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Artificial Weathering Effect on Surface of Heat-Treated Wood of Ayous (*Triplochiton scleroxylon* K. Shum)[†]

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Abstract: The wood characteristics due to weather conditions change over time, particularly the colour that reflects chemical changes. Natural ageing is usually a relatively slow process; therefore, artificial ageing plays an important role to assess the performance by shortening the time compared to natural weathering conditions. The aim of this research is to evaluate the colour and reflectance variation of wooden surfaces due to artificial weathering obtained through a solar box chamber simulating outdoor conditions and subsequent water leaching. With the increase of weathering times, untreated specimen surfaces darken whereas treated specimens lighten so as to tend to have a similar colour or in any case to decrease the chromatic difference that was at the beginning of the weathering tests. The measured values of conductance are higher in the leaching water of untreated specimens and tend to decrease after the first cycles. The values of pH range between 4.00 and 4.52 in untreated and treated specimens. FTIR spectroscopy demonstrated that water leaching caused loss of materials from the specimens, mainly from those thermally treated. FTIR spectra exhibit signatures of polysaccharides materials as main compounds. Bands of lignin and extractives are also visible. Water leaching seems to remove degraded surface micro-particle of wood.

Keywords: aging; colour; hypercolorimetric multispectral imaging; reflectance; FTIR

1. Introduction

Wood products and wood derivatives are widely used outdoors because of aesthetic qualities, insulation capacity, ease of supply, sustainability, even though wood is biodegradable and subject to alterations due to biotic and abiotic agents. The wood characteristics due to outdoor conditions change over time, particularly the colour, that reflects chemical changes [1-2]. Mainly Moisture and UV component of solar radiation induce the progressive alteration of the wooden surfaces exposed outdoors [3]. Natural ageing is usually a relatively slow process, therefore artificial ageing plays an important role in assessing performance by shortening the time compared to natural weathering conditions. An approach is the surface protection by means of different kinds of commercial products such as solvent-borne, water-borne, high solids, powder coatings and radcure products [4-6]. Another approach is wood modification, a set of processes that give the treated material a greater ability to cope with damage due to the outdoor environment by inducing greater durability; these process are also performed to enhance the physical, mechanical or aesthetic properties of wood and derived products with the advantage of not being harmful to users and the environment, just like natural wood [7]. Thermo-treatment proved to be an interesting approach [8-9]. The dark colour

obtained with the heat treatment gives the light-coloured wood an attractive appearance [10] as well as a better behaviour towards thermo-hygrometric variations in the outdoor environment. The heat treatment modifies the wood material by reducing hygroscopicity and consequently promotes greater dimensional stability; it also prolongs the service life of wooden products [11]. Colour stability is an important feature to maintain the aesthetics of the products over time in outdoor conditions, ensuring a low level of maintenance and therefore of management costs.

Ayous wood is widespread on the market. Untreated is used for panels, light carpentry and furniture; while heat-treated wood is mainly used for facade cladding and saunas covering. This material was chosen for this study as part of ongoing research, to better understand the influence of atmospheric agents on heat-treated wood. In this study artificial weathering of Ayous wood (*Triplochiton scleroxylon* K. Schum) was investigated using a laboratory system to accelerate the aging of the specimens. The artificial weathering simulates the effect of the UV and leaching, however the effect of the staining fungi and other pest cannot be simulated with these devices. The colour variation of wooden surfaces due to weathering was evaluated in thermally treated and untreated specimens. An innovative multispectral hypercolorimetric imaging (HMI) technique was applied on wood specimens for the first time.

2. Materials and Methods

Two untreated and two heat-treated Ayous wood specimens were used for the ageing tests. The thermally modification was conducted on planks of Ayous from Cameroon in an industrial system that used a slight initial vacuum in an autoclave (Maspell WDE Model TVS 6000) and a treatment temperature of 215 °C for three hours.

After cutting, specimens were stored at laboratory in darkness. To simulate weathering, specimens were subjected to 6 cycles of combined solar box and water leaching. A Model 1500E Solar Box (Erichsen Instruments) was used to simulate the exposure to solar radiation and a horizontal shaking agitator with a glass container of distilled water to simulate water leaching.

The weathering cycles were performed as follows (see Table 1 for a synthetic description of the ageing cycles): first cycle 72 h (3 days) of UV irradiation, followed by 5 h of water leaching; second cycle up to 168 h (7 days) of irradiation followed by another 5 h cycle of water leaching; third cycle up to 336 h (14 days) of UV irradiation followed by 5 h of water leaching; fourth cycle up to 504 h (21 days) of UV irradiation and 5 h of water leaching; fifth cycle up to 672 h (28 days) of UV irradiation and 5 h of water leaching; and finally sixth cycle up to overall 1008 h (42 days) of UV irradiation and 5 h of water leaching. The solar box chamber is equipped with a 2.5 kW xenon-arc lamp operating at 550 Wm^{-2}, 55°C and UV filter at 280 nm. The colour measurement was performed after each UV irradiation and after each complete cycle, taking care to equilibrate the specimens in an oven at 55 °C for 24 hours after leaching, so that the moisture content did not affect the sample colour.

Colour was monitored using an X-Rite CA22 reflectance spectrophotometer under the following conditions, according to the CIELAB colour system: illuminant D65, standard observer 10°, geometry of measurement 45°/0°, spectral range 400-700 nm, measurement diameter 4 mm, white reference supplied with the instrument. The points of colour measurement were 30 for each specimen. Three measures were acquired for each point, so that 90 measures were obtained for each specimen. Considering the two untreated and the two thermally treated specimens, 360 colour measurements were taken in total. Average values have been considered for each specimen.

Reflectance of the surface was obtained using an innovative multispectral hypercolorimetric imaging (HMI) technique developed by Profilocolore srl and for the first time applied on wood specimens. HMI was performed at time 0 h, 504 h and 1008 h of irradiation and after the water leaching. This imaging technique allowed for obtained calibrated multispectral images, from 300 to 1000 nm, of the entire surface of the specimens that were subsequently processed through the software PickViewer®. The tools of PickViewer® could perform comparison of reflectances and colour coordinates, and PCA on the images, etc. [12-14].

The water, after each cycle of leaching, was also measured for obtaining pH and conductivity values. Moreover, Fourier transform infrared (FTIR) spectroscopy was used to characterize the compound extracted by water leaching (Nicolet Avatar 360 Fourier transform spectrometer).

Table 1. Summary of the artificial ageing performed under UV irradiation and leaching.

Cycle Nr.	UV irradiation	Leaching (hours)
1	72 h	5
2	1° cycle + 96 h	5
3	2° cycle + 168 h	5
4	3° cycle + 168 h	5
5	4° cycle + 672 h	5
6	5° cycle + 336 h	5

3. Results and Discussion

The colour data are reported in the Tables 2-3 in terms of chromatic coordinate values and their changes respectively after each ageing cycle. The differences of the chromatic coordinates and the total colour change expressed by ΔE are calculated in respect to the time 0, i.e. before ageing.

Concerning the L* coordinates, it is possible to observe, from the data of Tables 2 and 3, that there are two different trends: it decreased (ΔL* negative) for untreated specimens and it increased (ΔL* positive) for the thermally treated ones. This means that untreated specimens darkened whereas heat treated specimens lightened as a consequence of ageing. In the first cycles, the changes of L* coordinate are higher in untreated specimens, but going on with the ageing cycles they become higher in the thermally treated specimens.

We can affirm that the at the end of the ageing cycles, i.e. 1008 hours of Solar Box exposure and six leaching steps, thermally treated specimens have undergone greater colour changes than the untreated ones, in particular concerning the L* coordinate.

The a* coordinate (representing the red-green axis) increased constantly for untreated specimens during the entire ageing period suggesting a redness of the wood surface.

This same coordinate had a different behaviour in thermally treated specimens. In the first cycles it didn't change significantly and the values of Δa* were positive and close to zero. In the last two cycles Δa* become negative and its absolute values underwent a little increase: this suggests a decrease of saturation, i.e. a graying of the wood surface.

Lastly, the b* coordinate increased significantly for all specimens and in each ageing cycle. Δb* values were always positive and higher for thermally treated specimens if compared to the untreated ones. An increase of b* (representing the yellow-blue axis) means that the wood surface yellowed; this change was more pronounced in the heat treated specimens.

In conclusion, by discussing the colour data, it is possible to affirm that the thermally treated specimens underwent colour changes higher than the untreated ones and that the values of ΔL* and Δa* mainly determined the ΔE values (total colour change).

Table 2. Chromatic coordinates at the different ageing cycles. Average values and standard deviation are shown.

Specimen	L*	a*	b*
	T=0		
1 (untreated)	72.44±1.01	7.56±0.37	28.23±0.58
2 (untreated)	73.71±1.10	7.22±0.27	27.53±0.53
3 (heat-treated)	40.26±1.06	11.07±0.77	19.82±1.23
4 (heat-treated)	39.12±0.89	10.11±0.51	17.65±0.82
	1° cycle		
1 (untreated)	66.09±1.84	9.07±0.26	34.11±0.76
2 (untreated)	66.30±1.07	9.25±0.38	34.83±0.64

3 (heat-treated)	42.23±1.83	11.25±0.23	25.26±0.88
4 (heat-treated)	39.66±1.16	10.43±0.26	22.19±0.72
	2° cycle		
1 (untreated)	64.87±1.80	10.46±0.29	34.80±0.70
2 (untreated)	65.34±1.26	10.29±0.44	34.55±0.75
3 (heat-treated)	46.01±1.25	11.38±0.22	27.24±0.70
4 (heat-treated)	42.86±1.58	10.92±0.27	24.21±0.80
	3° cycle		
1 (untreated)	63.81±1.90	10.80±0.37	33.10±0.83
2 (untreated)	63.83±1.25	10.80±0.36	33.36±0.49
3 (heat-treated)	48.35±1.36	11.04±0.34	27.60±0.68
4 (heat-treated)	45.60±1.25	11.01±0.28	25.47±0.64
	4° cycle		
1 (untreated)	64.56±1.94	10.74±0.38	32.62±0.81
2 (untreated)	63.96±0.87	10.64±0.34	32.42±0.49
3 (heat-treated)	51.18±1.22	10.24±0.45	27.08±0.59
4 (heat-treated)	47.93±1.45	10.48±0.44	25.52±0.61
	5° cycle		
1 (untreated)	64.12±1.92	10.44±0.42	31.41±0.79
2 (untreated)	63.97±0.13	10.33±0.43	31.22±0.62
3 (heat-treated)	52.92±1.14	9.58±0.47	25.89±0.61
4 (heat-treated)	49.71±1.83	9.92±0.56	24.78±0.72
	6° cycle		
1 (untreated)	64.78±1.70	9.70±0.43	30.44±0.91
2 (untreated)	64.05±1.19	9.50±0.40	30.04±0.58
3 (heat-treated)	55.84±1.20	8.43±0.44	25.76±0.57
4 (heat-treated)	52.10±2.54	9.17±0.87	25.00±0.99

**Table 2.** Changes of the chromatic coordinates as a consequences of the ageing cycles.

Specimen	ΔL^*	Δa^*	Δb^*	ΔE
	1° cycle			
1 (untreated)	-6.35	1.50	5.88	8.78
2 (untreated)	-7.41	2.03	7.30	10.6
3 (heat-treated)	1.97	0.18	5.43	5.78
4 (heat-treated)	0.54	0.33	4.55	4.59
	2° cycle			
1 (untreated)	-7.57	2.90	6.57	10.4
2 (untreated)	-8.37	3.07	7.02	11.3
3 (heat-treated)	5.75	0.32	7.42	9.40
4 (heat-treated)	3.74	0.82	6.57	7.60
	3° cycle			
1 (untreated)	-8.63	3.23	4.86	10.4
2 (untreated)	-9.88	3.58	5.82	12.0
3 (heat-treated)	8.09	-0.03	7.78	11.2
4 (heat-treated)	6.48	0.90	7.82	10.2
	4° cycle			
1 (untreated)	-7.88	3.17	4.39	9.56
2 (untreated)	-9.75	3.41	4.88	11.4
3 (heat-treated)	10.2	-0.82	7.26	13.1
4 (heat-treated)	8.81	0.38	7.88	11.8
	5° cycle			

1 (untreated)	-8.32	2.88	3.18	9.36
2 (untreated)	-9.74	3.11	3.69	10.9
3 (heat-treated)	12.7	-1.49	6.07	14.1
4 (heat-treated)	10.6	-0.18	7.14	12.8
		6° cycle		
1 (untreated)	-7.66	2.14	2.21	**8.25**
2 (untreated)	-9.66	2.28	2.51	**10.2**
3 (heat-treated)	15.6	-2.63	5.94	**16.9**
4 (heat-treated)	13.0	-0.94	7.35	**14.9**

Hypercolorimetric Multispectral Imaging, applied for the first time on wood specimens for evaluating the changes after ageing cycles, gave the images shown in the Figures 1-2 after calibration and processing. Reflectance values are shown in two different points of each sample at the seven calibrated bands from 350 nm (UV) to 950 nm (IR3). To do this, the "Reflectance" tool of the PickViewer® software was applied to show the potentiality of the system, but many other processing options could be applied to investigate the surface characteristics and changes.

In the Figures 1-2, calibrated images of the specimens are shown on the left side of the GUI (Graphical User Interface) of the PickViewer® software. Colour checker and white targets are also shown. On the right side, the processing data can be seen illustrating the reflectance values at the seven bands centred at 350, 450, 550, 650, 750, 850 and 950 nm and the position of the chosen areas in the CIE diagram.

By comparing the reflectance values at the three chosen times, for the untreated specimens, it can be observed that ranging from 0 to 504 h they always decreased. The main variation was observed at 650 nm, i.e. in the orange-red part of the visible spectrum. The chromatic coordinate Y, that represents the lightness in the CIE space, decreased (darkening) in accordance with the spectrophotometric colour measurements previously described.
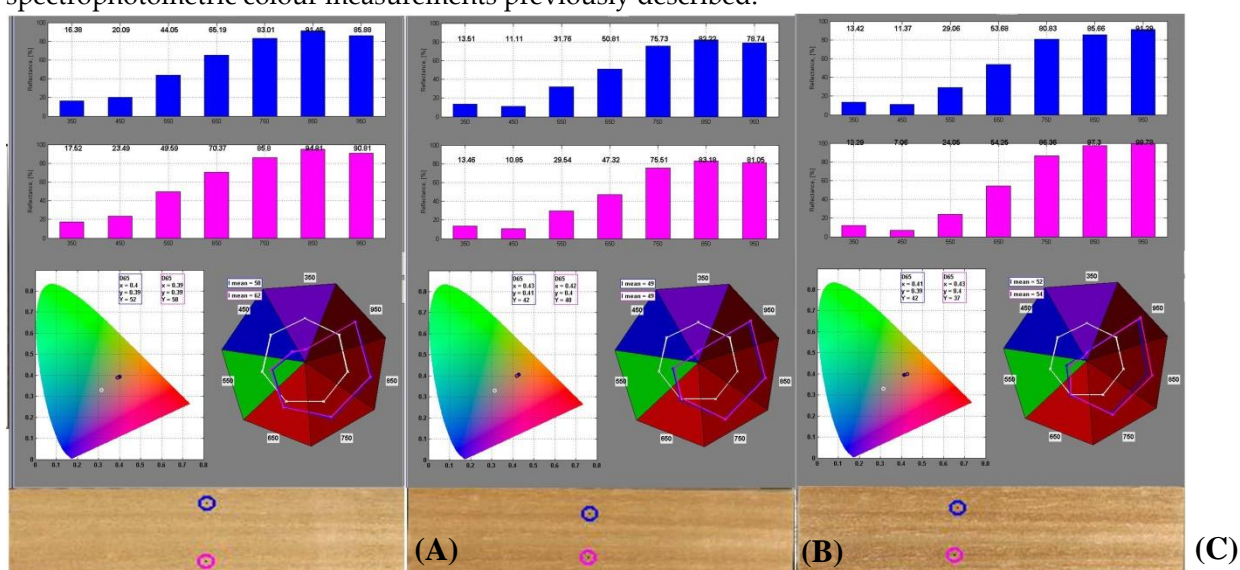
Figure 1. Reflectance and CIE values derived from HMI acquisition, calibration and processing for untreated specimens at 0h (A), 504h (B) and 1008h (C).
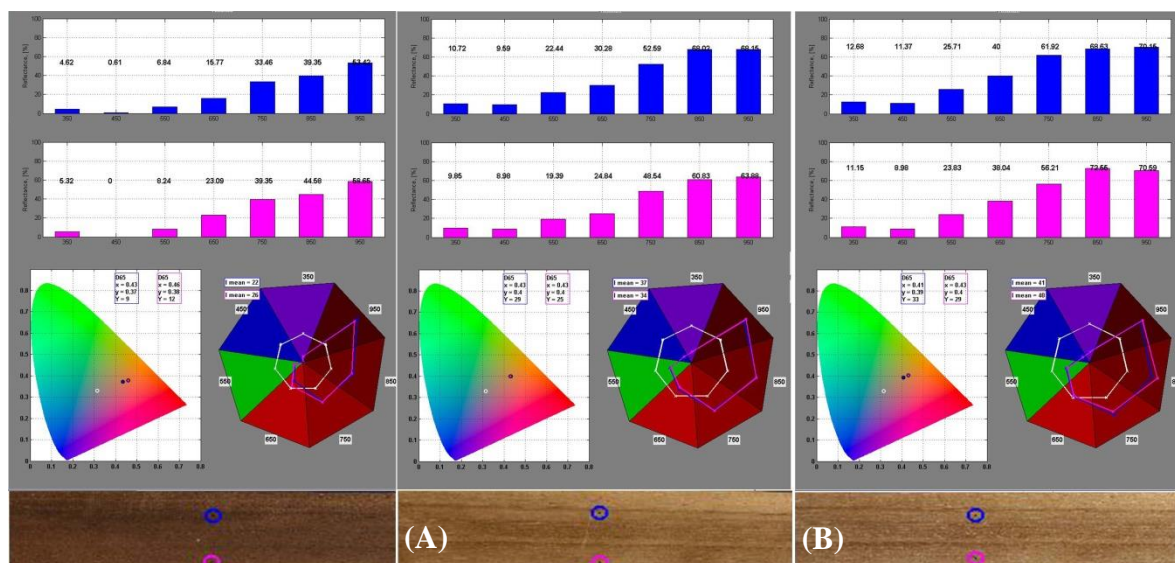
Figure 2. Reflectance and CIE values derived from HMI acquisition, calibration and processing for thermally treated specimens at 0h (A), 504h (B) and 1008h (C).

The values of reflectance exhibit a little decrease also ranging from 504 to 1008 h at 350 up to 650; on the other hand, they increased at 650 up to 950. It may be supposed that at longer exposure times wood surface undergoes chemical changes due to components other than lignin that is the polymer suffering the main degradation under UV radiation [15, 1, 5]. The different behaviour at 1008 h was not observed with the spectrocolorimeter because it occurs in the near infrared part of the electromagnetic spectrum, acquired with the HMI system.

Thermally treated wood specimens showed different behaviour in respect to the untreated ones. The reflectance values increased at each wavelength both at 504 and 1008 h of ageing. The main variations occur at 750 and 850 nm, i.e. in the near infrared region. Concerning the colour values in the CIE space, the increasing of Y (representing lightness) confirms that the heat treated specimens lightened as a consequence of the ageing.

Values of pH and conductivity measured after each cycle of combined Solar Box and leaching ageing are shown in Table 4. They were quite constant during the ageing. pH values were in the acid range of the water scale indicating the presence of weak acids in solution produced during ageing.

Conductivity values were quite low suggesting the presence of few charged species in the leaching solution. Generally they were higher in the water used for leaching untreated specimens (1-2) in respect to that used for thermally treated ones (3-4).

Table 4. pH and conductivity values of the leaching water measured after each ageing cycle.

Cycle Nr.	pH		Conductivity (μS/cm)	
	1-2	3-4	1-2	3-4
1	4.43	4.58	318	136
2	4.38	4.37	110	94
3	4.17	4.01	105	179
4	4.14	4.03	203	96
5	4.34	4.18	165	114
6	4.12	4.25	168	135

Lastly FTIR data are synthesized and discussed in terms of main signatures in the spectra of the solid fraction obtained after centrifugation of the water bath used for leaching.

FTIR spectra revealed the main signatures (data not shown) of wood components, mainly polysaccharides. Bands of lignin are also visible, demonstrating that the leaching caused the removal of wood micro-particles from the degraded surfaces.

4. Conclusion

The weathering cycles changed significantly the colour and the spectral reflectance of the specimen surface. Solar box irradiation causes darkening of the untreated specimens, whereas it causes lightening of the thermally treated ones. With the increase of weathering times, untreated specimen surfaces darken whereas treated specimens lighten so as to tend to have a similar colour or in any case to decrease the chromatic difference that was at the beginning of the weathering tests.

The measured values of conductance were generally higher in the leaching water of untreated specimens and tend to decrease after the first cycles. The values of pH are similar in untreated and treated specimens. FTIR spectroscopy demonstrated that water leaching caused loss of materials from the specimens, mainly from those thermally treated. FTIR spectra exhibit signatures of polysaccharides materials as main compounds. Bands of lignin are also visible. Water leaching seems to remove degraded surface micro-particle of wood.

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