Fire-resistant cellulose boards from waste newspaper, boric acid salts and protein binders

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Original Research

Keywords: Cellulose boards, waste newspaper, soy protein binder, borate salts, fire-resistant, mechanical properties

DOI: https://doi.org/10.21203/rs.3.rs-195149/v1

Abstract

Housing construction consumes more materials than any other economic activity, with a total of 40.6 Gt/year. Boards are placed between construction materials to serve as non-load-bearing partitions. Studies have been performed to find alternatives to conventional materials using recycled fibers, agro-industrial waste, and protein binders as raw materials. Here, fire-resistant cellulose boards with low density and adequate flexural strength were produced for use as non-load-bearing partitions using waste newspapers, soy protein, boric acid, and borax. A central composite design (CCD) was employed to study the influence of the board component percentage on flame retardancy (UL 94 horizontal burning test), density (ASTM D1037-12) and flexural strength (ISO 178–2010). The cellulose boards were characterized by thermal analysis (ASTM E1131-14) and scanning electron microscopy. Fire-resistant cellulose boards were successfully made with low densities (120–170 kg/m^3) and flexural strength (0.06–0.64 MPa). The mechanical performance and fire resistance of cellulose boards suggest their suitability for use as building materials. A useful and sustainable construction material with great potential is produced with the valorization of waste materials.

1. Introduction

Housing is an unsustainable industry, consuming approximately 44% of the globally extracted resources, which represents more resource input than any other economic activity (de Wit et al. 2019). Therefore, the development of renewable raw materials for housing and construction is fundamental for the fulfillment of the twelfth goal ("Ensure sustainable consumption and production patterns") defined in the Sustainable Development Goals, signed by more than 150 member states of the United Nations (PNUD 2017).

Boards used as suspended ceilings, partition walls, doors, and furniture constitute an important class of materials used in housing construction. Particleboards made from wood particles or flakes and a binder (resin or adhesive) are the most commonly used materials for these applications, while formaldehyde-based resins (phenol-formaldehyde, urea-formaldehyde (UF), and melamine-formaldehyde) are the standard binders for particleboards. Despite the advantages of these adhesives such as good performance, low cost, and high reactivity, these resins emit formaldehyde, which is carcinogenic to humans, according to the World Health Organization International Agency for Research on Cancer (IARC Working Group 2004; Cai 2012).

To develop environmentally-friendly boards, several studies have been carried out to find alternative raw materials for this application. Among these alternative raw materials, cellulose-rich materials, such as recycled fibers and agricultural waste, in combination with natural-based binders have received particular attention as construction materials due to their eco-friendly nature and favorable thermal and acoustic properties. The major drawback of these materials for construction applications is their low fire resistance, which can be improved by the use of additives such as boric acid and borax (Aksogan et al. 2018; Buratti et al. 2016; Gu et al. 2020; Lopez Hurtado et al. 2016; Nagieb et al. 2011, Ricciardi et al. 2014; Yeon et al. 2014). Although boric acid and borax are classified by the European Chemicals Agency (ECHA) as repro-toxic (Category 1B with the hazard statement of H360FD) in the Classification, Labelling and Packaging (CLP) Regulation, this same agency allows its use in building and construction work (ECHA) and there are companies which supply borates for urbanization applications (p.ex., U.S. Borax - Rio Tinto). The reason for that is probably the fact that the CLP-ECHA classification is based on animal experiments at high doses. Besides, recent results based on epidemiological studies give support for a down-classification of boric acid from the referred category (Duydu et al. 2016).

Table 1 shows some relevant studies related to the production of boards from alternative raw materials and natural-based binders and includes their fire-resistance information. Besides, Gu et al. (2020) have shown that soy protein can crosslink in the presence of borate, leading to good adhesive properties.

Table 1 Boards made using alternative raw materials and a protein-based binder

Raw materials	Adhesive	Density (kg/m3)	Flexural strength (MPa)	Fire resistant	Reference
Kraft pulp	Adhesive made from polyvinyl alcohol, urea, phosphoric acid and starch	55	No data	Yes	(Cai et al. 2016)
Oil palm frond	Corn Starch and glycerol	800	5	No data	(Mahmood et al. 2016)
Rice husk, corn pith, and barley straw.	Starch and alginate	58-223	No data	Yes	(Palumbo, Formosa, and Lacasta 2015)
Flax short fibers, aluminum trihydroxide, zinc borate, melamine phosphate, and melamine borate	Pea protein	70-90	0.15-0.39	Yes	(Lazko et al. 2013)
Flax shaves	Casein and vinegar	450-570	2.8-3.9	Yes	(El Hajj et al. 2012)
Wood	Soy protein and wheat gluten	569-700	No data	No data	(Khosravi et al. 2010)

The boards described in Table 1 have been produced through three manufacturing methods: hot-press molding (Mahmood et al. 2016; Khosravi et al. 2010), bond molding (Cai et al. 2016; Lazko et al. 2013; Palumbo et al. 2015) and blending microwave radiation (El Hajj et al. 2012). Boards manufactured by hot pressing have a higher density and flexural strength than boards manufactured by bond molding but lower thermal insulation properties due to the resulting decrease in porosity (Liu et al. 2017).

The data presented in Table 1 and in the previous paragraphs show that it is possible to obtain environmentally friendly boards with adequate properties for use in housing construction from different fibrous residues, using proteins as adhesives and boric acid and borax as fire-retardant additives. In the present work, this concept is extended to the production of fire-resistant and low-density boards using grounded waste newspaper as fibrous raw-material and the bond molding process. The effect of the contents of soy protein and flame-retardant percentages on flame behavior, density, and flexural strength of the obtained boards was investigated.

2. Materials And Methods

Materials

The collected waste newspaper was sorted to remove any foreign objects, such as clips, plastics, and low-quality or moist paper. Then, the waste newspaper was ground into fine flakes with an average size of approximately 0.3 cm in a TRF 60 foliage shredder with cutting blades and hammers. The final consistency of the flakes was similar to that of cotton. Powdered soy protein, boric acid, and borax were supplied by Suquin Laboratories.

Board manufacturing process

The boards were manufactured by bond molding. The fibers from the waste newspaper were mixed with a solution containing soy protein and flame-retardant agents for ten minutes in a hand mixer (Hamilton-Beach, Model 64650). The flame-retardant mixture with boric acid: borax ratio of 5:1 (w/w) was prepared by mixing the components before dissolving in water (Baysal et al. 2007). Samples were transferred to a bakery mold (325 × 195 × 38 mm, length ×width ×depth) and dried at 110 ° C for 16 hours. Finally, 125 × 13 × 13 mm and 150 × 75 × 13 mm specimens were cut from the produced boards for flammability and mechanical tests.

Experimental design

The influence of binder and flame-retardant percentages on flame behavior, density, and flexural strength was tested according to the central composite design (CCD) of experiments shown in Table 2. Testing on a control mixture without flame retardant was also performed. Each treatment was carried out at least three times.

Table 2 Central composite design

Treatment	Normalized factors		Absolute factors				% Recycled paper (C)	Burning rate (mm/min)	Density (kg/m^3)	Flexural strength (MPa)
	A	B	Binder (A)	Flame retardants (B) (% with respect to the sum of A+C)						
			% Soy Protein	Total	Boric acid	Borax				
1	-1	-1	15	10	8.3	1.7	75	5.3 ± 0.1	156 ± 3	0.72 ± 0.09
2	1	-1	25	10	8.3	1.7	65	3.5± 0.2	146 ± 2	0.66 ± 0.05
3	-1	1	15	20	16.6	3.4	65	SE*	143 ± 2	0.64 ± 0.01
4	1	1	25	20	16.6	3.4	55	SE	164 ± 3	0.83 ± 0.01
5	0	0	20	15	12.45	2.55	65	SE	126 ± 2	0.56 ± 0.01
6	-1	0	15	15	12.45	2.55	70	SE	157 ± 1	0.74 ± 0.03
7	1	0	25	15	12.45	2.55	60	SE	147 ± 4	0.66± 0.01
8	0	-1	20	10	8.3	1.7	70	4.5± 0.4	122 ± 3	0.57± 0.02
9	0	1	20	20	16.6	3.4	60	SE	153 ± 6	0.60 ± 0.01
10 (Control)	0	---	20	0	0	0	80	6.0± 0.3	169 ± 4	0.74± 0.08

* Self-extinguishing

Morphology

All samples were analyzed using a Nikon C-PS stereomicroscope with a digital camera. The control mixture without flame retardant and the sample with the highest flexural strength and a low density that satisfied the flammability test were further characterized by SEM on a Quanta FEG 650 model. The samples were coated with gold in stubs using a Quorum 150ES sputter coater.

Mechanical characterization

Mechanical characterization was performed with samples of 150 mm × 75 mm × 13 mm. For each treatment, three samples were tested. Density was measured according to ASTM D1037-12 (American Society for Testing and Materials 1999), and flexural strength was measured according to ISO 178-2010 (ISO 2010) on a Tinius Olsen H25Ks universal testing machine at a rate of 2 mm/min.

Flammability test

The flame behavior was characterized by the UL 94 horizontal burning test (Underwriters Laboratories 2017), which is widely used for flammability studies (Donmez Cavdar et al. 2015; Ren et al. 2015; Lazko et al. 2013; Arao et al. 2014). Boards from each treatment were cut into 125 mm × 13 mm × 13 mm pieces. The average burning rate (mm/min) for each treatment was calculated from the values measured for three samples that had burned to the mark. A material passed the UL 94-HB test if the burning rate was lower than 40 mm/min.

Thermogravimetric analysis

The control mixture without flame retardant and the sample with the highest flexural strength and a low density that satisfied the flammability test were further characterized by thermogravimetric analysis (TGA). TGA was performed with a TA Instruments TGA 5500 thermal analyzer under inert (nitrogen) as well as under an oxidative (synthetic air) atmosphere, according to ASTM E1131-08 (2014). Approximately 10 mg of the samples were heated from 30 to 800 °C at a heating rate of 10 °C/min. All analyses were performed in duplicate.

Statistical analysis

The experimental data were evaluated using analysis of variance (ANOVA) and response surface methodology (RSM). The statistical analyses were performed using Statgraphics Centurion software (Version 16.1.18, Statpoint Inc., Herndon, VA, USA) for a 95% degree of confidence ($P < 0.05$).

3. Results And Discussion

The results presented together with the description of the experimental data points in Table 2 (burning rate, flexural modulus, and density) and the results of morphological and thermogravimetric analyses are discussed in the following sections.

Morphology

The stereomicroscope images of the ten treated samples are shown in Fig. 1, while SEM micrographs of the boards obtained by Treatments 4 and 10 (selected based on CCD results, as discussed in the next section) are shown in Fig. 2. Fig. 1 shows that all boards exhibited an expanded structure with open porosity. More details of this structure emerge in the micrographs of Fig. 2, where the following features can be observed: (i) interconnected pores of heterogeneous pore size and morphology; (ii) an irregular thin layer, most likely composed of binder, partially covering the surface of the fibers; and (iii) irregularly shaped and heterogeneously sized microcrystals of borax and boric acid adhered to the fiber surface by the soy protein binder layer. The highly porous structure is a direct consequence of the low-pressure forming process employed. The good adherence of the components, reflected by the formation of a layer of protein binder on the surface of the fibers and the presence of borax and boric acid microcrystals adhered to this layer, can be explained based on the findings of Gu et al. (2020) about the crosslinking of the soy protein in the presence of borate. Similar behavior in terms of the formation of a protein layer on the surface of the fibers has also been reported for other systems such as soy protein/flax fibers (El Hajj et al., 2012) and pea protein/flax fibers (Lazko et al., 2013).

Density and flexural modulus

The boards presented density values ranging between 122 and 164 kg/m3 (Table 2). These low densities are characteristic of the mold bonding process, due to its low-pressure process, being within the range found in other works of the literature that use the referred process (55 – 223 kg/m3) (Lazko *et al.* 2013; Palumbo *et al.* 2015). The relatively wide variation of density among these works may be ascribed to their differences concerning the nature of the employed residues (percentages of cellulose, hemicellulose, and lignin), type of binder and fire-retardant, and formulation, i.e, the specific proportions of residue, binder, and fire- retardant employed in each case.

The flexural strength values were between 0.56 and 0.83 MPa (Table 2). These values are of the same order of magnitude but slightly higher than those found by Lazko (2013) for flax short fiber panels produced with the same manufacturing process, which is consistent with the higher densities of the samples produced in the present work and the good adherence of the components observed in the stereomicroscope images of Figure 1.

Pareto diagrams for ANOVA on the density and flexural modulus data in Table 2 are shown in Fig. 3. The intensity of the five effects evaluated (AA and A: quadratic and linear effects of the binder content; BB and B: similar meaning with relation to the flame retardant content; AB: interaction effect involving binder and flame retardant contents) is in the same order for both response variables. This result indicates a correlation between the flexural modulus and the density of the produced boards, which is theoretically correct since the flexural strength is related to the resistance of the material to a combined effect of tensile and compressive stresses, which depends on the amount of material per unit of volume. However, in heterogeneous materials, this correlation is not always straightforward because the final mechanical resistance is also affected by additional factors, such as interfacial adhesion. This explanation justifies the difference in relative intensity between effects for the two properties, and ANOVA indicated different numbers of significant effects for density (three significant effects: AA, AB, and B) and flexural strength (two significant effects: AA and AB).

The statistical models including the effects observed in Fig. 3 are presented in Equations (1) and (2) and represent 72.31% and 81.78% of the density and flexural strength data variability, respectively. The respective contour plots, presented in Fig. 4, illustrate the fact that the level of nonlinearity (reflected mainly by the quadratic effect of the binder content and by the interaction between binder and flame retardant contents) in the response is high, even though the considered ranges of soy protein (binder) and flame retardant contents are relatively narrow. In this way, the highest density and flexural strength were reached with the highest contents of soy protein and boric acid salts (Treatment 4; 25% of soy protein and 20% of boric acid salts), while the lowest values of the properties were reached at the central point (Treatment 5; 20% of soy protein and 15% of boric acid salts). This highly nonlinear dependence of density and flexural strength on the binder and flame retardant contents is likely due to the complex morphology observed in Fig. 2 and the fact that the additive content is expected to affect the different morphological parameters, mainly binder layer thickness and distribution uniformity over the fiber surface, the final granulometry of the flame-retardant crystals, and the level of adhesion of these crystals to the fibers.

$$\text{Density (kg/m}^3\text{)} = 532.962 - 37.346*A - 4.897*B + 0.822*A^2 + 0.303*A*B. \quad (1)$$

$$\text{Flexural Strength (MPa)} = 3.366 - 0.250*A - 0.045*B + 0.005*A2 + 0.002*A*B. \quad (2)$$

Additionally, it is important to mention that, in comparison to the control board, all treatments presented a lower density and only Treatment 4 presented a higher flexural strength. Similar results were reported by Lazko et al. (2013) in the production of boards with flax short fibers and pea protein as the binder, in which three of the four fire-retardant additives tested promoted a reduction in the flexural strength compared with boards produced without a fire retardant.

Flame retardancy and thermogravimetric analysis

According to the results shown in Table 2, all treatments, including the board without a flame retardant agent, fulfilled the safety requirement of the UL 94-HB test. Nevertheless, the positive impact of flame-retardant treatments is noticed, since the Control sample (Treatment 10) was the one with the highest burning rate (approximately 6 mm/min). The burning rates of the samples with 10% of flame retardant (Treatments 1, 2, and 8) were in the range of 3.0-5.5 mm/min, while the 25 mm mark was not exceeded for any of the samples with at least 15% (Treatments 3-5, 6, 7 and 9) because all of them presented self-extinguishing features. Taking into consideration that the higher the porosity of the panels the higher the oxygen availability for the burning process, it is particularly relevant that the self-extinguishing ability was obtained with relatively low contents of the flame-retardant agent. This can be attributed to the fact that the borax microcrystals, according to images in Figure 2, seem to be well dispersed on the surface of the fiber and effectively adhered to them by the soy protein layer. Lazko (2013) used 30% of MMB (melamine borate) to achieve a self-extinguishing response in flax fibers panels produced by the mold bonding process, but using pea protein as the binder. However, the comparison of these results is not straightforward due to the differences in composition and density.

TGA was carried out to provide additional support to the results of the flammability test regarding the role of borax and boric acid in the thermal stability of the waste-newspaper panels. This analysis was performed only for the control sample (Treatment 10) and the board obtained through Treatment 4, since it was the only one that presented a higher flexural strength than the control board, in addition to combining low density and good fire resistance. The thermograms of these samples, identified as M#4 and M#10, respectively, are shown in Fig. 5, while the main parameters obtained from these thermograms are summarized in Table 3.

Table 3. Thermogram parameters for samples M#4 and M#10.

Sample	Temperature range (°C) of the weight loss events	Temperature of the maximum rate of weight loss (°C)	Weight loss (%)
M#4 Air	29.8-221.9	50.2	13.6
	221.9-486.2	329.9	42.8
	486.2-796.8	605.4	31.6
M#4 N_2	29.5-152.2	51.9	8.8
	152.2-480.6	342.7	47.1
M#10 Air	30.2-104.1	30.7	7.9
	104.1-323.2	320.9	51.6
	323.4-575.0	457.7	34.3
M#10 N_2	30.0-141.8	51.2	7.0
	141.8-436.9	338.3	61.3

The samples analyzed under an air atmosphere presented three stages of weight loss and residual mass values at 800 °C of 1% and 10% for M#10 and M#4, respectively, similar to the results reported by other authors (Lazko et al. 2013; Cai et al. 2016; Mahmood et al. 2016). The three stages of weight loss may be attributed (in ascending order of temperature) to (i) moisture evaporation; (ii) cellulose, hemicellulose, and lignin degradation by dehydration and decarboxylation reactions; and (iii) oxidation of carbonaceous residue formed in the second stage (Bernabé et al.

2013, Gaan et al. 2009, Rantuch and Chebet 2014). The thermal decomposition of cellulose begins at 210–260°C by dehydration, followed by a major endothermic reaction of depolymerization from 310 to approximately 450°C. Hemicellulose is decomposed at a maximum of 290°C, and lignin thermally decomposes from 280 to 520°C (Monteiro et al. 2012). For pure soy protein, the rate of weight loss is slow (less than 0.1 wt%/°C) until 180°C, moderate between 180 and 200°C, and significant above 200°C (Hernandez-Izquierdo and Krochta 2008). Since stage three is prevented by the absence of oxygen, the samples analyzed under a nitrogen atmosphere showed only the first two-weight losses, with residual masses of 20% (M#10) and 27% (M#4) at 800°C.

Regarding the parameters related to the stages of degradation, M#4 presented greater thermal stability than M#10 under both inert and air atmospheres, demonstrating that the boric acid salts improved the thermal stability of the boards.

4. Conclusions

In this study, it was demonstrated that it is possible to obtain by the bond molding process, fire-resistant and low-density boards using grounded waste newspaper as fibrous raw-material, soy protein as adhesive, and boric acid and borax as fire-retardant additives. All the produced boards presented open porosity and the good adherence of the components. This behavior was attributed to the formation of a layer of protein binder on the surface of the fibers and with the borax and boric acid microcrystals adhered to it.

Flammability test and TGA data showed that the addition boric acid and borax led to boards with improved thermal stability and all the produced boards fulfilled the safety requirement of the UL 94-HB test. The density and flexural strength of the boards presented a non-trivial dependence on the formulation in the range of compositions studied. Density values ranged between 122 and 164 kg/m3 and flexural strength between 0.56 and 0.83 MPa. The lowest values of these properties were observed at the central point of the experimental design (20% of soy protein and 15% of boric acid salts), while their highest values were reached with the highest contents of soy protein and boric acid salts (25% of soy protein and 20% of boric acid salts). This behavior was adequately described by the obtained statistical models.

The low thermal conductivity of cellulose, the main component of the waste newspaper, and the fact that it was possible to produce boards with properties similar to those of other materials used for this application indicate that developed boards can be used as sustainable insulation materials in construction and promote valorization of waste newspaper.

Declarations

Conflict of interest:

The authors declare that they have no conflicts of interest.

Acknowledgements/ Funding

The authors acknowledge the financial support from the Colombian Department of Science, Technology, and Innovation—COLCIENCIAS (Young researcher program in alliance SENA), and the Vicerrectoria de Investigaciones, Universidad de Santander UDES.

Nilo Sérgio Medeiros Cardozo would like to thank CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brazil) and CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brazil) for their financial support.

Conflict of interest: The authors declare that they have no conflict of interest or competing interest that could have appeared to influence the work reported in this paper.

Availability of data and material: Data are available from the corresponding author upon request.

Code availability: Not applicable

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