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PERFORMANCE OF BIOBASED INSULATION BOARD FROM CROP BY-PRODUCTS AND NATURAL GUMS

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Abstract

In the present paper, the performance of an innovative thermal insulation rigid board is evaluated in terms of fire behaviour and fungal resistance. The board is based on vegetal pith and a natural gum (corn pith and sodium alginate) and it is completely compostable. This new composite was developed in previous work. Here boric acid and aluminium hydroxide are used as fire retardants and montan wax and acetic acid and lactic acid are used as water repellent and fungicides respectively. Interactions between these different treatments is investigated. Both flaming and smouldering combustion processes of the different formulations are evaluated by small-scale techniques which include pyrolysis microcalorimetry and thermogravimetric analysis. A medium-scale device is also designed in order to study the impact of the different additives to the smouldering kinetics. Fire behaviour tests show that good improvement is obtained, both in flaming and smouldering combustion when boric acid is added. Although smoldering is not avoided in any case, the addition of 8% of boric acid or aluminium hydroxide slows down the speed of combustion propagation. The effect of the different additives on the moisture content and mould growth at 97% RH and 27ºC is analysed. Under such severe conditions none of the additives is able to prevent mould growth, with the exception of boric acid. None or marginal mould growth was observed on samples containing 8% of boric acid although moisture content was higher than the other cases.

Keywords:

Corn pith, alginate, thermal insulation, fire behavior, smouldering combustion, fungal attack

1 INTRODUCTION

The building sector is moving towards new approaches to energy efficient design, which includes not only the decrease of the thermal transmittance of the building envelope but also the reduction of the embodied energy through the use of low embodied carbon and locally available building materials.

The use of crop by-products as raw materials in building insulation products might contribute in this respect [Korjenic 2011; Madurwar 2013; Mohanty 2000]. Together with the good hygrothermic properties of these natural materials, their availability from renewable resources is considered as one of their main advantages compared with other petroleum-derived insulations [Palumbo 2014]. In addition, the use of crop by-products has a positive environmental impact because implies the revaluation of accumulated agricultural waste. All of these beneficial aspects encourage their use in building insulation, but it is necessary to analyse their potential response under real conditions. In particular resistance to fungal attack and fire behaviour are the two main issues to be considered before establish the feasibility of their use. In this research, the performance of an innovative thermal insulation rigid board is evaluated. The board is based on corn pith agglutinated with sodium alginate [Palumbo 2015; Palumbo 2014]. Here, the corn pith is pre-treated with different substances i.e. boric acid, aluminium hydroxide, montan wax, acetic acid and lactic acid before forming the board in order to improve the fire reaction and fungal resistance of the material.

1.1 Fire reaction

Several authors have analysed the thermal degradation and flammability of different natural fibres and composites that include such fibres [Alvarez 2004; Dorez 2013; Yao 2008]. In a previous paper [Palumbo 2015], the fire behaviour of experimental insulation materials based on food crop by-products (rice husk, barley straw and corn pith) and natural binders (sodium alginate and corn starch) was evaluated. Both, small-scale pyrolysis combustion flow calorimetry (PCFC) and fire reaction tests indicated that the fire properties of the six experimental natural insulation materials were very favourable when compared with other organic foamy materials commonly used in building insulation, such as polystyrene and polyurethane. The use of alginate as a binder improved the properties of the crop by-product alone, especially in the case of corn pith, where both the total heat release HR and the peak of heat release rate PHRR were reduced by 30%.

In addition to flaming combustion, that take place in gas phase between the generated volatile gases and oxygen, smouldering was also observed in corn pith based panels [Palumbo 2015]. Smouldering is a slow flameless form of combustion that is sustained by the exothermic surface reaction between solid fuel and oxygen. This type of combustion is characteristic of porous materials which form a solid carbonaceous char when heated, and is frequently observed in cellulosic materials [Ohlemiller 1983; Dougal 1998; Hagen 2011]. Smouldering propagation is about ten times slower than flame spread over a solid. In spite of its weak combustion characteristics, smouldering is a significant fire hazard. The initiation and propagation of smouldering are controlled by several interrelated factors as surface area per unit mass of fuel, permeability and thermal insulation [Drysdale 1998; Shafizadeh 1979; Moussa 1976]. He et. al. [He 2009] investigated the reaction heat of agro-stalks using a simultaneous thermal analyser (STA), in air, using a crucible with lid. Based on the analysis of the DSC curves, oxidative polymer degradation heat and char oxidation heat were obtained from experimental data. Ohlemiller et al.[Ohlemiller 1990] analysed both unretarded cellulosic insulation and insulation having 25 wt% of the smoulder retardant, boric acid, added on. Boric acid was unable to halt the smoulder process but it slowed its spread by about a factor of 2.

The strategy commonly used to analyse both flaming and smouldering combustion combines small-scale thermal analysis such as Microscale combustion calorimetry (PCFC), Thermogramivetric Analysis (TGA) and/or Differential Scanning Calorimetry (DSC) with fire reaction tests. Several medium-scale devises have been proposed by different authors [Ohlemiller 1990; Hagen 2011; Hagen 2015] to analyse the kinetics of smouldering processes.

1.2 Mould growth resistance

Bio-based materials have been used in construction for centuries and can last many thousands of years under proper conditions. However they can also degrade due to the action of microorganisms such as fungi or bacteria which compromises their durability. The activity of such microorganisms depends on environment factors, mainly moisture and temperature and on the substrate characteristics such as nutrient content or hygroscopicity.

Crop by-products, like other bio-based materials, represent a potential source of nutrients for fungi and bacteria. However, under the same environmental conditions, they are not equally affected by mould growth. Their resistance against mould growth determines their suitability for application and is generally assessed in terms of critical moisture content or of the limit environment conditions from which mould growth is possible. Hofbauer et. al. [Hofbauer 2008; Sedlbauer 2011] proposed to use an isopleth system to create a material specific profile showing at which climatic conditions (temperature and relative humidity) similar mould activity takes place. They found that biobased materials present extremely different resistance profiles: hemp showed a rather high resistance to mould growth (similar to mineral based materials) while straw was far less resistant.

Nevertheless, the mould growth resistance of a material can be improved with the addition of biocides. Boric acid and derived salts such as borates are commonly used as biocide in some commercial insulation materials such as blowing cellulose (e.g. Homatherm flexCL, Thermaflox and Isocell cellulose insulations). Such substances have the advantage to act both as a biocide and as fire retardant and used to be considered a greener alternative to metal based fungicides, commonly used in wood preservation. However nowadays their toxicity is under evaluation and their use is limited by the European regulation and the international GHS to a concentration under 5.5% (w/w) of the final products [ECHA 2010]. In previous work (unpublished) Lesar compared the fungal and fire resistance of cellulose insulation boards treated with boric acid and aluminium hydroxide. They used different percentages of both products and found that a mixture of 3% of boric acid and 6% of aluminium hydroxide gave the best results. The use of lactic acid bacterium may be an environmental friendly alternative to boric acid and its derivatives. The fungal inhibition action of lactobacterium has been largely studied on food products. Among the metabolites of such bacterium, lactic acid and acetic acid are often detected but it's not clear to which extend they are responsible of their antifungal activity [Schnürer 2005]. Ocallahan [O'Callahan 2012] used a lactobacillus brevis cell-free supernatant to impregnate pine wood samples and found that treated timber exhibit resistance to degradation from all fungi studied. Acid and lactic acid were detected as main metabolites of these lactobacillus. Yang [Yang 2004] found that a 1:2 dilution of L. casei supernatant inhibited growth of three mold -and one stain fungus associated with wood-based building materials. Lactic acid was identified as one of the produced metabolites, but antifungal activity was attributed to one or more unknown metabolites.

A further approach, which is gaining importance in the last few years, is the use nonbiocidal techniques for protection against fungi. Biobased raw materials can be impregnated with water repellents, such as waxes [Lesar et al., 2011], or pretreated to reduce their higroscopicity. Treatments with acetic anhydre [Bledzki 2008; Karr 2000] or laccase mediated grafting are two examples of such treatments [Dong 2014; Garcia-Ubasart 2011].

2 EXPERIMENTAL

2.1 Board formation

Specimens are made using corn pith that is manually removed from corn stalks, grinded and sieved to form a 4 to 2 mm diameter granulate. It is then pre-treated with a 3% water solution of boric acid, aluminium hydroxide, montan wax, acetic acid or lactic acid. The mixture is binded with sodium alginate. Calcium sulphate dihydrate ($CaSO_4 \cdot 2H_2O$) is added as a source of Ca^{2+} ions to achieve alginate gelation. The blend is energetically stirred and is thereafter poured in a mould and hot pressed at 60ºC for 10 minutes to a target density of 50 kg/m^3. Afterwards, the specimens are dried at 60ºC for 24h and unmould. The formulations used for the different specimens are shown in Tab. 1. Moreover, plain samples (OO) and samples containing a larger amount of alginate (AG) are also produced. Probably due to an interaction between the binder and the acid substances, some of the specimens were weakly bonded and felt apart during their manipulation, especially AA and LA.

Tab. 1: Formulations of the different tested specimens.

Code	Corn pith (%)	Alg (%)	$CaSO_4 \cdot 2H_2O$ (%)	Boric acid (%)	Ad (%)	Additive name
OO	96.0	2.9	1.1	0.0	0.0	-
BA						boric acid
AH						aluminium hydroxide
AG						alginate
AA	88.4	2.6	1.0	0.0	8.0	acetic acid
LA						lactic acid
MW						montan wax
BAH						aluminium hydroxide
BAG						alginate
BAA	88.4	2.6	1.0	2.7	5.3	acetic acid
BLA						lactic acid
BMW						montan wax

2.2 Pyrolysis Combustion Flow Calorimetre

Small-scale flammability tests are carried out on a Fire Testing Technology Pyrolysis Combustion Flow Calorimetre (PCFC). The heating rate at the pyrolyser is set to 60°C/min up to a maximum temperature of 750ºC. Products from the anaerobic thermal degradation completed in a nitrogen atmosphere are mixed with a 20 cm^3/min stream of oxygen prior to entering the combustion furnace at 900°C. The Heat Release Rate (HRR) is determined from the oxygen consumption. Total heat release (HR) is calculated as the integral of the HRR over the complete time of the test. For each experiment a mass of 10.0 mg ± 0.1 mg is used.

2.3 Simultaneous thermal analysis (STA)

Small-scale smouldering reaction heat tests are performed by simultaneous thermal analysis (STA) using a TA Instruments SDT Q600. The equipment provides simultaneous measurement of weight change (TG) and differential heat flow (DSC). Experiments are made in air, in a crucible with lid. Due to the lid, the sample is oxidized without flame because of the limitation of air penetration into the crucible. Measurements are taken out with a heating rate of 10°C/min, from 30ºC to 900ºC. For each experiment a mass of 5 mg ± 0.5 mg is used and the flow rate of gas is 50 ml/min.

2.4 Medium-scale smouldering set-up

An experimental set-up similar to the one described by Hagen et al. [Hagen 2011] is designed to determine the velocity of the smouldering front. A specimen of 40 x 40 x 160 mm is hold on top of a hot plate. Five type K thermocouples are placed every 3 cm along the vertical centerline of the sample and one on direct contact with the hot plate. In experiments, the hotplate is heated until a pre-determined temperature and it is then allowed to cool to room temperature. If the pre-determined temperature is sufficient, smouldering process initiates and the test continues until the char and ash of the sample has cooled down to less than 100ºC.

2.5 Mould growth resistance

In this study sealed growth jars are used to monitor mould growth. A saturated solution of sodium sulphate (Na_2SO_4) is used to regulate relative humidity at 97% RH (±2%) within the jars while temperature is restraint to 27ºC (±0.3ºC) throughout the experiment by placing the jars within a laboratory oven. Four replicate specimens of each formulation measuring 20 x 20 x 50 mm are prepared for this test. After oven drying (105°C; 2h), the mass of the specimens is gravimetrically determined and they are placed in pairs in the growth jars. The specimens are not sterilised nor inoculated with specific fungal spores to prevent unknown changes to the substrate and to closely reproduce a real exposure situation [Thomson 2014]. Therefore, naturally occurring spores are used to determine the resistance to mould growth of the specimens. After 4 weeks of exposure, the mould growth is qualitatively determined by visual inspection with the aid of a binocular loupe, following the methodology proposed by Johansson [Johansson 2012] and quantitatively by mass loss as indicated at EN 113 [Humar 2013].

3 RESULTS AND DISCUSSION

3.1 Flaming combustion analysis

The different formulations presented in Tab. 1 are analysed with the PCFC technique. In this kind of microcalorimeter, the sample is heated in an inert atmosphere, and the pyrolysed gasses are mixed with a stream of oxygen to combust, completely, at the combustion furnace. Therefore, the measured heat release rate (obtained from the oxygen consumption) corresponds, strictly, to flaming combustion. The heat release rate (HRR) curves draws a characteristic shape with a main peak around 320ºC which is related to cellulose, a shoulder at smaller temperatures related with hemicelluloses and a tail that some authors relate with lignin and other extractives [Yao 2008].

Tab. 2: PCFC results for the insulation materials incorporating different additives.

	HR (KJ/g)	T_0 (ºC)	TPHRR (ºC)	PHRR (W/g)	Mloss (mg)
00	7.2	191.0	320.6	70.5	0.73
BA	3.1	266.2	327.7	25.8	0.59
AH	5.8	218.2	337.8	73.5	0.67
AG	5.9	205.6	319.8	54.8	0.68
AA	7.8	202.5	326.9	82.1	0.74
LA	8.2	185.5	329.8	83.3	0.77
MW	8.1	183.9	308.2	66.2	0.72
BAH	7.3	236.6	332.8	50.0	0.72
BAG	4.9	243.2	325.5	46.4	0.70
BAA	5.2	241.1	333.7	53.8	0.67
BLA	6.3	203.1	311.0	58.6	0.67
BMW	5.5	215.4	324.2	45.0	0.66

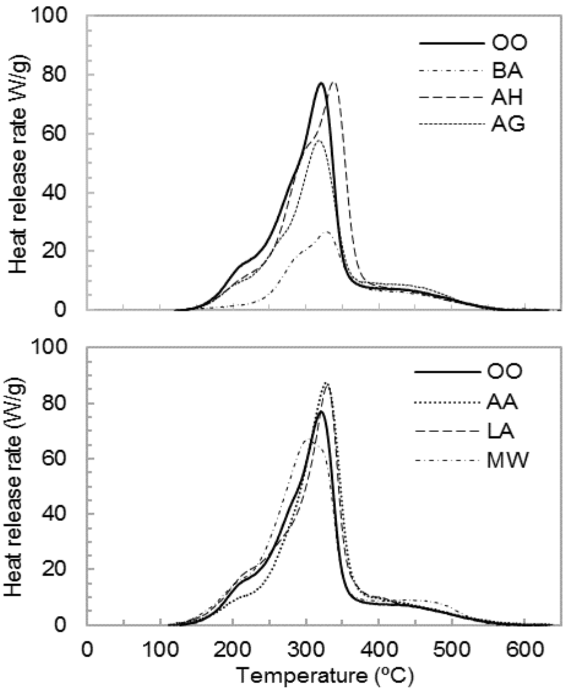

Fig. 1. Heat release rate as a function of temperature for specimens incorporating boric acid, aluminium hydroxide and sodium alginate (top) and montan wax, acetic acid and lactic acid (bottom). Results are compared with plain boards (OO).

The effect of each additive on the fire behaviour of the corn pith-alginate boards is compared in Fig. 1 and Tab. 2. Results show that specimens incorporating boric acid present a remarkably better fire behaviour than the rest of the samples. The onset temperature T_0 (defined here as the temperature at which HRR reaches 10 W/g) is almost 100ºC higher than non-treated boards as the initial shoulder is displaced to the right, the PHRR is reduced more than three times and the mass loss is lower. Alginate show a significant improvement, in agreement with the results previously found [Palumbo 2015], although it is moderate compared with boric acid. Aluminium hydroxide doesn't seem to have an important effect on the fire behaviour of the material although the peak temperature (TPHRR) is slightly higher and montan wax even worsens it, as expected, due to the presence of a second peak at lower temperature. In the light of the results further specimens are made incorporating mixtures of 3% of boric acid and 6% of one of the other additives to determine whether there is a coupled effect. Results are shown in Fig. 2 and Tab. 2. As expected all the specimens show a better behaviour than untreated samples. The fire behaviour of the different specimens is similar although on BAH, BAG and BAA samples the first shoulder is prevented and thus T_0 is 40ºC higher than untreated samples.

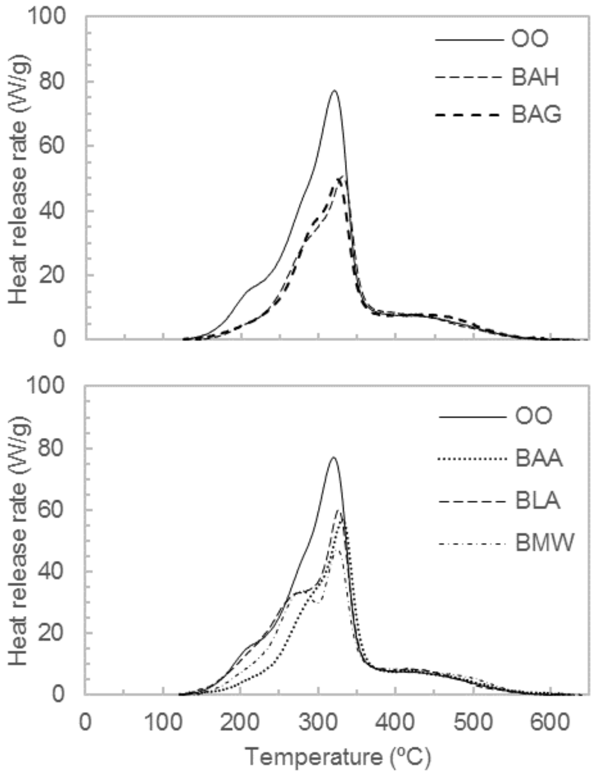

Fig. 2. Heat release rate as a function of temperature for specimens incorporating boric acid and one of the other additives. Results are compared with boards containing only boric acid (BA).

3.2 Smouldering combustion analysis

In previous work it was found that corn pith-alginate composites presented a remarkable propensity to smouldering combustion [Palumbo 2015]. Although fire reaction tests yielded good results, even for non-treated specimens, as they showed self-extinguishing behaviour, after flame extinction a slow smouldering process proceeded until the complete consumption of the specimens. In order to analyse these phenomenon and their possible improvement by flame retardants, two kind of test are done, as described in section 2. At small-scale, a simultaneous thermal analysis STA is performed for the measurement of weight change (TG) and differential heat flow (DSC). These results correspond to smouldering combustion, because the tests are done in air, using a crucible with lid that limit the air penetration into the crucible.

Fig. 3 shows the DSC curves for the untreated sample (OO) and that with boric acid (BA). Both curves present an endothermic peak, at low temperatures, associated to water release, being a bit higher for sample BA. The untreated sample OO has a first exothermic peak at 315ºC associated with oxidative polymer degradation, and a second peak at 444ºC associated with char oxidation. The addition of boric acid delays and decreases the exothermic peaks. The first one appears at 335ºC and is reduced in about 14%. The second one appears at 515ºC and is reduced in about 15%. Results are in a qualitative agreement with the results reported by Ohlemiller for a cellulosic insulation, although in this case the addition of boric acid caused a higher reduction in the second peak [Ohlemiller 1990]. The curve for the untreated sample is quite similar to that obtained by He et al. [He 2009] for corn stalk.

Differential thermogravimetric (DTG) results are shown also in Fig. 3 for the same samples as before. We observe that the shape of the DSC and DTG curves are similar, but the peaks do not occur at the same temperatures. For example, in the untreated sample the first exothermic peak (oxidative pyrolysis) for DSC appears at 314ºC while the corresponding DTG peak do at 273ºC. Similarly, the second exothermic peak (char oxidation) occurs at 444ºC for DSC and at 423ºC for DTG. These differences were also observed by He et. al. [He 2009]. The delay caused by de addition of boric acid is also clearly seen in the DTG curves, as well as the reduction in the magnitude of the first exothermic peak. However, the char oxidation peak seems to be only delayed but not reduced.

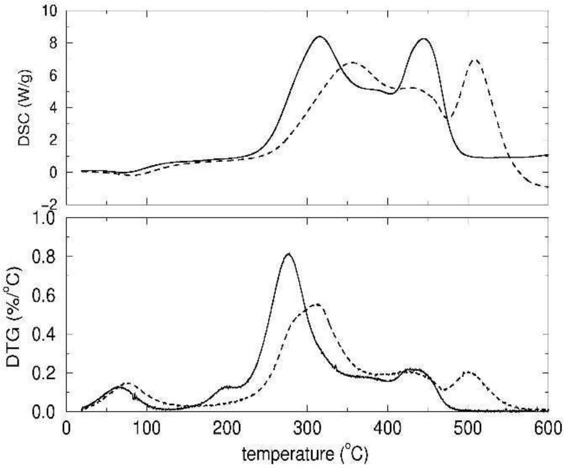

Fig. 3. DSC curves (top) and DTG curves (bottom) for untreated sample (continuous line) and sample with boric acid (dashed line).

Medium-scale smouldering tests are performed according to the device and protocol described in section 2.4. During the experiments, the evolution is visualized with an infrared camera. Temperature is recorded by 6 thermocouples located every 3 cm along the sample. Fig. 4 shows the infrared images in 6 different times (every 5 minutes) for untreated and boric-acid retarded samples. White colour represents the region where combustion is occurring. Red edges correspond to temperatures around 150-250ºC. A significant delay is observed when boric acid is added. Indeed, the high temperature region seems to be smaller in extension.

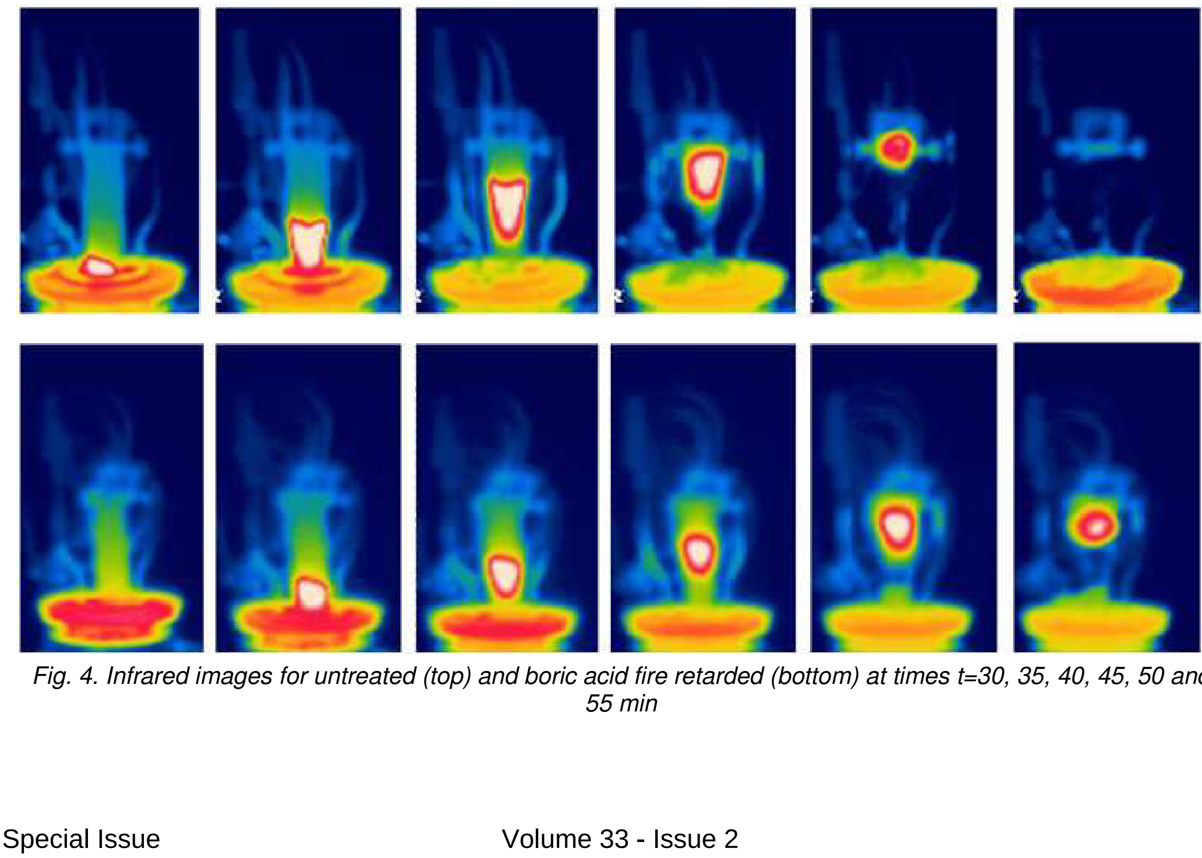

Fig. 4. Infrared images for untreated (top) and boric acid fire retarded (bottom) at times t=30, 35, 40, 45, 50 and 55 min

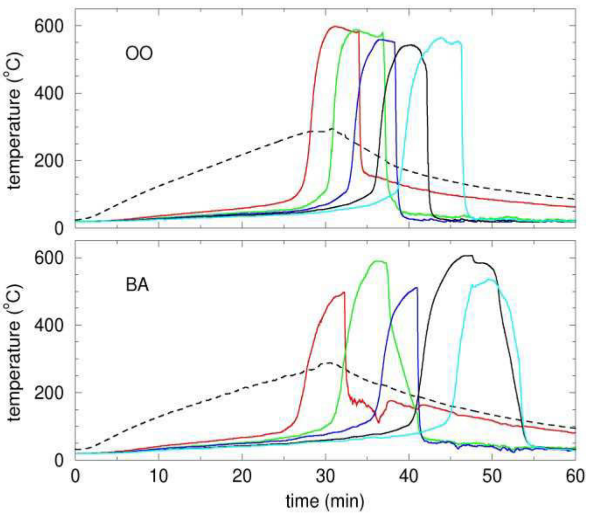

Fig. 5. Temperature evolutions for thermocouples located each 3 cm along the sample for untreated (top) and boric acid fire retarded (bottom).

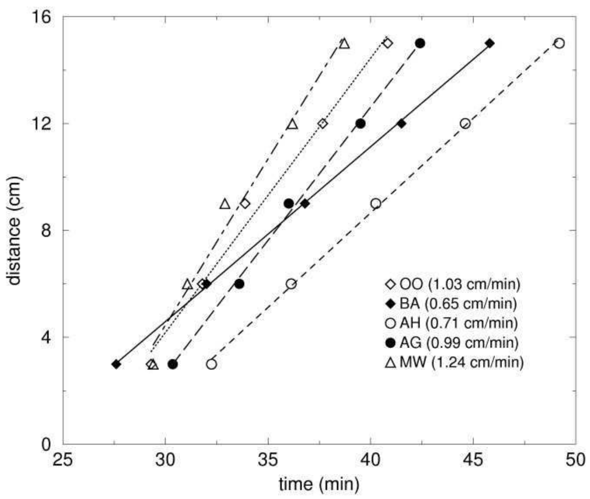

Fig. 6 Thermocouple position versus time at which temperature reaches 250ºC for the different samples analysed. Smouldering propagation velocities (shown in the inset of the figure) are calculated as the slope of the obtained lines.

Fig. 5 shows the temperature recorded by the thermocouples. The dashed lines correspond to the temperatures measured in the hotplate surface, and thus shows the heating protocol: temperature is increased up to 280ºC in 30 min and then it is stopped. From these curves, smouldering velocity can be evaluated by determining the times at which each thermocouple position reaches a temperature, here chosen as 250ºC. In all the cases analysed, position and time exhibit a linear behaviour (Fig. 6). The slopes (smouldering velocities) are shown in the inset of the figure. The velocities for the untreated samples and for samples with added alginate are very similar, but it is interesting to note that aluminium hydroxide reduces the velocity in about 30%, and boric acid does in 37%. As expected, montan wax increases the smouldering velocity (in about 20%).

3.3 Mould growth resistance analysis

Results for mould growth resistance are summarised in Tab. 3. As mentioned in section 2.5, fungal growth is determined qualitatively and quantitatively. Qualitative assessment is based on a five grade rating system (from 0 to 4) where 0 correspond to no mould growth and 4 to heavy growth over more or less the entire surface [Johansson 2012; Johansson 2014]. This method is a fast and effective but, as signaled by Johansson, it is somewhat subjective. In order to reduce measurement uncertainty, five people independently rated all the test specimens. It is found that differences among raters do not significantly affect the final rating, probably due to the fact that most of the specimens are severely affected by mould growth and thus rating is easy to determine. Moreover, mould decay is also determined quantitatively by mass loss. It is important to underline that, as the initial mass of each specimen is only around 800 mg, results are very sensitive to slight variation in mass. Such variations might be due to material loses during manipulation or to remnants of mycelium due to incomplete cleaning of the specimens. Nevertheless, results qualitatively agree with those obtained with the rating method and are in concordance with those presented by Humar et. al. using the same methodology [Humar 2013]. Their results indicate that, after 16 weeks of exposure, the average mass loss for cellulose insulation specimens is 0.1 g; 0.3 g for hemp insulation mats and 0.83 g for wood fibre, which is in the same order of magnitude as the obtained values.

Results show that none of the studied substances, except boric acid, are able to prevent mould growth. Specimens containing 8% of boric acid experience little fungal growth. Mould growth on these samples is rated as 1, corresponding to initial growth, and the mass loss registered is 0.02 g. On the other cases, mould growth is rated as 4 (heavy growth over more or less the entire surface) and the mass loss registered is between 0.10 and 0.15 g, exept from OO and BLA samples, where mass loss is slightly higher (0.17 and 0.19 g respectively). Results for BAA are not shown as the samples felt apart before the measurements. Moreover, the populations found at the different specimens differ (Fig. 7). Only a kind of white filamentous fungi grow on the specimens with boric acid. As the aim of the study was to test the effect of different substances on mould growth resistance the specimens are subjected to highly severe conditions that are unlikely to happen in real situation. Further work needs to be done in order to find the resistance profile of both the untreated and treated materials. Moreover, the effect of fibre pretreatment (e.g. acetylation) on mould growth resistance of the final product may be likewise investigated.

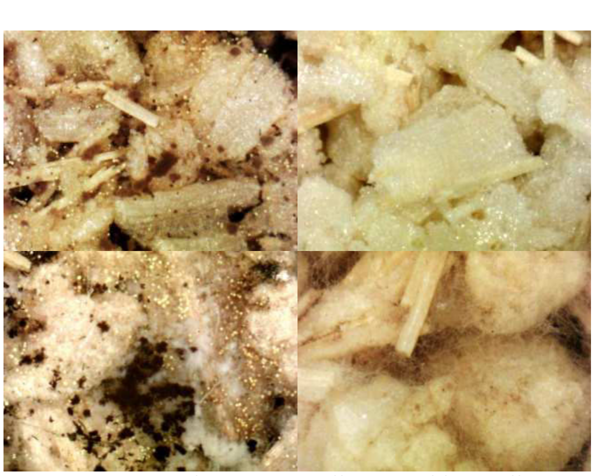

Fig. 7 Images of the mould affection of four different specimens.

Tab. 3: Comparative of the results obtained with the qualitative and quantitative methods. Moisture content of the samples at the end of the essay is also shown.

	Δm (g)	MC %	Δm (%)	Rating
OO	0.17 ±0.01	56 ±6	18 ±0.3	4
BA	0.02 ±0.03	135 ±8	2 ±3	1
AH	0.15 ±0.08	55 ±13	23 ±7	4
AG	0.14 ±0.01	65 ±11	19 ±2	4
AA	0.13 ±0.02	63 ±9	15 ±2	4
LA	0.13 ±0.01	59 ±12	16 ±1	4
MW	0.10 ±0.01	58 ±3	15 ±2	4
BAH	0.15 ±0.05	70 ±5	22 ±4	4
BAG	0.14 ±0.07	77 ±4	17 ±6	4
BLA	0.19 ±0.06	69 ±9	23 ±7	4
BMW	0.15 ±0.02	76 ±7	20 ±2	4

4 CONCLUSIONS

The effect of six different substances on the fire reaction and the fungal growth resistance of an insulation board made with corn pith and alginate is evaluated. The best results are obtained with specimens treated with 8% of boric acid. Regarding flaming combustion, all the studied parameters improve with the addition of boric acid. HR is reduced by about 50%, the PHRR by about 60% and mass loss after combustion is 20% lower. Although smouldering is not prevented, the speed of propagation is reduced by almost 40%. Moreover, resistance against mould growth is also improved. After four weeks exposed to a very high relative humidity only marginal growth is observed which corresponds to a rating of 1 instead of 4. The use of boric acid is restricted to 5.5% (w/w) of the final product at the European Union as it is classified as toxic to reproduction category 2 [ECHA 2010]. However, due the low density of the studied material, the actual amount of boric acid on samples containing 8% of this substance is very low. For instance, the mass of a square meter of a 80 mm thick corn pith-alginate insulation board (density 50 kg/m^3) is 4 kg. Thus, the amount of boric acid contained would be 320 g.

Samples containing mixtures of 2.7% of boric acid and 5.3% of one of the other substances still show an improved fire behaviour. However, this improvement is attributed mainly to boric acid, which is supported by the fact that the PCFC results are similar for all these specimens. Nevertheless, this percentage of boric acid doesn't seem to be enough to prevent mould growth. In fact, similar results are obtained between these samples and non-treated samples.

5 SUMMARY

This paper presents results of an ongoing research on the fire behaviour and fungal growth resistance of a new insulation board based on corn pith and sodium alginate. Specimens are treated with six different substances among which only boric acid gave remarkably good results.

6 ACKNOWLEDGMENTS

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