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Wood Sciences and Engineering  
(WSE)**

**September 14-15, 2015**

**Poznan, Poland**

Poznan University of Life Sciences

Faculty of Wood Technology

Edited by Waldemar Perdoch & Magdalena Broda

Poznan University of Life Sciences

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## **Title**

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## PREFACE

There is an evident tendency in the recent global economy to fully utilize the natural renewable resources. One of the possibilities offered by our modern technology is to increase a raw material acquire. However, the increase of the efficiency of the raw material processing is even more important. I believe that the development and improvement of wood processing will be soon a key factor for economic progress in this part of Europe. Reviewing research topics of this Proceedings, I am all the more convinced that the mechanical and chemical wood processing, supported by our scientific projects, will play a significant role in the economy of the 21 century. Therefore, this Proceedings is dedicated to the researchers and students involved in wood science and technology as well as forest products engineering.

Prof. Dr Bartłomiej Mazela  
Dean of the Faculty of Wood Technology  
Poznań University of Life Sciences

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# EFFECT OF HEAT TREATMENT ON THE WEATHERING AND HARDNESS PROPERTIES OF SOME WOOD SPECIES

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## ABSTRACT

Thermal modification is known as a potential method to improve dimensional stabilization of wood and enhance its biological resistance. Scots pine (*Pinus sylvestris*), spruce (*Picea orientalis*), ash (*Fraxinus* spp.), tali (*Erythrophleum ivorense*), and iroko (*Chlorophora excelsa*) timbers were exposed to heat treatment at a temperature of 180 °C for 1.5 hr and at 210 °C for 2 hr based on Thermowood process in the presence of steam. The treated timbers were cut in to small pieces for color measurement as well as brinell hardness and accelerated weathering performance.

The total color change was found similar in Scots pine, spruce and ash when the samples heat treated at 180 °C, whereas it was found highest in tali and lowest in iroko. The color change re increased by the increasing temperature up to 210 °C. The color change was affected by accelerated weathering depends on the wood species, heating temperature and duration of weathering. The increasing in temperature led to higher color change especially for heat treated Scots pine and spruce after weathering. Heat treated ash samples (180 °C and 210 °C) were more affected by weathering than other woods with regard to color change. There was no significant difference between Scots pine (1,72 N/mm<sup>2</sup>) and spruce (1,78 N/mm<sup>2</sup>) in relation to the brinell hardness of the untreated samples. However, the reduction in hardness was found higher in spruce than Scots pine during the heating process at 180 °C and 210 °C respectively. The hardness of iroko and tali was increased by thermal treatment about two times at 210 °C in comparison to their controls.

Key words: Thermal treatment, tropical woods, hardness, weathering

## INTRODUCTION

Thermal modification is known as a potential method to improve dimensional stabilization of wood and enhance its biological resistance. Heat treatment is generally carried out between the temperatures of 180 °C and 260 °C, and it is limited up to 260 °C in modern processes even though severe degradation emerges above 300 °C. In addition, the

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commercial progress has been achieved the most on thermal modification in comparison to other wood modification processes (Hill 2006). In this process, hemicellulose decomposes, lignin softens and cellulose and hydrophilic groups are modified since the wood is exposed to higher temperature above 200 °C (Kocaefe et al. 2006).

It was reported that the major contribution by heat treatment to wood is reduced wood hygroscopicity, furthermore increased resistance to microorganisms as well as improved dimensional stability (Tjeerdsma et al. 1998). Many authors reported that thermal treatment improves the dimensional stability and durability, but reduces the mechanical properties (Tjeerdsma et al. 2000; Boonstra et al. 2007; Ayilimis et al. 2011), and mechanical brittleness was mentioned as a main drawback of heat treated wood (Santos 2000; Unsal and Ayilimis 2005; Yildiz et al. 2006 ). For these reasons, heat treated wood is suggested to be used for furnitures, wall, ceiling, roofing, flooring and fencing instead of load-bearing constructions (Mburu et al. 2007).

It is known that UV-light is the most effective factor causing weathering of wood. The reason of the wood discoloration and photodegradation is the photochemical reactions which require the absorption of all wavelengths composed of UV-light penetrates approximately 75µm and visible light approximately 200 µm in to the wood (Hon 1981; Ayadi et al. 2003). The light is absorbed by lignin component below 500 nm, whereas phenolic extractives, such as tannins, flavanoids, stilbenes and quinones do it above 500 nm in the wood. In addition, wood polysaccharides cellulose and hemicellulose do not absorb light within the visible range (Hon and Shiraishi 1991: Sundqvist 2002).

Color stability for heat-treated wood was found better than untreated wood during the artificial weathering (Ayadi et al. 2003). Effect of high temperature on mechanical properties and physical properties such as dimensional stability and color change with spruce wood was investigated by Bekhta and Niemz (2003), including different relative humidities, heat treatment temperatures and durations. They found that heat treatment led to darkening of wood, improving of the dimensional stability, decreasing in mechanical properties, and proposed that treatment time and temperature were more important than relative humidity in relation to the color properties. Johansson and Moren (2006), investigated the color measurement for strength prediction of thermally treated wood, concluded that temperature was the most important parameter, but color was not a suitable for predicting the strength according to multivariate prediction model of bending strength.

The aim of this study was to investigate the color and hardness properties of heat treated different wood species including softwoods as well as tropical hardwoods.

## MATERIAL AND METHODS

Thermowood process was applied on the wood species such as; Scots pine (*Pinus sylvestris*), spruce (*Picea orientalis*), ash (*Fraxinus* spp.), tali (*Erythrophleum ivorense*), and iroko (*Chlorophora excelsa*) timbers according to heat treatment conditions as follow; all wood species were exposed to the temperature of 180 °C for 1.5 hr and at 210 °C for 2 hr according to Thermowood process. The treated timbers were cut in to small samples, for

accelerated weathering (1,5x7,5x15 cm), and for hardness test (2,5x7,5x20 cm). Color measurement was performed with a Konica Minolta spectrophotometer to the heat treated samples before weathering and after weathering as well as for untreated samples. Total color change  $\Delta E^*$  was determined using CIE  $L^*a^*b^*$  color measuring system, where  $L^*$  describes the lightness,  $a^*$  and  $b^*$  explain the red-green and yellow-blue axes on the chromatic system.  $\Delta L^*$ ,  $\Delta a^*$   $\Delta b^*$  describe the differences between the initial and the final values of  $L^*$ ,  $a^*$ ,  $b^*$  that means before and after treatment or weathering.

$$\Delta L^* = L^*f - L^*i$$

$$\Delta a^* = a^*f - a^*i$$

$$\Delta b^* = b^*f - b^*i$$

$\Delta E^*$  was calculated according to equation;

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \quad (\text{Eq.1})$$

Weathering of the heat treated and untreated samples were performed in the QUV accelerated weathering chamber including UV radiation with UVA-340 lamps, water spray and conditioning. The exposure period covers four cycles, each cycle content 125 hours, thus weathering process lasted 500 hours totally. The changes in color (L, a, b) and glossiness were measured for each cycle that means 125, 250, 375 and 500 hours successively during the weathering process. Glossness of the wood surface was measured with a glossmeter which set up 60° angle, included three values on each point. Hardness performance of the samples was determined by HBE-3000A Electronic Brinell Hardness Tester.

## RESULTS AND DISCUSSION

### Color change after heat treatment

L, a and b values for untreated and heat treated samples were measured, and  $\Delta L$ ,  $\Delta a$  and  $\Delta b$  values were given in Table 1. Table 1 indicated that the effect of heat treatment was shown on the lightness ( $\Delta L$ ) values of the all samples which led to darkness of the woods. It was found highest in Tali woods, while lowest in iroko although the other species had similar values at the temperature of 180 °C. The darkness continued to increasing when the temperature reached to 210 °C. The results on the lightness value were agree with Bekhta and Niemz (2003), because they studied on the spruce wood with different temperatures up to 200 °C and various durations, and concluded that the darkening most of which took placed in 4 hours of exposure, and rapidly increased when treatment temperature was above 200 °C.

**Table 1**  $\Delta L$ ,  $\Delta a$  and  $\Delta b$  values of the samples exposed to heat treatment

Wood/heat treatment	$\Delta L$		$\Delta a$		$\Delta b$	
	180 °C	210 °C	180 °C	210 °C	180 °C	210 °C
Scots pine	-16,18	-31,84	4,61	5,67	5,98	0,87
Spruce	-16,05	-27,70	5,36	7,67	8,21	9,06
Ash	-17,01	-34,19	3,57	3,02	6,42	-0,99
Iroko	-3,48	-16,20	0,97	0,96	0,92	-8,75
Tali	-34,20	-37,52	-0,71	-0,59	-11,04	-13,14

According to  $\Delta a$  values, red hue was dominant in the Scots pine, spruce, ash and less in iroko after thermal treatment with 180 °C and 210°C. However, green hue emerged in the low for tali wood after thermal treatment.

$\Delta b$  was changeable depends on the wood species as well as heat treatment conditions. It was shown that yellow hue increased for Scots pine, spruce, ash and also little bit in iroko heat treated at 180 °C, then decreased in Scots pine, ash and iroko heat treated at 210 °C.

The lower temperatures such as 65 to 95 °C were studied by Sundqvist (2002) with green wood including Scots pine, Norway spruce, and birch by heat treatment. Birch sapwood showed much redder and darker compared with pine and spruce. Pine and spruce displayed similar color properties, and the temperature over 80 °C was found crucial for darkening. Mitani and Barboutis (2014) performed the heat treated fir wood (200 C for 2, 3 and 4 h.) in terms of the color changes and dimensional stability. The darkened wood and improved dimensional stability were provided by heat treatment, the correlation was found between total color difference and treatment time.

### Total color change ( $\Delta E$ )

The total color change was determined based on the  $\Delta L$ ,  $\Delta a$  and  $\Delta b$  values, and presented in Table 2.

**Table 2** Total color change in the heat treated samples

$\Delta E$	180 °C	210 °C
Scots pine	17,87	32,37
Spruce	18,82	30,16
Ash	18,58	34,35
Iroko	4,08	18,45
Tali	36,18	42,56

Table 2 showed that there was no important difference among Scots pine, spruce and ash with regard to total color change. Unlike these species iroko had very lower, whereas tali higher color change when heating at 180 °C. As an expected, color change increased for all species when the temperature moved from 180 °C to 210 °C. The remarkable change in color was obtained by tali wood not only in 180 °C, but also for 210 °C.

### **Color change after weathering**

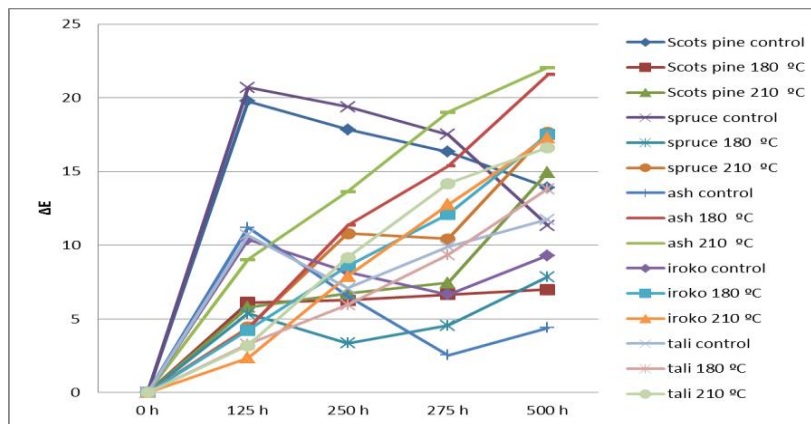
During the accelerated weathering test  $\Delta L$  values were determined and evaluated based on the heat treatment and weathering parameters. Minus values defined the change of the lightness which indicated the darkness in the experiments. Control samples of softwood species Scots pine and spruce exhibited higher darkness than hardwoods in all cycles such as 125, 250, 275 and 500 hours because of the UV irradiation, they were below the coordinate axis. The most change in lightness value was obtained on the surface of ash samples heat treated at 180 °C, but this change was in the positive direction. It was found interesting that untreated tali woods had the smallest change in lightness (0,65) even they gave the higher darkness (-8,38) after exposing to 125 hours weathering . The same trend was also observed on the untreated iroko samples.

$\Delta a$  value gives comment regarding with red and green according to color coordinate system. Accelerated weathering increased the  $\Delta a$  on the scots pine and spruce control samples to be 5,08 and 5,00 respectively, which indicated the red hue after 500 hours exposition. However, it was little changed between minus 0 and 1 for untreated hardwood samples indicating slightly green color. Heat treatment at 210 °C resulted in a negative value on Scots pine and spruce samples during the accelerated weathering process. Similarly, when the hardwoods exposed to both heating, the red hue was reduced by weathering conditions.

$\Delta b$  value mentioned about the yellow and blue hue on the control and heat treated samples exposed to 500 hours accelerated weathering.  $\Delta b$  value was reduced by weathering parameters for all variations including control and heat treatment at 180 °C and 210 °C, in spite of the increasing in 125 hours exposing for the control samples and Scots pine and spruce involved in heat treatment of 180 °C. In overall treatments, scots pine control samples was the least affected (0,55) by accelerated weathering with regard to  $\Delta b$  value. Maximum decrease in  $\Delta b$  was found in ash 180 °C (-14,47) and followed by spruce 210 °C (-14,01) and iroko 180 °C (-13,31) which indicated the blue hue according to CIE L\*a\*b\* color coordinates.

The total color change ( $\Delta E$ ) resulted in different values during the weathering process depends on the wood species, heating, degree of temperature and duration of the weathering as shown in Figure 1.

125 hours weathering greatly affected the spruce (20,71) and Scots pine (19,78) control samples in relation to the color change. However, heat treated samples showed resistance to the color change in the first stage of weathering (125 hours). Further weathering (250 h) increased the color change of spruce with 210 °C, ash with 180 °C and 210 °C, heat treated iroko and tali, whereas decreased the color change of untreated samples of ash, iroko and tali.



**Fig.1** Total color change ( $\Delta E$ ) of heat treated samples during the weathering

The remarkable decrease in color change was shown in the untreated ash samples in 275 hours duration of weathering. Consequently, ash samples heat treated with 180 °C and 210 °C exhibited the highest color change when compared to other treatments and untreated ash achieved the lowest change in 500 hours. The color change was lower in untreated tropical species, iroko and tali than their heat treated samples at the end of the weathering cycle. Another important finding is that the increasing in temperature by heat treatment led to considerable color change in Scots pine and spruce after 500 hours weathering.

The comments on the change in color of wood exposed to heat treatment and weathering either naturally or accelerated were introduced by several authors. Changes in color of spruce by light-irradiation and heat treatment was examined by Mitsui (2006), found that  $L^*$  decreased remarkably and  $a^*$  and  $b^*$  changed dramatically. Lignin degradation was indicated the cause of the color change in wood exposed to light irradiation (Mitsui et al. 2004). In another study, ash, beech, maritime pine and poplar heartwood samples were heat treated at 240 °C for 2 hours, afterwards exposed to UV-light for 835 hours. Heat treated samples resulted in better color stability than untreated samples during the exposure period which was attributed to the less attack of lignin (Ayadi et al. 2003). According to Huang et al. (2012) the improvement in resistance of heat treated wood to photo-degradation might be due to the lower impact of heat treatment on lignin component than hemicelluloses. However, the color difference increases between before and during weathering since weathering degrades the lignin and extractives in heat treated wood, which makes the wood lighten. The heat treatment at 180°C in teak sapwood was found effective in resistance to color change, demonstrated the highest color stability to ultraviolet radiation under accelerated weathering after 168 hours (Garcia et al. 2014).

## Brinell Hardness

Brinell hardness values for control and heat treated samples are presented in Table 4.

**Table 4** Brinell Hardness of the samples exposed to heat treatment

Brinell hardness(N/mm <sup>2</sup> )	Control	180 °C	210 °C
Scots pine	1,72	1,51	1,57
Spruce	1,78	1,41	1,19
Ash	6,79	7,01	6,95
Iroko	4,63	5,15	8,56
Tali	4,51	10,81	9,19

As can be seen in Table 4, hardness performance of control samples of the hardwood species ash, iroko and tali was significantly higher than softwood species Scots pine and spruce which can be attributed to the density of the hardwoods. Another important finding for the control samples was that ash had more hardness value than tropical species with iroko and tali.

Heat treatment at 180 °C considerably increased the hardness in tali woods, and small amount in ash and iroko, but reduced it in Scots pine and spruce to some extent. In the case of heat treatment with 210 °C, there was reduction in the hardness of spruce but no significant increase in Scots pine. As to hardwoods, outstanding increase was obtained with iroko in hardness by 210 °C heat treatment in comparison to the heating at 180 °C .

Impact of heat treatment on strength properties like hardness was studied by earlier workers. An experiment dealt with the color and hardness properties of heat treated softwoods and hardwoods after exposing to accelerated weathering. Higher temperature and time decreased the hardness while the lower temperature and time caused the increasing in the hardness value of heat treated wood. (Karamanoğlu and Akyıldız 2013). Hornbeam (*Carpinus betulus* L.) was subjected to heat treatment at different temperatures (170, 190 and 210 °C) and times (4,8 and 12 h), increasing in temperature and duration resulted in the decreasing compression strength and hardness (Gündüz et al. 2009). Heat treatment did not affect the tangential hardness but reduced the longitudinal hardness in an experiment involved the effect of heat treatment at 190 °C for 4 hours on the mechanical properties of beech wood (Popadic et al. 2010).

Over all treatments carried out in this study concluded that heat treatment with 210 °C significantly improved the hardness of the iroko and tali when compared to their untreated ones. This improvement was determined to be 84 % in iroko and 104 % in tali by heat treatment.

## CONCLUSIONS

Heat treatment increased the darkness of the wood species for the both temperatures at 180 °C and 210 °C. Total color change was found higher in the wood samples heat treated at 210 °C than those treated at 180 °C. It was found highest in tali wood for both temperatures, however the difference in total color change between 180 °C and 210 °C was found low for this species. Heat treated samples showed resistance to the accelerated weathering in the earlier times with regard to color change, which was then increased by weathering parameters in 500 hours exposure. Heat treatment at 210 °C was found effective in the improvement of hardness in iroko and tali.

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