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Formaldehyde emission in wood based panels: effect of curing reactions

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The influence of hexamine formed as byproduct during the UF cure was ignored in the past. However, with the increasing interest on low formaldehyde emission boards, this issue has become more pertinent. Formaldehyde release from hexamine degradation is extensive and could limit the success of the development of low formaldehyde emission adhesives if the conventional latent catalysts remain in use.

In this study, formaldehyde released from recycled wood, hexamine and cured UF resin was assessed. Citric and oxalic acid, in solid form, were used as catalysts for UF resin in the production of particleboard. Mats with solid acids are less susceptible to resin pre-cure than with a latent catalyst. However, they can have higher formaldehyde emissions than latent catalyst. When both catalysts were used, combined with an ultra-low formaldehyde to urea molar ratio resin the internal bond was improved and formaldehyde content was below 4 mg per 100 g of oven dry board.

Keywords: Formaldehyde, Hexamine, Wood based panels

Introduction

Wood is the most popular building material in the world (Thoemen 2010), presenting many positive characteristics, including low embodied energy, low carbon impact, and sustainability (Anonymous 2010). Wood plays a major role in the Earth's carbon cycle, removing carbon from the atmosphere through the photosynthesis process. Carbon in wood remains stored until the wood deteriorates or is burned (Anonymous 2010). Extending the lifetime of wood through the production of wood based panels is also extending the carbon storage in these composites.

Wood based panels (WBPs), namely fibre and particleboards can incorporate between 50 to 100% of recycled wood in their composition (Costa et al. 2012a).

WBPs are bonded, usually, with formaldehyde based resins. UF resins are the widely used adhesives due to their high reactivity, low cost, excellent adhesion to wood and colourless joints. However, these resins emit formaldehyde as a result of emission of unreacted formaldehyde remaining following curing, as well as from hydrolysis of methylene-ether bridges (Pizzi and Mittal 1994).

Formaldehyde is the simplest aldehyde. At room temperature it is a colourless gas with a pungent odour. However, it is toxic by inhalation, ingestion and skin absorption (Blair et al. 1986), causing nasal and eye irritation at higher concentrations. Until recently, formaldehyde was classified by World Health Organization (WHO) as a 'probably carcinogenic to humans' (Group 2A). In 2004, the International Agency for Research on Cancer (IARC), following a WHO recommendation, changed the reclassification to 'carcinogenic to humans' (Group 1). This recommendation was finally published in 2006 (IARC 2006). Since then, several efforts have been made in order to reduce formaldehyde from wood based products. Several approaches were taken, such as the optimization of resin synthesis protocols (Ferra et al. 2010; Ferra et al. 2012; Costa et al. 2013a); the study of reinforcing additives incorporation into polymeric matrix (Paiva et al. 2012; Costa et al. 2012b); as well as new characterisation techniques such as NIR technology to improve reliability in resin characteristics and properties (Costa et al. 2013b; Henriques et al. 2012a; Henriques et al. 2012b). Several studies focussed on the use of formaldehyde scavengers during board production were also published. Porous materials (Eom et al. 2006; Kim et al. 2006; Kim, 2009) present some scavenging ability, but compromise the bending strength. Tannins, in addition to reducing formaldehyde also improve the internal bond due to the presence of hydroxyl groups (Kim et al. 2006; Eom et al. 2006). Amine compounds such as urea and urea-formaldehyde co-polymers also can be used as formaldehyde scavengers (Boran et al. 2011; Park et al. 2008; Costa et al. 2013c). Sodium metabisulphite is also an effective scavenger, however, as shown by Costa et al.(2013c, 2013d), the form of addition plays a major role on its efficiency and on the physico-mechanical properties of the resulting WBPs.

Formaldehyde emission in WBPs not only comes from formaldehyde based adhesives, formaldehyde can be found in solid wood (Meyer and Boehme 1997), being formed from the main components of wood (cellulose, hemicelluloses and lignin), as well as from its extractives (Schafer and Roffael 2000; Roffael 2006). The use of recycled wood can lead to an increase in formaldehyde emission, particularly because it could include mat and panel rejects (Martins et al. 2007).

UF resins are acid catalysed, meaning they need an acidic environment to cure. Usually ammonium sulphate is used as a latent catalyst (sometimes called a hardener). Despite not being an acid, ammonium sulphate, under heat reacts with formaldehyde forming sulphuric acid, water and hexamine as a by-product (equation (1)). However, under acidic conditions and heat, the hexamine that is formed may decompose releasing formaldehyde (Walker 1944). Hexamine may also react slowly with water, forming ammonia and formaldehyde (equation (2)) (Dreyfors et al. 1989; Pizzi 1983). Therefore both mechanisms may lead to formaldehyde emission during the lifetime of the panel

$$2(NH_4)_2SO_4 + 6CHOH \leftrightarrow 2H_2SO_4 + 6H_2O + (CH_2)_6N_4 \quad (1)$$

$$(CH_2)_6N_4 + 6H_2O \leftrightarrow 6CHOH + 4NH_3 \quad (2)$$

Scarce information can be found in the literature about alternative catalytic systems for UF resin. The use of buffer solutions was tried in the past, but the results obtained were not adopted by industry due to the low reactivity of the glue mix when compared to the latent catalysts (Gunnells and Griffin 1998). Costa et al.(2012c) showed that on using phosphoric acid as catalyst it was possible to have a good internal bond and following conditioning under forceful conditions the particleboards produced with phosphoric acid had a formaldehyde content which was 1 mg per 100 g oven dry board lower than boards with ammonium sulphate. After 7 months of storage, formaldehyde content of boards with UF resin catalysed by acid decreased to around 50% while formaldehyde content of boards with ammonium sulphate increased to around 15%. The higher formaldehyde content with phosphoric acid, after pressing, could be due to unreacted resin and the inability of phosphoric acid to react with formaldehyde while the higher formaldehyde content of boards made with ammonium sulphate could be due to the decomposition of hexamine (equation (2)) formed during the curing reaction (equation (1)). This supports the observation that hexamine acts as a formaldehyde release agent during the lifetime of boards. However, phosphoric acid reduces the pot-life of the adhesive, even at moderated temperatures of 40^0C. As resin blending normally operates at average temperatures of 40^0C plus, this is not practical. The small amount of phosphoric acid added does not allow the addition to be done separately from the resin, and higher concentrations in wood could cause wood hydrolysis, damaging the mechanical performance of wood and wood based panels (Levan and Winandy 1990; Lebow and Winandy 1999). These results support the importance of studying new and alternative catalytic systems for UF resins in order to keep this type of adhesives in the forefront of wood adhesives. Production of ultra-low emitting wood based panels has increased the relative significance of formaldehyde release from sources other than the adhesive, as well driving the study of better alternative catalysts.

In this work, the formaldehyde content of recycled wood collected in a Portuguese particleboard plant was measured and the contribution of hexamine to the cure of UF resins and the formaldehyde content was assessed using the perforator method (EN 120). Particleboards were produced using oxalic and citric acid as catalysts and their performance compared with ammonium sulphate. Different concentrations of acid were tested as well as the combination of ammonium sulphate with acid. The influence of mat pot life was also assessed.

Materials and methods

Materials

Recycled wood was supplied by Sonae Indústria PCDM (Oliveira do Hospital – Portugal). UF resins, urea, hexamine and ammonium sulphate were provided by Euroresinas – Indústrias Químicas, S.A. (Sines –Portugal). Analytical grade oxalic and citric acid were purchased from VWR Portugal.

Methods

Wood particles were blended with resin, paraffin, and catalyst in a laboratory glue blender. Acids in solid state were hand distributed after adhesive (mixture of resin, paraffin and water to adjust moisture content) in the blending step. The resin load in all trials was 7 wt-% (dry solids per oven dry wood). Three-layer particleboards were hand formed in a square aluminium deformable container with dimensions of 220x220x80 mm. Wood mass distribution was 20% in the upper face layer, 62% in the core layer, and 18% in the bottom face layer with a density target of 650 kg m^{-3}. The pressing schedule of an industrial continuous process (16 mm boards) was adapted to a batch cycle laboratory scale press with a computer controlled linear variable displacement transducer (LVDT), a pressure transducer and thermocouples.

The boards were tested according to the European standards for internal bond (IB) (EN 319). Density (D)(EN 323), moisture content (MC) (EN 322) and thickness swelling (EN 317) were also evaluated for quality control purposes and are not reported here. For each series, one board was randomly selected for the analysis of formaldehyde content (FC) based on EN 120 (perforator method).

Results and discussion

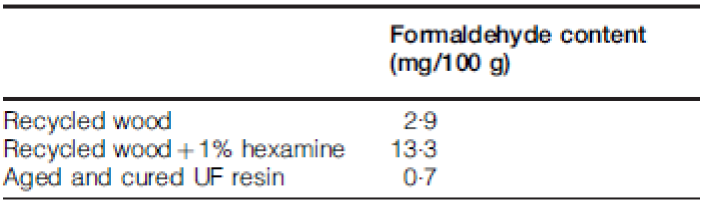
Hexamine as formaldehyde release agent

The formaldehyde content of particleboards was assessed by perforator method (based on EN 120 standard procedure). Hexamine powder was evaluated by placing it in testing flask with recycled wood from same lot (1,0 wt-% hexamine). Standard UF resin (E1) was cured at 60°C during 24 h in an oven and then tested. Table 1 shows the formaldehyde content obtained in the tests.

As expected, the formaldehyde content of recycled wood is considerably high. These results are according to those obtained by Martins et al. (2007). It is important to note that formaldehyde content from recycled wood is closely dependent on the type of wood used.

Standard particleboards are produced with 7 wt-% of UF resin (solid based) and ammonium sulphate is used as catalyst (3 wt-% solid content based on solid resin). Therefore, 100 g of oven dry board contains 7 g of UF resin and 0,21 g (1,6 mmol) of ammonium sulphate. According to equation (1) and assuming the total consumption of catalyst, 111 mg (0,8 mmol) of hexamine will be formed. Data from Table 1 shows that 1 g of hexamine releases 10?4 mg of formaldehyde in perforator method, which corresponds to 1,2 mg of formaldehyde released from the hexamine formed during cure. The aged and cured UF resin, which can be considered as completely reacted, had 0,7 mg of formaldehyde per 100 g of resin. As the resin load in particleboard production is around 7%, this means that less than 0,05 mg of formaldehyde per 100 g of oven dry board could be expected. Therefore, in particleboards produced with this recycled wood and this resin, the minimum formaldehyde content that could be expected is 4,1 mg per 100 g of oven dry board (adding the contributions from recycled wood, hexamine and fully cured resin). However, boards are nowadays produced with emission values well below 4,0 mg per 100 g despite using recycled wood with a high formaldehyde content. As discussed in previous works, UF resins actually may act as formaldehyde scavengers due to the presence of excess unreacted urea (Paiva et al. 2012). However, this is not an efficient approach, because the amount of urea needed to reduce this formaldehyde content dramatically reduces the resin reactivity, and the thickness swelling is considerably increased (Costa et al. 2013c).

Table 1. Formaldehyde content of recycled wood, hexamine and cured UF resin by perforator method



	Formaldehyde content (mg/100 g)
Recycled wood	2·9
Recycled wood + 1% hexamine	13·3
Aged and cured UF resin	0·7

Solid acids as catalyst for UF resins

Standard E1 UF resin

The performance of acids in solid form (oxalic and citric acid) as catalysts was evaluated and the results compared with a latent catalyst (ammonium sulphate). Pot life of the mat was also evaluated.

A series of five particleboard types were produced using different catalysts and concentrations. Oxalic acid (OA) and citric acid (CA) were added in solid state after resin blending in concentrations of 0,6 and 1,0 wt-%(acid based in oven dry wood). Ammonium sulphate (AS) was used as reference at 3,0 wt-% (based in solid resin). After blending, each type of mat was treated or stored under three different conditions: 'standard' mats were pressed immediately after forming; '4 h' mats were stored at room temperature for 4 h before pressing and '4 h 40°C' mats were conditioned in an oven for 4 hours at 40°C before pressing.

Figure 1 shows the results of internal bond for the panels produced.

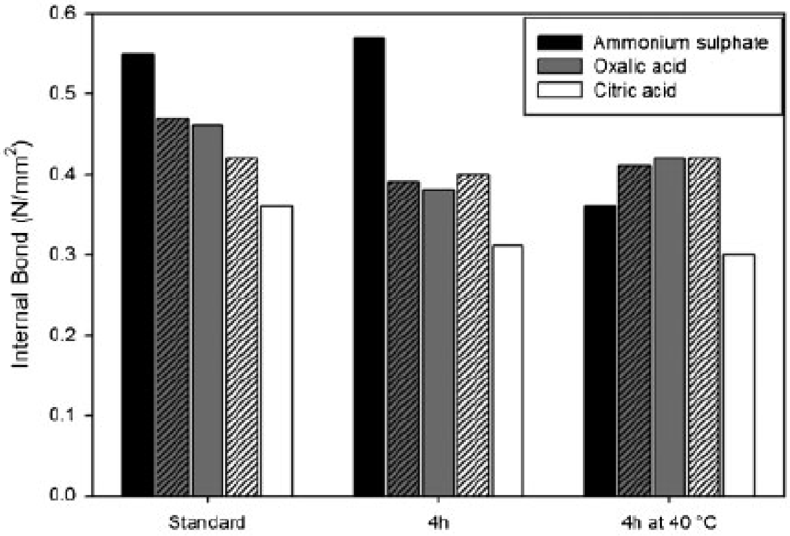


Fig 1. Internal bond of boards produced with standard E1 UF resin catalyzed by different hardeners (pattern bars correspond to amount of acid of 1,0 wt% and unpattern bars to 0,6 wt%)

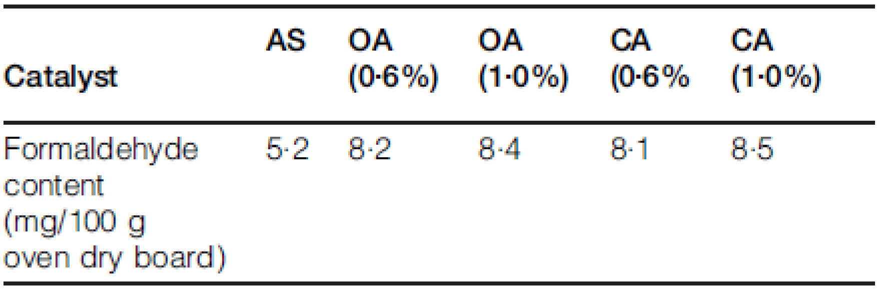
Boards produced with ammonium sulphate had a higher internal bond when compared to standard conditions. When acids were used as a catalyst, under the same conditions, oxalic acid had a better internal bond than citric acid, this being more evident when a lower concentration was used. However, oxalic acid performance is lower than ammonium sulphate. The improved performance of the latent catalyst could be due to the direct addition into the liquid resin, which improved the homogeneity of distribution onto the wood particle surfaces and increased for probability of reaction with formaldehyde. When solid acids are used, direct addition to resin is not possible, since this would initiate resin pre-cure. These catalysts must therefore be mixed with wood particles in the powder form, leading to a less homogeneous distribution. Direct addition of acid catalysts to the resin could be achieved by encapsulation, as already proposed by Costa et al.(2012c).

Storage at room temperature does not present significant variations in internal bond in boards produced with ammonium sulphate. When acids in solid form are used, a slight reduction in internal bond is observed, mainly when higher concentrations are used. However, the internal bond obtained is still above 0,35 N mm^{-2}, which is the minimal requirement for standard particleboards with the same thickness (type P2 according to EN 312).

Storage at 40^{o}C shows the opposite effect: internal bond of boards with ammonium sulphate decreases substantially, while boards with acids maintain the same internal bond. This reduction is caused by resin pre-cure during mat storage. These early formed bonds are destroyed during the pressing stage. Note that Costa et al. (2012c) showed that a standard E1 UF resin with 3 wt-% of ammonium sulphate had a pot life of 2 h. When solid acids were used, on the other hand, resin pre-cure did not occur, since the catalyst remained in solid powder form in the mat and therefore not in contact with the resin. During the pressing stage, the water vapour dissolves the solid acid mobilising the hardener, which results in resin cure.

Table 2 shows the formaldehyde content of particle-boards produced under standard conditions. As expected, ammonium sulphate has a lower formaldehyde content due to its scavenging ability (equation (1)). Formaldehyde content in boards produced with solid acids is similar. Boards with higher amounts of acid added have a slightly higher formaldehyde content, possibly due to some wood degradation caused by the excess of acid. However, as shown by Costa et al.(2012c), in service, boards produced with resins catalysed by acids will have a lower formaldehyde content, during their life time, due to the absence of hexamine. Formaldehyde content values of boards produced with latent and acid catalysts were according to previously published results (Costa et al. 2012c).

Table 2. Formaldehyde content of boards produced with standard E1 UF resin catalyzed by different hardeners



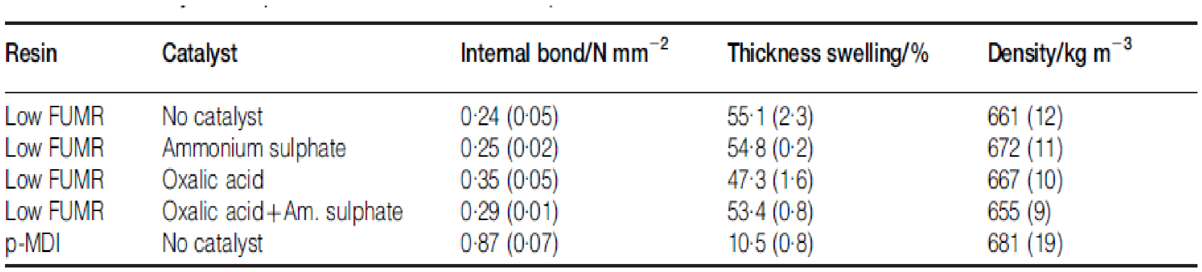
Catalyst	AS	OA (0·6%)	OA (1·0%)	CA (0·6%	CA (1·0%)
Formaldehyde content (mg/100 g oven dry board)	5·2	8·2	8·4	8·1	8·5

Low formaldehyde to urea molar ratio (FUMR) resin

The efficiency of ammonium sulphate as catalyst is strongly related to the concentration of free formaldehyde present in the resin. UF resins with a low formaldehyde to urea molar ratio have a very low concentration of free formaldehyde, which reduces the catalytic efficiency of the ammonium sulphate.

Table 3 presents the physico-mechanical properties of particleboards produced with a formaldehyde to urea molar ratio of 0,90. As expected, when ammonium sulphate was used (control), the internal bond was similar to the particleboard without catalyst. Thickness swelling and density are also quite similar. However, when oxalic acid was used, the internal bond was considerably higher (0,35 N mm^{-2}) and similar to the value obtained with a standard E1 UF resin, confirming that the catalytic mechanism in this case is independent of the free formaldehyde concentration. A combination of ammonium sulphate and oxalic acid results in higher internal bonds than ammonium sulphate, but lower that oxalic acid. Thickness swelling is similar to boards made with ammonium sulphate. On the other hand, particle-boards produced with isocyanate present higher internal bond and lower thickness swelling values. However, these properties cannot be directly related because the adhesive loads were not the same. In addition, the costs of the two adhesives are also completely different. Isocyanate was used as a reference adhesive which has no influence or contribution to formaldehyde emission.

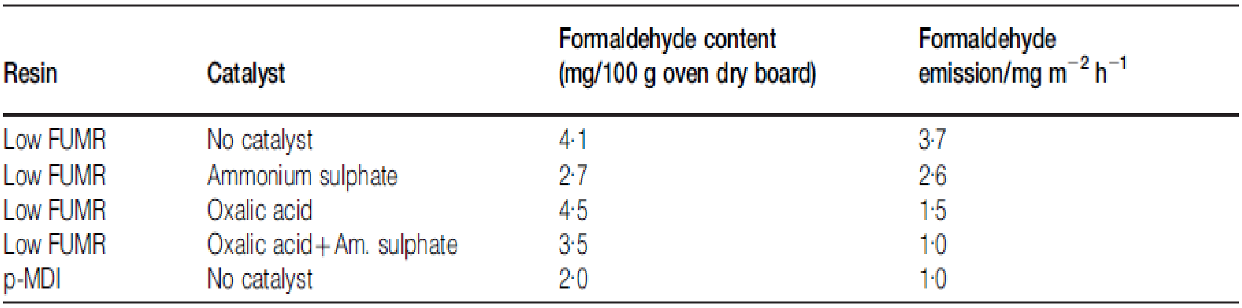
Table 3. Physico-mechanical properties of particleboards produced with low formaldehyde to urea molar ratio (FUMR) and isocyanates (mean and standard deviation)



Resin	Catalyst	Internal bond/N mm^{-2}	Thickness swelling/%	Density/kg m^{-3}
Low FUMR	No catalyst	0·24 (0·05)	55·1 (2·3)	661 (12)
Low FUMR	Ammonium sulphate	0·25 (0·02)	54·8 (0·2)	672 (11)
Low FUMR	Oxalic acid	0·35 (0·05)	47·3 (1·6)	667 (10)
Low FUMR	Oxalic acid + Am. sulphate	0·29 (0·01)	53·4 (0·8)	655 (9)
p-MDI	No catalyst	0·87 (0·07)	10·5 (0·8)	681 (19)

Table 4 summarises the formaldehyde content and emissions. When isocyanate was used, formaldehyde content and emission can be related directly to the formaldehyde from wood particles. Thus, these values can be considered as a lower threshold value for particleboards produced with low FUMR resins.

Table 4. Formaldehyde content and emission of particleboards produced with low formaldehyde to urea molar ratio (FUMR) and isocyanates



Resin	Catalyst	Formaldehyde content (mg/100 g oven dry board)	Formaldehyde emission/mg m^{-2} h^{-1}
Low FUMR	No catalyst	4·1	3·7
Low FUMR	Ammonium sulphate	2·7	2·6
Low FUMR	Oxalic acid	4·5	1·5
Low FUMR	Oxalic acid + Am. sulphate	3·5	1·0
p-MDI	No catalyst	2·0	1·0

The formaldehyde content of particleboards produced with low FUMR resin with no catalyst present had a formaldehyde content of 4,1 mg per 100 g of oven dry board and a formaldehyde emission rate of 3,7 mg m^{-2} h^{-1}. When ammonium sulphate was added, formaldehyde content decreased to 2,7 mg per 100 g of oven dry board and emission rates decreased to 2,6 mg m^{-2} h^{-1}. When the catalyst was oxalic acid the formaldehyde content was 4,5 mg per 100 g of oven dry board, above the formaldehyde content of particle-boards without catalyst. This increase may be related to some degradation of wood constituents into formaldehyde during the test conditions. In the gas analysis determination the formaldehyde emission is substantially reduced to levels near to wood only emissions. When ammonium sulphate is combined with oxalic acid, the formaldehyde content was in between the values obtained for each individual catalyst when used separately. Formaldehyde emissions with this mixture of catalysts were at the same level as particleboards made with formaldehyde-free adhesives.

Conclusion

This work has shown that hexamine, formed as a byproduct of ammonium sulphate reactions during the cure stage, can act as a formaldehyde release agent in wood based panels. Formaldehyde content in recycled wood is significantly higher than in a completely cured UF resin.

Acids in solid form can catalyse UF resins during pressing stages, and the internal bond of the boards is still above the minimal standard requirements. Moreover, when solid acids are used, the mat is less susceptible to pre-cure than when using a latent catalyst and stored for 4 h at 40ºC. When using a standard UF resin, the formaldehyde content in the produced boards can reach the upper limit of the E1 emission class. When applied to low formaldehyde to urea molar ratio resins, solid state acids present higher performance in internal bond strength, with acceptable formaldehyde contents. These results reinforce the importance that more work is needed to look at new catalytic systems for UF resins and it supports that the use of acids should not be discarded as an effective alternative.

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