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## ABSTRACT

According to FAO – ISPM No 15, in international trade wood packaging material on the basis of sawn timber requires to be heat-treated to avoid spreading of harmful organisms. “Heat-treatment” means a minimum core temperature of the wood of 56° C for a minimum of 30 minutes. ISPM No 15 does not define moisture content and therefore does not include rules for the drying process.

Practitioners report that fresh sapwood, after having undergone a pure heat treatment without any kind of drying, shows a great affinity to fungi (mould and blue stain). Even though the infestation does not affect the strength properties of the wood, it must be considered as an optical and a sanitary problem. An environmentally friendly, cheap and easy to use technique for temporary anti-mould protection of heat-treated wood is needed to bridge the time span between sawing, heat treatment and air or kiln drying.

Results derived from laboratory experiments have shown that the heat-treatment affects the elution of the lipid components within pine sapwood. While searching for alternatives to conventional preservation methods, different environmentally compliant agents (particularly aqueous solutions containing sodium- and potassium carbonate) were found to inhibit the germination of mould and blue stain fungi spores on wood packaging material surfaces in a temporary manner. Besides their effectiveness, the usage of these agents to achieving the temporary protection (e.g. for a couple of weeks) is economically feasible and easily applicable in industrial practice. However, drying (either naturally or technically) is indispensable to avoid fungal hazard on the long run. A combination of temporary treatment immediately after sawing followed by a drying-process (long lasting protection) seems to be the most effective method to protect ISPM No 15 treated wood packaging material against mould.

**Keywords:** Anti-mould treatment, temporary protection, *Pinus sylvestris*, ISPM 15, heat treatment, blue stain, packaging material

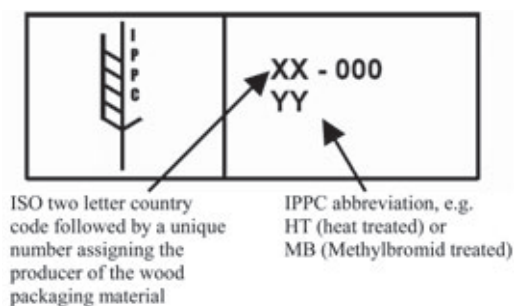
## INTRODUCTION

The increasing international trade bears a growing risk of spreading harmful pests and diseases likely to cause considerable economic and ecological impact once they have established in the new region. Wood packaging material can be considered as one of the major sources for spreading harmful organisms from their normal habitat to regions where natural antagonists do not exist. In the past quite a number of organisms have caused considerable damage. Examples are Dutch Elm Disease, pine wood nematodes, Asian Longhorned Beetle (ISSG, 2007).

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To combat the spreading of pests and diseases from one territory to another, the FAO released the ISPM No 15 in 2002, which in the meantime has been accepted by a large number of countries. According to ISPM No 15 all wood packaging material based on solid wood has to be heat treated (56°C for at least 30 minutes in the centre of the largest piece) or fumigated with methyl bromide (bromomethane) by enterprises which beforehand have been certified. In most countries fumigation is no longer accepted because methyl bromide is classified as toxicologically harmful. As a consequence, heat-treatment is the only way to achieve ISPM No 15.

All material has to be marked accordingly (see Figure 1) so that each piece of wooden packaging material can be followed to the company which has carried out the phytosanitary treatment. Heat treatment of freshly sawn timber is normally carried out in heated chambers similar or identical to kilns used for drying of timber. Another option is to heat-treat the finished product (e.g. wooden pallets, grates, etc).



**Figure 1.** Marking of packaging material according to ISPM No 15 regulation

It is generally accepted that wooden packaging material does not require top quality class. Packaging must be cost effective and therefore, further processing and treatment is often limited to the minimum. ISPM No 15 does not specify any moisture content (MC). As a result time in the heat-treatment chambers is kept as short as possible. Before ISPM No 15 was introduced the major part of wood packaging material was not kiln dried. Pallets, crates and boxes were air dried or stored in such a way that sufficiently low MC was attained before mould could develop on the surface. Even nowadays kiln drying is considered too expensive (Anonymous 2007).

Practitioners report that ISPM No 15 green/un-dried heat-treated material shows an extreme affinity towards mould growth. Discolorations develop on packaging materials surfaces after very short periods of time. This is considered a serious quality problem. Even though the functional properties of the packaging system are not affected, clients do not accept mould on surfaces because of aesthetic and sanitary reasons. Therefore, a simple and cheap method for preventing mould development in connection with ISPM No 15 heat treatment is urgently needed (Lambertz, 2005).

## OBJECTIVES

- Verification of the differences in speed and intensity of mould development between heat treated and normal wood
- Development of a environmentally friendly temporarily effective treatment against mould

## MATERIAL AND METHODS

### *ISPM No 15 Heat-treatment*

Freshly sawn sideboards and scantlings of small diameter German grown pine (*Pinus sylvestris*) were used for assessing the differences in affinity towards mould for ISPM No 15 heat treated and non-heat-treated material. The sawn timber was placed in a laboratory kiln (1x1x2 m<sup>3</sup>) on 25 mm stickers. Vents were kept closed while heating up the kiln to 70°C. Temperature development in the core of the material was controlled by means of temperature sensors. Heat treatment of 80x100 mm scantlings was completed after 4.5 hours. The kiln was unloaded and the heat treated material was left outside in front of the kiln for cooling and “infestation” with mould spores.

### *Storage Conditions after Heat-treatment*

Heat-treated and non-heat-treated sample boards were placed on stickers and subsequently stored under plastic foil for several weeks. The rather unfavourable conditions (~20°C, ~95% rh) which develop under plastic cover were chosen to simulate real life conditions in which in real life wood packaging material can not dry to moisture contents below 18-20% within reasonably short periods of time (e.g. container transport).

### *Chemical Analyses of Heat-treated and Non-heat-treated Wood*

Differences in the composition and distribution of wood constituents were investigated between heat-treated and non-heat treated material. Experiments were carried out with matched samples to minimise variation between test pieces. Parameters analysed were moisture content (MC), content of starch, free sugars and extractives. Methods applied were: oven dry method for MC, staining of micro-slices with Lugol’s solution, methanol extraction and, after subsequent processing, AEC-PAD analysis for free sugars, successive extraction with 1. petroleum ether, 2. ether, 3. Acetone/water over 48 hours for extractives.

### *Comparative Evaluation of Effectiveness of Anti-mould Treatment*

A large variety of formulations was tested, each of them corresponding to a group of chemicals known to be environmentally friendly and easy to use substances. Heat-treated and control material was submerged shortly into the solutions. Liquid solution on the surfaces was allowed to drip off for some time. Subsequently the test material was stored under the same conditions as stated above.

### *Assessment of Mould Growth*

Development of mould growth on the surfaces of the heat-treated and normal wood was assessed using the valuation scheme described in CEN/TS 15082 (July 2005). The scheme is based on the percentage of the surface covered with mould or other coloured fungi. Definitions used for assessing the level of surface infestation are listed in Table 1.

**Table 1.** Evaluation scheme for mould on wood surfaces

Rating	Description	Definition
0	0%	clean
1	< 10% coverage	some
2	11% – 25% coverage	moderate
3	26% - 50% coverage	severe
4	> 50% coverage	very severe
Remark: In case individual sides have been rated differently the mean value of the inspected surfaces shall be taken as the rating for the sample		

## RESULTS

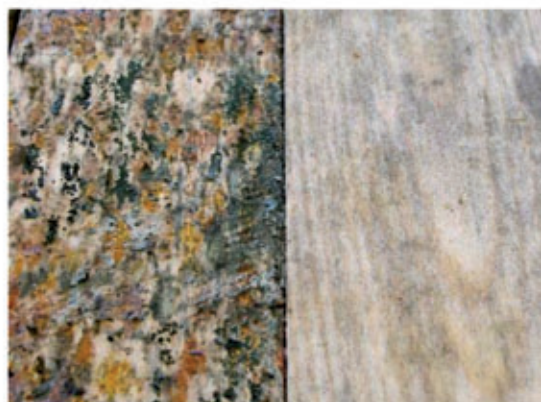
### *Differences Between Heat-treated and Normal Wood*

In all pre-trials the comparative evaluation of heat-treated and normal sapwood surfaces revealed that on the heat-treated surfaces mould and blue-staining fungi developed much faster than on normal not heat-treated sapwood. Differences were not only found in the surface coverage but also on fungal growth depth. In heat-treated material the spreading of discolouring fungal growth from the surface towards the core of the boards and scantlings was much faster and more pronounced.

Figures 2 and 3 show photos of the heat-treated pine sapwood and non-heat-treated control material for the first two evaluation periods. The series of photos covers a storage time of 2 weeks under plastic cover. After three weeks all surfaces were completely covered with mould.



**Figure 2.** Development of mould on ISPM NO 15 heat-treated pine sapwood (left) and untreated control (right) after 7 days under plastic cover



**Figure 3.** Development of mould on ISPM No 15 heat-treated pine sapwood (left) and untreated control (right) after 14 days under plastic cover

Laboratory results impressively confirm reports obtained from operational practice. Practitioners assert that mould problems on green sawn timber for packaging purposes have increased dramatically after introduction of the ISPM No 15 heat-treatment. As a consequence, a few producers of packaging material have decided to use kiln dried material, some use anti-blue stain treatment, whilst others kiln dry the finished pallets. The remaining rest tries to optimize logistics in order to minimize time between production of sawn timber, heat-treatment, production of packaging material and delivery to customer. In such cases the customer might receive mould free pallets, but even in this case mould might develop after some days of storage without sufficient ventilation, e.g in containers.

### ***Reasons for increased mould growth on ISPM No 15 treated wood***

Quite a number of analyses were conducted to find out why mould propagates so rapidly on heat-treated pine sapwood. Examining the differences between heat-treated and untreated material the following results were obtained:

**Table 2.** Results of comparative test of heat-treated pine sapwood and un-treated control

Property/Feature	equal	different
Average moisture content	x	
Moisture distribution	x	
Sugar content at surface	x	
Sugar content distribution	x	
Starch content at surface	x	
Content of extractives:		
➤ Petroleum ether		x
➤ Ether		x
➤ Acetone/water		x

**Table 3.** Comparison of yields obtained after extraction of heat-treated (HT) and non heat-treated control samples with different extraction agents

Extracting agent	Series	Yield [ % ]		Ratio HT/Control
		HT	Control	
Petroleum ether	1	3,29	1,91	1,72
	2	3,54	2,19	1,62
Ether	1	0,56	0,4	1,40
	2	0,17	0,12	1,42
Acetone/Water	1	1,42	1,03	1,38
	2	0,92	0,65	1,42

During the ISPM No 15 heat-treatment no significant redistribution of water or any of the soluble organic nutrients could be distinguished. The only pronounced differences found were the percentages of extractives gained after soft extraction with petroleum ether, ether, and acetone/water. In all cases the percentages of extractable substances gained from heat-treated material were considerably higher (30-70%) as compared to non heat-treated wood. The highest rates were found for extractive groups containing free fatty acids, resin acids and triglycerides, which are known to be attractive feed stock for mould and blue staining fungi (Schmidt, 2006).

Based on these findings two possible modes of action for environmentally friendly temporarily effective anti-mould agents were considered:

- saponification of the fatty acids and resin acids
- alkanalisation<sup>1</sup> of the wood surface to avoid germination of spores

#### ***Chemicals and Substances Acting Against Mould***

In a screening process many different substances and concentrations were tested to identify environmentally friendly temporarily effective anti-mould agents. Tests were carried out on small sapwood specimens which were shortly submerged in the solutions and later stored in very humid climate at 20°C to prevent drying (Fig. 4). Those that proved to be effective for the target period of 2-3 weeks were solutions of sodium carbonate or washing soda (E500), sodium sorbate (E201) and potassium carbonate (E501). Going back into very old literature it could be shown that carbonate treatment has a fungistatic (Heinzerling, 1885; Löfflad, 1978) and fire retarding (Mahlke-Troschel, 1950) effect on dried wood.



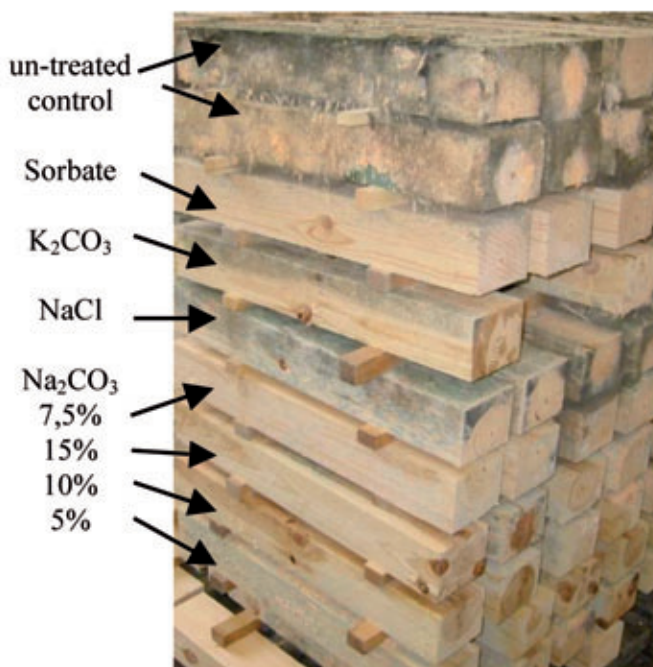
**Figure 4.** Small size test specimens used in screening fungi-static effect of environmentally compliant agents

In a series of field tests larger pieces of sawn timber (80x100 mm<sup>2</sup> scantlings with 1 meter length) were exposed to very unfavourable conditions. Heat-treated controls as well as dip-treated scantlings were stacked together in one single pile and covered with plastic foil. Fungal growth was examined every week according to the procedure described in CEN/TS 15082.

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<sup>1</sup> The acidification pathway, which has been proven to prevent molding of stored sugarcane bagasse by using propionic acid in Trinidad (Walter 1977) and to act against bacterial discolouration (propionic acid, formic acid) of Ilomba wood during transport and drying (Schmidt 2006, page 117) was not considered in this investigation.





**Figure 5.** Mould growth on pine scantlings, control and dip-treated with different anti-mould formulations, after two weeks storage under plastic foil cover

Sodium sorbate, which is also used as a food preservative because of its pH close to neutral, only proved to be effective when used in high concentrations. Due to the relatively high price of sodium sorbate this path was not followed further. Sodium and potassium carbonate were tested with various concentrations. Good results were achieved with solutions in water having concentrations of 7,5% and 10%. These solutions had a pH of 11-12, which is beyond the pH value, which fungi can tolerate for germination (Neubrand, 2004; Schmidt, 2006). Two alternatives for dip-treatment, before heat-treatment and after heat-treatment, were tested. The two alternatives did not show pronounced differences, what allows the conclusion that the ISPM No 15 heat-treatment does not negatively affect the efficacy of the anti-mould agent if the dip-treatment is carried out directly after sawing before the heat-treatment.

In figure 5 the differences in fungal growth on differently treated scantlings are clearly distinguished. After two weeks under cover, scantlings without any anti-mould treatment were completely covered with mould mycelium, whereas especially the carbonate treated specimen did only show very limited fungal growth.

## DISCUSSION

The field tests have shown that the problems associated with mould growth in ISPM No 15 heat-treated green pine sapwood for packaging application can effectively be overcome by a simple dip-treatment in solutions of sodium or potassium carbonate. The treatment can be applied before and after the ISPM No 15 heat-treatment. However, results clearly demonstrate that the anti-mould effect is a temporary and not a permanent effect. Therefore, the carbonate treatment should not be compared to conventional wood protection and long lasting anti-blue stain treatments.

When freshly sawn pine sapwood is dipped into carbonate solutions the pH at the surface is changed. The solution does not penetrate into the wood, but due to diffusion processes the ions will slowly migrate towards the core and consequently the concentration of the carbonates at the surface will decrease. In the presence of CO<sub>2</sub> and water carbonates are transformed into bicarbonates or hydrogen carbonates which have a lower pH as compared to carbonates. If the alkalinity at the surface is the reason for the anti-mould effect of the treatment, this effect will vanish after some weeks because alkalinity decreases over time.

Carbonates are soluble in water. They do not fix to cell wall components (Jakob, 2004). Therefore, the carbonates can easily be washed off from the surface when the wood packaging material is exposed to rain. To avoid mould growth the wood carbonate-treated ISPM No 15 packaging material should be stored under shelter to prevent rewetting. The carbonate treatment can not replace drying, but rather reduce the risk of surface discolouration while the wood moisture content is still above 20%. The drying itself should not be negatively influenced by the carbonate treatment, even though carbonates are slightly hygroscopic. Due to the ionic character of the carbonates an irritation of MC measurement by means of the electrical resistance method can be anticipated.

## CONCLUSION

Based on solutions of sodium and potassium carbonates a temporary effective anti-mould treatment for ISPM No 15 heat-treated sawn timber was successfully tested in laboratory and field tests. The treatment can easily be applied in industrial practice and the wood treated in this way will not be considered as being "contaminated" with wood preservatives. Large scale tests have to be carried out to prove the applicability of the proposed method under industrial conditions.

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