat flour, respectively, and three pressing cycles) had a MOR, MOE, and IB sufficiently high to meet the requirements for general uses as can be observed in Figs. 2 and 3. Panel D2 (10% potato starch) exceeded the MOR and MOE requirements for indoor fitment (including furniture manufacturing) but failed to achieve the IB requirement. Panel D3 met the requirements for load bearing (MOR, MOE, and IB).

All this suggests that potato starch and wheat flour are the better adhesives for producing particleboards under these conditions. Potato starch is rich in esterified phosphorus and exhibits higher swelling power and solubility than cereal starches. On the other hand, wheat flour has, beside starch, proteins, which include gluten that has been used as adhesive for particleboard manufacturing (El-Wakil *et al.* 2007). The presence of lipids in the bran rye flour reduced the solubility of starch, resulting in panels with worse mechanical properties. Panels B1, B2, B3, Reed 1, and Reed 2 had the lowest MOR, MOE, and IB values (rice flour and binderless). These panels had the lowest densities, thus suggesting that the density of particleboard plays a very important role on the bending strength as expected. The rice flour may need a higher temperature or pressure for the complete gelatinization/melting of its starch.

In order to improve the general properties of these particleboards, substances such as NaOH and tannins can be added to the adhesives. Tondi *et al.* (2012) demonstrated that adding these substances to starch increased the mechanical properties of experimental particleboard samples.

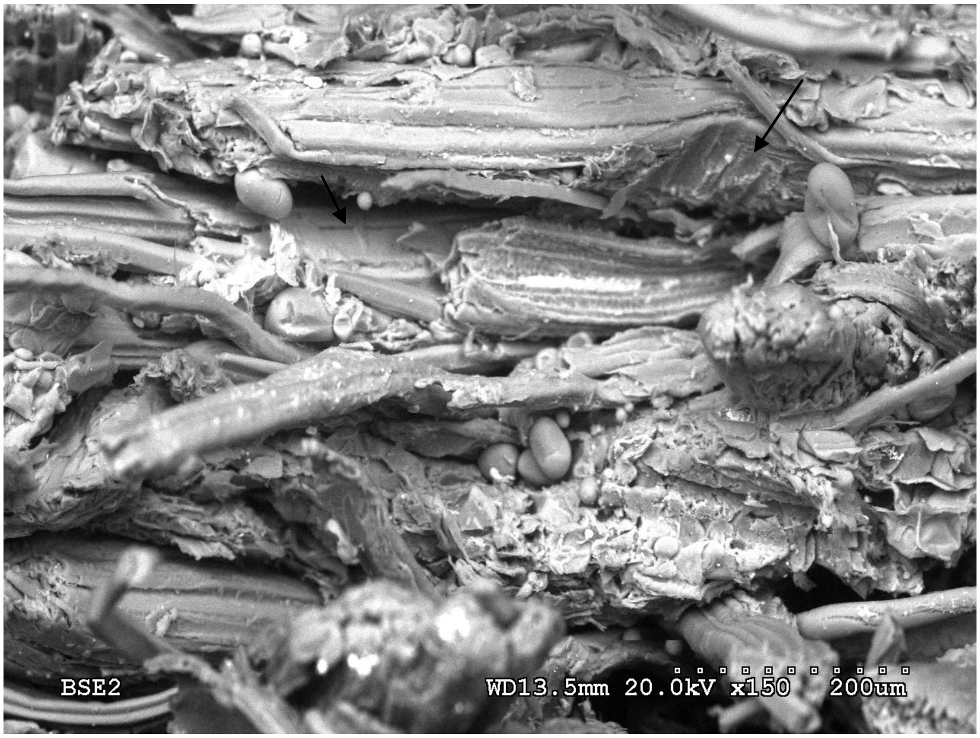
SEM Observations

Pieces of samples from the particleboards tested were fractured and then observed by SEM in order to elucidate the mechanism of bonding. Another panel was made placing the components separated in the mold before the hot-pressing: potato starch in one side and particles on the other side, to view how the starch gelatinized or melted.

Figures 5A and 5B show the fractured surfaces of panels bonded with potato starch at a 5% level and two pressing cycles, and at a 10% level and three pressing cycles, panels D1 and D3, respectively.

- In micrograph A, the granules of native potato starch are evident. Some granules look bigger than the others. Some granules look damaged (see the black arrows). This is due to the thermopressing process. Gaps were also visible, meaning that the consolidation of the mat had not been totally achieved. This is consistent with the results of the mechanical properties.
- In micrograph B, the granules are no longer visible, and there are areas where the starch has been gelatinized, appearing like a polymer matrix (see the white arrows). This particleboard (D3) had better mechanical properties than panel D1. This suggests that the bonding capability of potato starch is enhanced when the gelatinization/melting is produced while in contact with the lignocellulosic particles during the consolidation of the mat in the hot press, after three pressing cycles. In the center of micrograph 5B, a piece of tissue of the outer skin of giant reed stems can be seen. The white spots are silica bodies, also known as phytoliths.

Figure 6 shows a micrograph taken from a fractured piece of the panel manufactured with potato starch as a polymer matrix on one side and particles of giant reed on the other side. It can be seen that the gelatinized/melted starch looks like plastic. The white spots that can be seen here are crystals of potassium chloride.



A

B

Fig. 5. SEM micrographs: (A) fractured surface of particleboard D1 manufactured with 5% of potato starch and two hot-pressings; (B) fractured surface of panel D3 made with 10% of potato starch and three hot-pressings

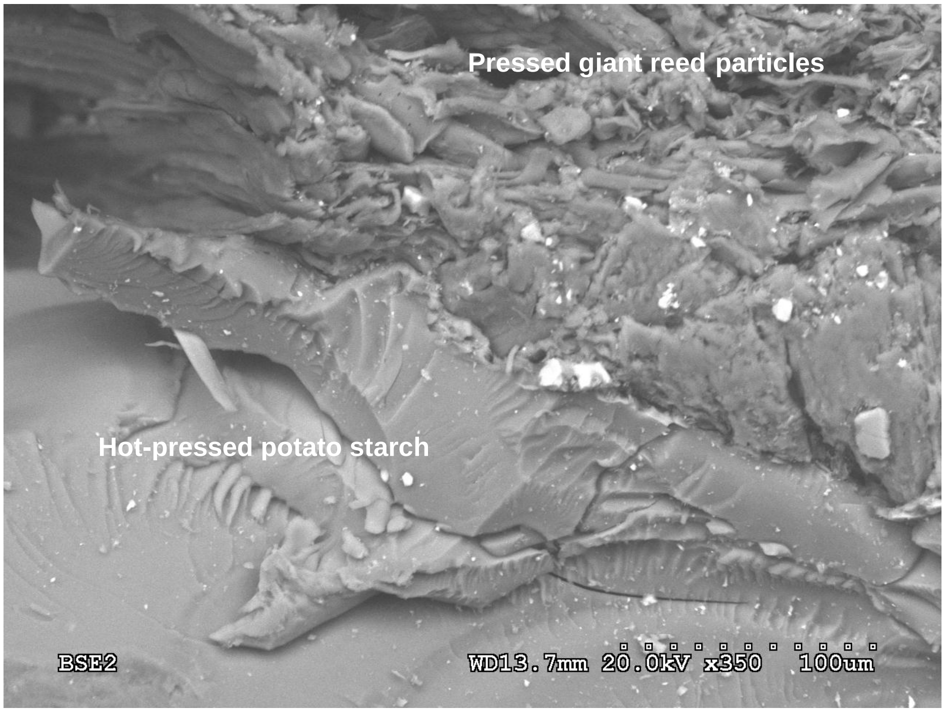


Fig. 6. SEM micrograph of the fractured surface of potato starch as a polymer matrix on one side and particles of giant reed on the other side

CONCLUSIONS

1. Panels of giant reed particles were produced using different nonmodified cereal flours and native starches as binders without the addition of chemicals by hot pressing at low pressing temperature (110 ºC) and pressure (2.6 N/mm^2).
2. The best performance in terms of mechanical properties was obtained using potato starch. With 10% of potato starch and three pressing cycles, panels exceeded the MOR, MOE, and IB values for the P4 grade (load bearing in dry conditions) standard, but failed to achieve the requirement of thickness swelling after 24 h. Particleboards obtained with a 10% of potato starch and two pressing cycles met the requirements for general uses and indoor fitment, including furniture manufacture (in dry ambients).
3. Panels made with corn starch and wheat flour met the standards for general uses (in dry conditions).
4. The SEM observations confirm that gelatinization of the starch is achieved during the hot pressing of the mats.
5. Since the particles were not pre-treated, the starches were not modified, and the pressing conditions were very low; this method can be considered to be a low-cost procedure to manufacture environmentally friendly particleboards.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Ministerio de Economía y Competitividad of Spain (MINECO, ref. BIA 2009-11605).

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Article submitted: July 4, 2012; Peer review completed: October 15, 2012; Revised version received and accepted: October 23, 2012; Published: October 26, 2012.