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ORIGINAL

# Scavengers for achieving zero formaldehyde emission of wood-based panels

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**Abstract** This work examines the performance of three formaldehyde scavengers in wood-based panels. Sodium metabisulfite, ammonium bisulfite and urea were applied in different physical forms during particleboard production, and the resulting physico-mechanical properties (internal bond strength, thickness swelling, density and moisture content) and formaldehyde emission levels were compared. Formaldehyde content was measured using the perforator method, and formaldehyde emission was evaluated both by desiccator and gas analysis methods. The chemical reactions involved in each formaldehyde scavenging process are proposed and discussed. The tested scavengers showed distinct performances under the different emission testing conditions, which were interpreted in terms of the stability of the chemical compounds formed upon formaldehyde capture. Sodium metabisulfite proved to be an excellent scavenger for all formaldehyde methods allowing the production of particleboard panels with zero formaldehyde emission.

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## Introduction

Urea–formaldehyde (UF) resins are the most widely used adhesives in the manufacture of wood-based panels. The production of particleboards (PB) and medium density fiberboards (MDF) consumes a large fraction of UF resins produced worldwide. The industrial success of these resins is associated with their high reactivity, excellent adhesion to wood and low price (Dunky 1998). However, their big disadvantage is formaldehyde emission due to hydrolysis of weak chemical bonds during board production and lifetime use.

Formaldehyde was reclassified in 2004 by the International Agency for Research on Cancer (IARC) as "carcinogenic to humans (Group 1)" (IARC 2006), compelling companies to reduce formaldehyde emission to lower levels. Since 2009, California Air Resources Board (CARB) imposed more restrictions on formaldehyde emission limits, which had a great impact on the wood-based panels industry.

Reduction in F/U molar ratio has been a strategy adopted in the last decades to decrease formaldehyde emission (Myers 1984). However, this reduction decreases the reactivity of UF resins. Currently, reactivity of industrial UF adhesives is near the minimum limit accepted for industrial panel production (Dongbin et al. 2006).

Substitution of UF resins by other formaldehyde-free adhesives does not convince industrial producers due to their higher price or lower reactivity (Amazio et al. 2011; Despres et al. 2010; Tang et al. 2011). In order to increase the degree of cure and reduce free formaldehyde at the end of cure, new catalysts were studied, but reactivity is still too low (Costa et al. 2012a; Gunnells and Griffin 1998).

The use of scavengers, such as natural or bio-based scavengers (Eom et al. 2006; Kim 2009; Kim et al. 2006) or other compounds with good affinity to capture formaldehyde (Boran et al. 2011; Costa et al. 2012b; Park et al. 2008), to reduce formaldehyde emission from wood-based panels is commonly adopted. Costa et al. (2012b) studied the use of sodium metabisulfite ($Na_2S_2O_5$) as formaldehyde scavenger in particleboards produced with UF and melamine–formaldehyde (MF) resins with good results. The reaction of sodium metabisulfite with water forms sodium bisulfite, also called sodium hydrogen sulfite (Eq. 1). The reaction between formaldehyde and sodium bisulfite forms a bisulfite adduct (Eq. 2) (Barberá et al. 2000; Walker 1944).

$$Na_2S_2O_5 + H_2O \rightarrow 2NaHSO_3 \tag{1}$$

$$NaHSO_3 + HCHO \rightarrow NaSO_3CH_2OH\ (\text{adduct}) \tag{2}$$

When neutralized with sodium hydroxide, sodium bisulfite forms sodium sulfite ($Na_2SO_3$), as seen in Eq. 3. Sodium sulfite is used to quantify formaldehyde by titration of the sodium hydroxide formed as by-product (Eq. 4) (Walker 1944).

$$NaHSO_3 + NaOH \rightarrow H_2O + Na_2SO_3 \tag{3}$$

$$HCHO + Na_2SO_3 + H_2O \rightarrow NaOH + NaSO_3CH_2OH \tag{4}$$

When strongly heated, sodium sulfite decomposes into sodium sulfate and sodium sulfide (Eq. 5) (Gerrans et al. 2004). In the pulp and paper industry, sodium sulfide combined with sodium hydroxide is used in kraft process, an alkaline chemical process used for treating wood chips in order to break the bonds between lignin and cellulose and separate the fibers (Miller et al. 2009).

$$4Na_2SO_3 \rightarrow 3NaSO_4 + Na_2S \quad (5)$$

Ammonium and sulfite ions are in equilibrium in ammonium sulfite aqueous solution (Eq. 6). In the presence of sodium ions (existent in the UF resin) and formaldehyde, the sulfite ions react forming an adduct (Eq. 7). Ammonia in aqueous solution reacts with formaldehyde forming hexamine (hexamethylenetetramine) (Eqs. 8, 9) (Dreyfors et al. 1989; Walker 1944).

$$NH_4HSO_3 \leftrightarrow NH_4^+ + HSO_3^- \quad (6)$$

$$Na^+ + HSO_3^- + HCHO \rightarrow NaSO_3CH_2OH\ (\text{adduct}) \quad (7)$$

$$NH_4^+ + HO^- \leftrightarrow NH_3 + H_2O \quad (8)$$

$$4NH_3 + 6HCHO \rightarrow (CH_2)_6N_4 \quad (9)$$

There are several methods for the determination of formaldehyde emission on wood-based panels. They can be divided into two main groups: emittable potential and measurable emission of formaldehyde. The first type measures the formaldehyde content without establishing whether it will actually emit or in which time this emission may occur. The second type determines the actually emitted formaldehyde amount under the test conditions (Dunky et al. 2001).

Emittable potential is evaluated by the perforator method (EN 120), whereas actually emitted formaldehyde can be evaluated by several methods: chamber method (EN 717-1, ASTM E 1333 or ASTM D 6007), gas analysis (EN 717-2) and desiccator method (JIS A 1460). Many authors have tried to relate formaldehyde emissions measured by different methods (Park et al. 2011; Que and Furuno 2007; Risholm-Sundman et al. 2007; Salem et al. 2012), but the relations are influenced by other variables, such as resin type, type of wood-based panels, thickness or manufacturing conditions. Salem et al. (2011a) and Kim and Kim (2005) studied formaldehyde emission from different types of formaldehyde-based resins. Salem et al. (2011b) showed that the manufacturing variables interfere significantly with formaldehyde emissions, and correlations between methods to assess different products were not possible.

The present work studies the performance of sodium and ammonium bisulfite and urea as formaldehyde scavengers in particleboard production. Formaldehyde emissions evaluated by perforator, desiccator and gas analysis methods are compared and discussed.

## Materials and methods

### Materials

Last generation commercial UF resins (0.94 $F/(NH_2)_2$ molar ratio) and urea were provided by EuroResinas—Indústrias Químicas, S.A. (Sines Portugal). Wood particles, paraffin and ammonium sulfate for the production of particleboard were supplied by Sonae Indústria PCDM (Oliveira do Hospital, Portugal). Analytical grade sodium metabisulfite and ammonium bisulfite solution (70 wt%) were used.

### Particleboard production

The adhesive system is composed of UF resin, paraffin, catalyst (ammonium sulfate 30 wt% solution) and water for adjusting the mat moisture content. Wood particles were blended with the adhesive in a laboratory glue blender. The solid resin load was 7 wt% based on oven dry wood. Scavengers, when applied in liquid form, were added to wood particles at the beginning of the blending operation prior to resin blend. Scavengers applied in solid form were dispersed by hand on wood particles after resin blending.

Three particle layer mats were hand formed in a square aluminum deformable container with the dimensions of $220 \times 220 \times 80$ mm$^3$. Wood mass distribution was 20 % in the upper face layer, 62 % for the core layer and 18 % in the bottom face layer. Core and face layers differ in moisture content (11 % in face layer and 8 % in core layer) and size distribution of particles (smaller particles in face layer and larger in core layers, as used in industrial production). Particleboards were designed to obtain a target density between 650 and 700 kg/m$^3$.

The pressing schedule of an industrial continuous process was adapted to a batch cycle in a computer-controlled laboratory scale hot-press equipped with a linear variable displacement transducer (LVDT), a pressure transducer and thermocouples.

For all series, six boards with a thickness of 16 mm were produced with a pressing factor of 9.5 s/mm. Control series were produced using the same operating conditions as for the other series.

### Particleboard analysis

All boards were hermetically conditioned until tested. The boards were tested according to European standards for density (D) (EN 323), internal bond (IB) (EN 319), moisture content (MC) (EN 322) and thickness swelling (TS) (EN 317). For each series, one board was randomly selected for formaldehyde content (FC) analysis according to EN 120 (perforator method) (the formaldehyde content was adjusted to 6.5 % of moisture content according to EN 312). Three boards were used for the desiccator method (JIS A 1460). In the second part of this work, one panel of each series was submitted to the gas analysis method (EN 717-2) for determination of formaldehyde emission.

## Results and discussion

### Evaluation of performance of different formaldehyde scavengers

Table 1 presents the properties of the particleboards produced with 5 wt% scavenger (solid content based on solid resin) added in three different forms:

**Table 1** Properties of particleboards produced with sodium metabisulfite and bisulfite

| | Control | SMBS | SB40 | SB_NaOH |
|---|---|---|---|---|
| Internal bond ($N/mm^2$) | 0.58 | 0.57 | 0.41 | 0.45 |
| Density ($kg/m^3$) | 690 | 696 | 690 | 679 |
| Thickness swelling (%) | 33.3 | 33.5 | 38.2 | 32.9 |
| Moisture content (%) | 7.3 | 7.3 | 7.8 | 7.3 |
| Formaldehyde content (mg/100 g oven dry board) | 2.2 | 1.8 | 1.7 | 2.6 |
| Formaldehyde emission (mg/L) | 0.45 | 0.13 | 0.30 | 0.43 |

(a) solid sodium metabisulfite (SMBS), (b) sodium bisulfite aqueous solution (40 wt% sodium metabisulfite in water) (SB40) and (c) SB40 partially neutralized with sodium hydroxide to pH = 5.8 (SB_NaOH).

As reported by Costa et al. (2012b), small amounts of sodium metabisulfite reduce substantially the formaldehyde content of particleboards. Table 1 supports these results and shows that formaldehyde emission is lower when solid sodium metabisulfite is used. It should be noted that the precision of the perforator method is not defined, but the exclusion criteria in EN 120 standard mention deviations higher than 20 % between two replicates. The method precision can therefore be assumed to be of that order, and smaller differences in formaldehyde content between different test conditions should not be taken into account.

The release of small particles of sodium metabisulfite into air causes respiratory tract irritation (Barberá et al. 2000; Costa et al. 2012b), which can be avoided when the scavenger is applied in liquid solution. However, dissolution of sodium metabisulfite in water forms sodium bisulfite. Due to its proton donor ability, it shows acidic characteristics, which can cause resin pre-cure in the blending operation (Eq. 1). The sodium bisulfite solution, prepared with 40 wt% of sodium metabisulfite, has a pH of 3.7. Internal bond decrease and thickness swelling increase (Table 1) can be related to pre-cure of the resin at this low pH, or to premature consumption of formaldehyde due to higher mobility/dispersibility of the scavenger in liquid form. The formaldehyde content obtained is similar using either metabisulfite in solid form or in solution (Table 1). Differences between formaldehyde content of SMBS and SB40 values are not significant. In case of formaldehyde emission of SB_NaOH and control (without scavenger), the values are also similar and differences are not significant. The lower effectiveness in reducing formaldehyde emission can be related to migration of sodium bisulfite toward the core layer of the mat, dissolved in the vapor phase formed during hot-pressing (Carvalho et al. 2010). Scavenger depletion in the external layers reduces effectiveness of formaldehyde capture in emission testing (desiccator method). Formaldehyde content measurements (perforator method) are not affected by scavenger distribution, since this test evaluates all formaldehyde present in the sample.

The purpose of the trial performed with sodium bisulfite solution neutralized with sodium hydroxide was to avoid resin pre-cure due to low pH, as discussed above.

However, as seen in Table 1, this led to similar emission to the control and slightly higher formaldehyde content. Internal bond has decreased. Sodium bisulfite reacts with sodium hydroxide forming sodium sulfite (Eq. 3). During cure, sodium sulfite reacts with formaldehyde forming sodium hydroxide (Eq. 4) and increasing pH, thus unfavoring the cure reaction. In addition, at high temperatures (190 °C), decomposition of sodium sulfite into sodium sulfide (Eq. 5) can possibly cause some degradation of wood components, which supports the reduction in physico-mechanical properties of the boards (Dreyfors et al. 1989).

Table 2 shows the properties of particleboards produced with ammonium bisulfite solutions (70 wt%, pH = 5.0) with two different incorporations: 5 % (ABS_5) and 10 % (ABS_10) based on solid resin. As observed with sodium bisulfite, the application of scavenger in liquid form reduces formaldehyde emission but negatively affects the physico-mechanical properties of the particleboard.

## Comparison of different formaldehyde emission methods

In the previous part of this work, it was shown that sodium metabisulfite presents higher formaldehyde scavenging ability when applied in solid form. Ammonium bisulfite also presented good performance, but negatively affects the internal bond and thickness swelling. Another effective scavenger is urea, which has been widely used in industry due to its good performance and low price (Costa et al. 2011; Lehmann 1983; Park et al. 2008).

In this section, the scavenging performance of sodium metabisulfite in solid form, ammonium bisulfite in liquid solution and urea is evaluated using three different formaldehyde evaluation standard methods: perforator, desiccator and gas analysis.

Urea was applied in solution (30 wt%) in the face layer to avoid the "blistering effect" noted when a decorative paper is pressed over a urea prill. In the core layer, urea was applied in solid form (prills) to avoid an increase in internal moisture content that inhibits heat transfer (Carvalho et al. 2010). The amount of scavengers added was 5, 10 and 15 wt% (solid scavenger based on solid resin). Physico-mechanical properties (internal bond and thickness swelling) and formaldehyde content (perforator method) and emission (desiccator and gas analysis method) were evaluated for each series of particleboards, and the results are presented below. A control series was produced with the same resin and operating conditions, but without scavenger addition.

**Table 2** Properties of particleboards produced with ammonium bisulfite

| | Control | ABS_5 | ABS_10 |
|---|---|---|---|
| Internal bond ($N/mm^2$) | 0.54 | 0.33 | 0.12 |
| Density ($kg/m^3$) | 669 | 684 | 643 |
| Thickness swelling (%) | 30.8 | 36.3 | 51.3 |
| Moisture content (%) | 7.9 | 7.9 | 8.6 |
| Formaldehyde content (mg/100 g oven dry board) | 2.5 | 1.6 | 0.9 |
| Formaldehyde emission (mg/L) | 0.47 | 0.37 | 0.22 |

All particleboards were produced with the same resin and using the same pressing conditions (16 mm of thickness, 150 s of pressing at 190 °C). Figure 1 shows the internal bond and thickness swelling of the particleboards produced. As previously concluded, particleboards produced with sodium metabisulfite do not present a significant reduction in internal bond, neither suffer substantial penalty in thickness swelling. Urea presents similar behavior. Ammonium bisulfite presents higher penalty in internal bond and thickness swelling.

Figure 2 presents the formaldehyde content of particleboards produced with different scavengers. Urea presents the lowest ability for scavenging formaldehyde.

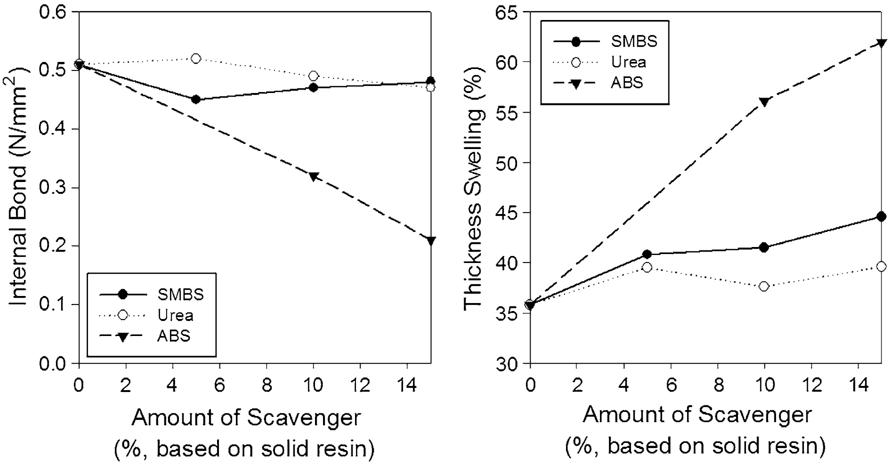


**Fig. 1** Comparison of internal bond and thickness swelling with different amounts of formaldehyde scavengers. *SMBS* sodium metabisulfite, *ABS* ammonium bisulfite)

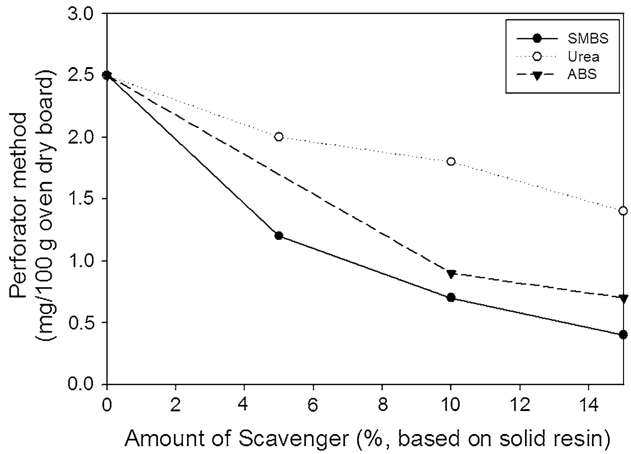


**Fig. 2** Formaldehyde content by perforator method (EN 120) of particleboards produced with different formaldehyde scavengers

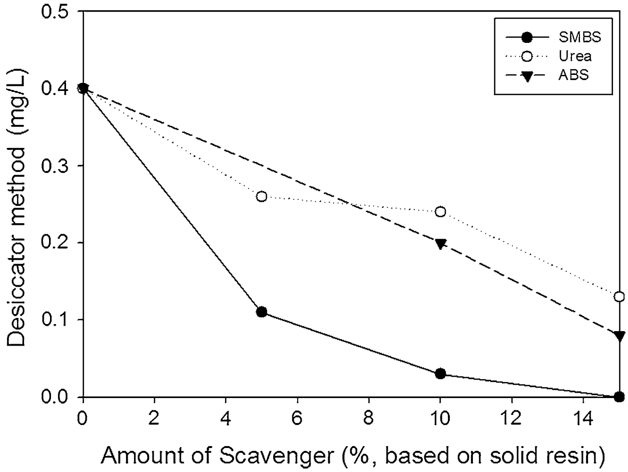


**Fig. 3** Formaldehyde emission by desiccator method (JIS A 1460) of particleboards produced with different formaldehyde scavengers

This result supports the idea that urea is no longer a preferred formaldehyde scavenger to produce ultra-low emission wood-based panels. Sodium metabisulfite and ammonium bisulfite present similar results using the perforator method. Boards produced with 15 wt% sodium metabisulfite show a formaldehyde content near values typical of solid wood (Meyer and Boehme 1997).

Figure 3 shows formaldehyde emission of particleboards analyzed by desiccator method. Sodium metabisulfite presents zero formaldehyde emission when incorporated at 15 wt%, while ammonium bisulfite and urea present a significant reduction in formaldehyde emission. Figure 4 shows formaldehyde emissions by the gas analysis method. The test performed with ammonium bisulfite at 15 wt% is not shown due to experimental errors during analysis.

The better performance of sodium metabisulfite in the desiccator method can be related to higher hydrolysis resistance of the adduct formed in the reaction with formaldehyde. The addition reaction between formaldehyde and urea used as scavenger forms methylolureas, which tends to undergo hydrolysis in the presence of moisture, releasing formaldehyde (Dunky 1998; Pizzi and Mittal 1994). This explains the poor performance of urea addition in the desiccator method, since test pieces are subjected to a high relative humidity. Ammonium bisulfite shows a similar behavior, indicating that the compound formed by reaction with formaldehyde also has low moisture resistance.

In the gas analysis method, urea does not show scavenging ability. This may be related to the low thermal stability of the oligomeric species formed by reaction of urea scavenger with formaldehyde even at a relatively low temperature of 60 °C. Ammonium bisulfite presents a similar trend. Compounds formed between ammonium bisulfite and formaldehyde do not present the same stability as those formed with sodium metabisulfite. The product formed in the presence of ammonium ions is probably less stable than the sodium salt adduct described in

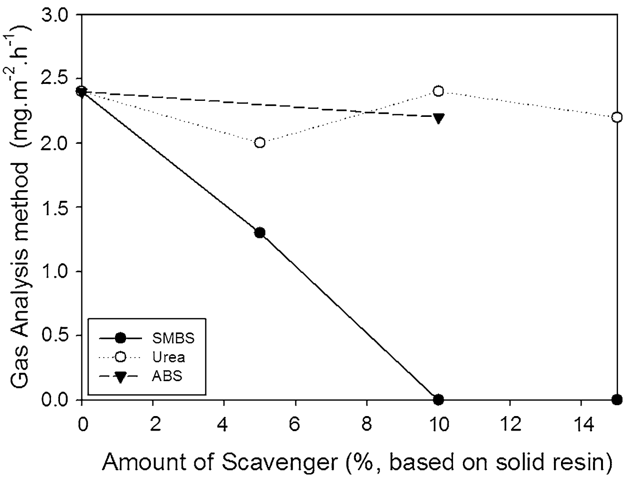


**Fig. 4** Formaldehyde emission by gas analysis method (EN 717-2) of particleboards produced with different formaldehyde scavengers

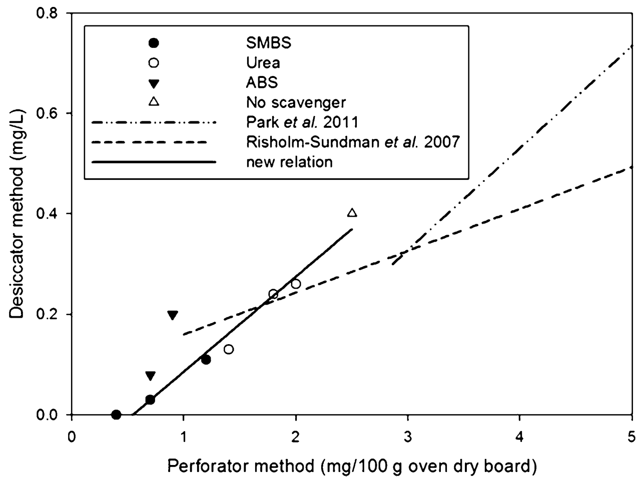


**Fig. 5** Comparison of formaldehyde content by perforator method (EN 120) and formaldehyde emission by desiccator method (JIS A 1460) of particleboards produced with different scavengers

Eq. 7. The higher temperature (60 °C) of the test can also reverse some of the scavenging reactions.

Figure 5 shows the relation between formaldehyde emission obtained by the desiccator method and formaldehyde content measured by the perforator method. Sodium metabisulfite and urea exhibit a similar linear trend, unlike ammonium bisulfite. Park et al. (2011) have compared both methods for particleboards within the range between 2.9 and 16.2 mg per 100 g of oven dry board (o.d.b) and 0.3 and 3.0 mg/L, for perforator and desiccator values, respectively. No scavengers were used in this case. The data of the present study show a similar slope to these authors, but cover lower emission values, including zero emission (Fig. 5). Risholm-Sundman et al. (2007) present a relation between both methods, also without scavenger addition, for formaldehyde contents between 1.0 and 8.0 mg per 100 g (o.d.b.) and emissions between 0.16 and 0.74 mg/L. In this case, the linear relation obtained is different to the work here.

## Conclusion

In this work, different formaldehyde scavengers were studied: powder sodium metabisulfite, aqueous solutions of sodium, and ammonium bisulfite and urea, either in aqueous solution or particulated. Formaldehyde emission of particleboards was evaluated using three standard methods: perforator (EN 120), desiccator (JIS A 1460) and gas analysis (EN 717-2). Boards produced with sodium metabisulfite exhibited formaldehyde content (perforator value) near solid wood levels and zero formaldehyde emissions (desiccator method). The other tested scavengers yielded much lower performances.

The scavenging performance of each scavenger is strongly dependent on the formaldehyde analysis method. The test conditions, such as temperature, relative humidity or air exchange, may interfere in different ways with the stability of the chemical compounds formed in the scavenging reactions with formaldehyde. This study confirms that comparisons between different formaldehyde emission standard methods should be carefully analyzed.

Sodium metabisulfite showed the best formaldehyde scavenging performance for all methods, even the gas analysis method, where the other additives did not exhibit scavenging ability. Boards with higher content of sodium metabisulfite showed zero emission without deteriorating physico-mechanical properties.

A relation between desiccator and perforator method values was given, but it was not possible to establish a correlation between gas analysis and the other methods, because this relation is strongly dependent on the type of scavenger used.

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