Photodegradation of Three Hardwood Species by Sunlight and Xenon Light Sources

Xin You Liu,[a,b,*] Mihaela Liu,[a] Meng Qi Lv,[a] and Jiu Fang Lv [a,b]

The behavior of three hardwood species was investigated in response to natural and artificial light irradiation. The effects of irradiation were evaluated by their color measurement in the CIELab system and Fourier-transform infrared (FTIR) spectra. Both natural light irradiation and artificial light irradiation induced changes in color, such as yellowing and darkening, in the wood species. Comparative in-time evolution of changes in color during artificial light irradiation and natural light irradiation under indoor conditions resulted in an estimated acceleration index that ranged from 60× to 90×.The FTIR spectra highlighted specific changes in surface chemistry during irradiation. Ultraviolet light from natural or artificial sources primarily resulted in lignin degradation.

Keywords: Photodegradation; Sunlight irradiation; Xenon light irradiation; Color change; Infrared spectrum; Wood

*Contact information: a: College of Furnishing and Industrial Design, Nanjing Forestry University, Str. Longpan No.159, Nanjing, China; b: NFU Academy of Chinese Ecological Progress and Forestry Studies Str. Longpan No.159, Nanjing, China; *Corresponding author: liu.xinyou@njfu.edu.cn*

INTRODUCTION

Wood has a long history of usage and is an important material because of its properties that are linked to visual, tactile, auditory, and olfactory stimulation, as well as its modifiable nature and desirable color (Yang and Liu 2018). However, wood color is highly sensitive to light and temperature. When exposed to light, the wood surface is rapidly degraded because of the strong absorption of ultraviolet (UV) light on the wood structure. Previous reviews by Hon (2001) and Williams (2005) summarize the current understanding of the photochemistry of wood. The UV degradation is initiated by the absorption of light by wood constituent polymers, mainly lignin, which consists of numerous chromophoric structures, including quinones. Pandey and Vuorinen (2008) further indicate that the most widely accepted mechanism underlying lignin photodegradation is the formation of quinone structures.

Various factors affect wood photodegradation, including the type of light source, intensity of irradiation, ambient temperature, and relative humidity, together with the omnipresent oxygen in the atmosphere (Sonderegger *et al.* 2015; Kránitz *et al.* 2016). Sunlight is an important factor for wood photodegradation. Its energy in the form of photons is continuously emitted onto the surface of the earth. While the ultraviolet region of the light wavelengths of solar radiation comprises the smallest proportion of the spectrum (6.8%), it has the highest spectral energy and is the most effective part of the spectrum for photodegradation (Qin and Yu 2009). Comparative studies have been conducted to examine the effect of sunlight and artificial light sources on the weathering of wood. Liu *et al.* (2016) investigated and compared the behavior of Paulownia wood by using different aging tests. Comparative in-time evolution of color changes during an accelerated UV aging test and a simulated natural aging test under indoor conditions

resulted in an estimated acceleration index ranging from 50× to 100×. Tolvaj and Varga (2012) studied the photodegradation of three hardwood species via exposure to sunlight, a xenon lamp, and a mercury vapor lamp. The results indicated that the mercury lamp induced more intense changes, both in color and in infrared spectrum, compared with the other two light sources. Pandey and Vuorinen (2008) examined in detail the photodegradation of wood surfaces by using a xenon lamp and a UV laser emitting at 244 nm. The UV resonance Raman spectra of laser-irradiated wood exhibited similar behaviors as those of the wood irradiated by xenon light—that is, overall broadening and rapid reduction in the intensity of the lignin aromatic structure. The degradation rate caused by the UV laser was relatively high. Tolvaj and Mitsui (2005) compared the effects of natural and artificial light (xenon and mercury lamps) on wood. The results concluded that xenon light simulated sunlight only under long-term irradiation. Ota *et al.* (1996) investigated the color stability of acetylated veneers of kiri (*Paulownia tomentosa* Steud.) irradiated by sunlight and light generated by a mercury lamp. Podgorski *et al.* (1996) evaluated the effect of outdoor and artificial weathering of coated wood by measuring the glass transition temperature. The maximum glass transition temperature was approximately 24 °C.

In addition to the type and intensity of the light source, temperature and humidity also affect the photodegradation of wood. Previous publications (Persza and Tolvaj 2012; Tolvaj *et al.* 2013; Liu *et al.* 2017; Varga *et al.* 2017; Prekleta *et al.* 2018) determined that temperature significantly influenced the degradation of wood. Tolvaj *et al.* (2016) measured the effects of air, relative humidity, and temperature on the photodegradation of beech and spruce wood. The results showed that the presence of vapor and an increase in temperature accelerated the photodegradation of wood.

The natural photodegradation of wood is affected by sunlight as well as geographic position, climate, and other factors. The current study focused on the photodegradation behavior caused by sunlight under indoor conditions through natural simulation of three locations in China and by using artificial light (xenon lamp). Color can be greatly affected by aging; thus, color measurements in the CIELab system have often been employed to monitor wood aging (Roşu *et al.* 2010; Persze and Tolvaj 2012; Teacă *et al*. 2013; Calienno *et al*. 2014; Timar*et al.* 2016). Fourier-transform infrared–attenuated total reflectance (FTIR–ATR) spectroscopy is often employed to reveal chemical changes associated with degradation. Infrared spectra are sensitive indicators of chemical changes, as demonstrated in previous research in wood aging, particularly light-induced natural and accelerated aging (Colom *et al.* 2003; Pandey 2005; Pandey and Vuorinen 2008; Chang *et al.* 2010; Roşu*et al.* 2010; Tolvaj *et al.* 2013; Bonifazi *et al.* 2017). Other techniques also have been used for evaluating wood irradiation. For instance, Baur and Easteal (2014) used electron spin resonance (ESR) spectroscopy to measure the increment of free radicals generated and to monitor wood photodegradation under different sources of light irradiation. Evolved gas analysis–mass spectrometry and pyrolysis–gas chromatography / mass spectrometry with an on line irradiation system were used to determine the effect of irradiation on thermal behavior (Mattonai *et al.* 2019). FTIR and fluorescence spectroscopy were conducted to study irradiation-induced chemical changes in softwood and hardwood (Pandey (2005). In the current study, the similarities and differences in the effects of natural and artificial light were monitored by color measurement and FTIR–ATR spectrometry.

EXPERIMENTAL

Materials

Three wood species — Mongolian oak (*Quercus mongolica*), black locust (*Robinia pseudoacacia*), and white birch (*Betula dahurica*)—from three trees grown in Dandong, Liaoning in northeast China were used for this study. After felling, each log was cut into timber pieces and then kiln-dried to a moisture content of 10% to 12%. These randomly selected kiln-dried wood pieces were sapwood and light in color. A total of 50 test samples for each wood species measuring 150 mm (L) ×100 mm (W) ×10 mm (H) were prepared. These samples were sanded successively with 120-grit, 180-grit, and 240-grit sandpaper. Ten replicates were exposed to each type of photodegradation test, and 10 replicates were assigned as the controls.

Methods

Natural light irradiation procedure

Artificial irradiation testing and natural irradiation testing in three locations were chosen to compare the effect of an artificial light source to that of the regularly applied sunlight. Natural solar irradiation was conducted between September 1, 2018 and April 30, 2019 (the tests are currently ongoing). The three test locations were identified as Dandong, Qingdao, and Nanjing, China. The geographic data and climate information are listed in Table 1. The specimens were exposed to natural sunlight on a special test rack (Model No. NAW 1500; Golden Dragon Aluminum Co., Ltd., Dandong, China) facing south in a vertical position filtered through a window glass, which is generally recognized as a filter of UVB and transmits UVA and visible light; however, the degree of UVA transmission depends on the type of glass (*e.g.* Tuchinda *et al.* 2006; Almutawa *et al.* 2013; Liu *et al.* 2016). After sunlight exposure for 1 month, the specimens were measured in four areas 2 cm from the edge to quantify the color and chemical changes attributable to the direct exposure to sunlight filtered using a glass window. The experimental data pertain to the control unirradiated samples (denoted as 0) and naturally irradiated samples for 1 to 8 months (denoted as 1N, 2N, 3N, 4N, 5N 6N, 7N, and 8N).

Table 1. Geographic Data and Climate Information in Three Test Locations (China Meteorological Association 2019)

Locations		Dandong	Qingdao	Nanjing
Geographic Data	Latitude	40º44’ N	36º18’N	32º05’N
	Longitude	124º47’E	118º48’E	120º02’E
	Elevation (m)	561	25	20
Outside conditions	Daily Total Solar Radiation (K J/m^2)	4.92-17.48	5.86-20.13	5.49-18.97
	Rainy/cloudy/sunny (days)	6/98/140	11/107/124	33/143/66
	Day time ratio	47.24%	47.62%	48.11%
Inside conditions	Temperature (°C)	15-25	20-25	20-25
	Relative Humidity (%)	50-55	50-55	50-55

UV-induced artificial irradiation

Another set of specimens was irradiated with an artificial light source—that is a xenon lamp at 500W/m^2 in the 300 to 800 nm range at 40 °C (black panel) and 50% relative humidity (RH) in a xenon lamp test chamber (OK-XD-100; Hongjin Test Instrument Co., Ltd., Dongguan, China). A quartz glass filter was placed around the lamp to cut off light wavelengths measuring less than 295 nm. Ultraviolet irradiation was conducted based on a programmed cyclic protocol, including the steps of UV irradiation at 40 °C, alternating with dark periods, as well as initial and final conditioning periods (20 °C, 55% RH, no light). After UV exposure for 12, 24, 36, 48, 60, 72, 84, and 96 h, the specimens were measured to quantify the photodegradation occurring as color and chemical changes.

Color measurement

Before and after aging at varying periods, the samples were measured using the CIELab system with a colorimeter (K-M 2600d; Konica Minolta Holdings, Inc., Tokyo, Japan). The *L**, *a**, and *b** color coordinates were calculated based on the CIE standard illuminant D65 and the 10° standard observer with a test-window diameter of 8 mm. The relatively large test window was used to measure the average colors of earlywood and latewood for all species. To ensure that the measurements were recorded at the same position after different periods, all measured areas were established at points 2 cm from the sides. Thus, each sample was measured in four areas, and an average of 40 measurements was obtained.

FTIR investigation

Fourier-transform infrared spectrometry was conducted on an Alpha FTIR Spectrometer (Bruker Vertex 70; Bruker Optik GmbH, Ettlingen, Germany) equipped with an ATR module used to monitor the chemical modification of the wood surfaces after irradiation for different periods. The spectra were recorded from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} and 24 scans. Three replicates from each batch of samples (control and irradiated) were investigated using FTIR. Spectra from three randomly chosen measuring areas were registered for each sample, resulting in nine spectra for each tested variant. The spectra were further processed for baseline correction and smoothing, and the average of nine individually recorded spectra was determined. The average spectra were further normalized (max–min normalization) and compared to emphasize the chemical changes attributable to irradiation. Assignment of characteristic absorption bands was based on the literature. A semi-quantitative evaluation of chemical changes caused by irradiation was made based on the calculated ratios of the integrated areas of some relevant absorption bands. The OPUS software was employed for all spectral processing and calculations.

RESULTS AND DISCUSSION

Monitoring Irradiation by Color Measurement

Figures 1, 2, and 3 present the color changes caused by natural irradiation in three locations. These figures clearly show that natural irradiation under different conditions on different wood species resulted in color changes reflected by the three CIE Lab color coordinates *L**, *a**, and *b**. These color changes evolved over time. Natural irradiation mostly led to yellowing, an increase in *b** value, darkening, and a decrease in *L** value.

However, the values for *a**, the red hue, remained constant. In the first two months, the color quickly changed and eventually slowly changed. The rapid period of discoloration caused by sunlight contributed 44.58% to 61.26% of the total change in black locust, 56.37% to 67.30% in Mongolian oak, and 47.17% to 57.5% in white birch. The notable behavior of the black locust was attributed to its high extractive content. The UV light degraded the extractives, and the degradation products subsequently underwent rapid oxidation. The modified chromophores act as an energy trap that delays the photodegradation of the main components of wood (Németh *et al.* 1992).

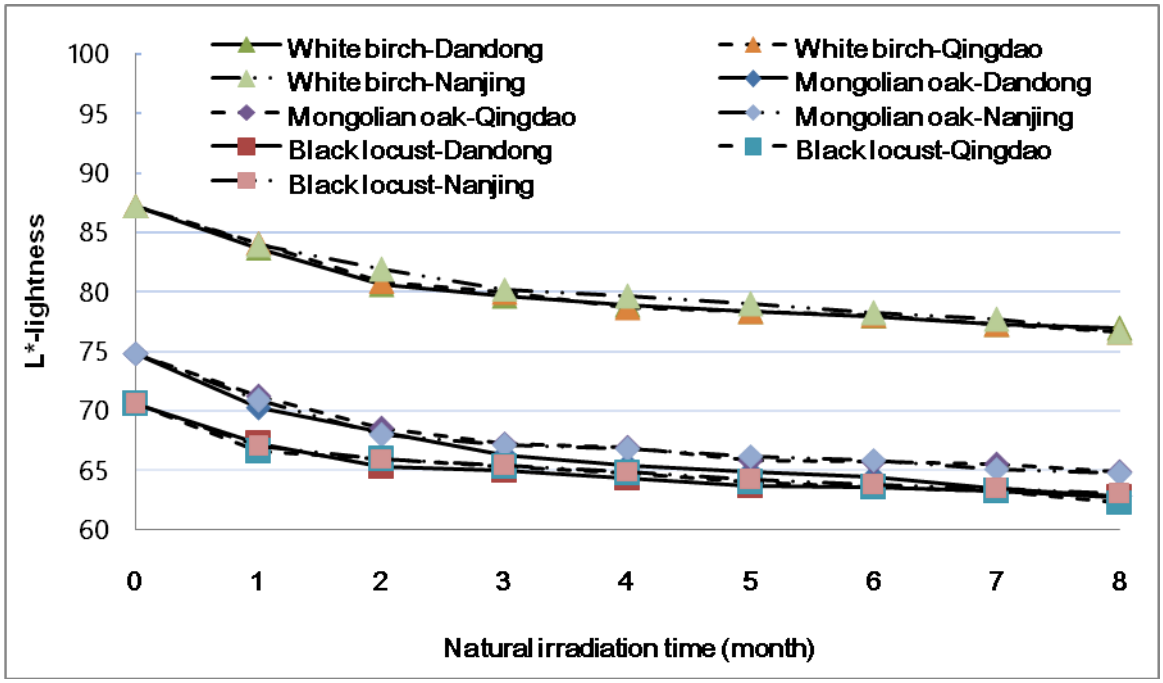

Fig. 1. Change in lightness in three wood species under natural light irradiation in three locations

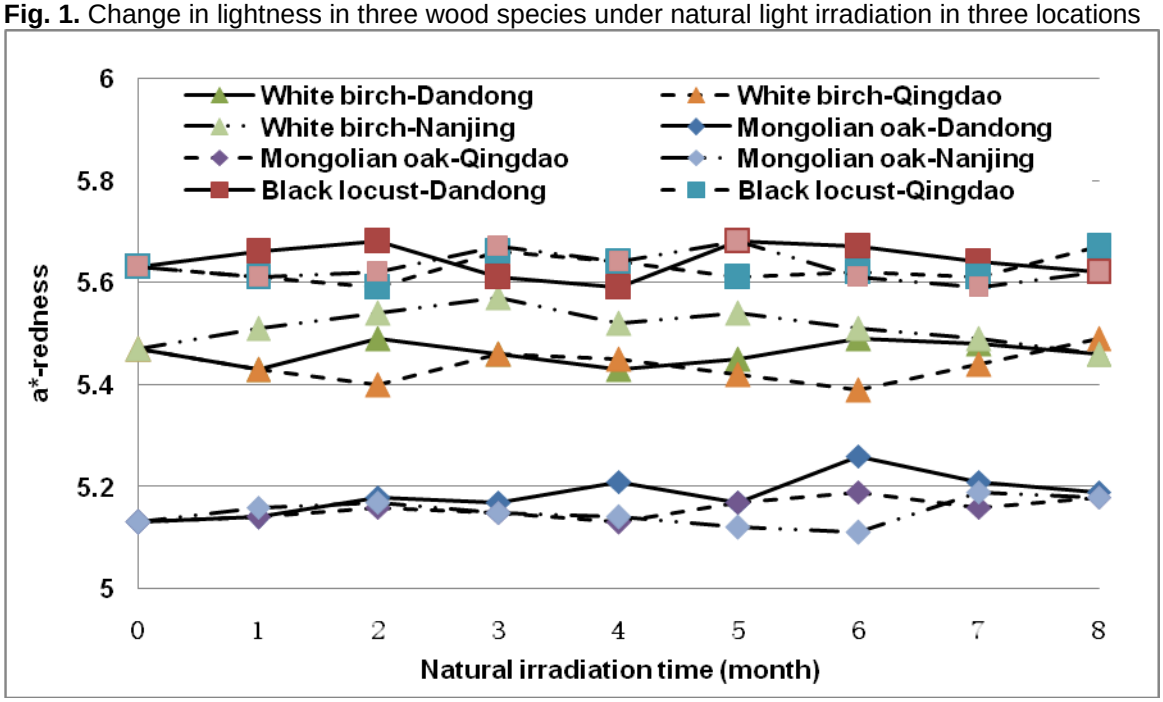

Fig. 2. Change in redness in three wood species under natural light irradiation in three locations

Comparison of the change in color caused by natural light irradiation in the three locations revealed a small difference (Table 2). After irradiation for 8 months, the differences in color (Δ*E*) for the three wood species in three locations (Dandong, Qingdao, and Nanjing) were 18.90, 21.48, and19.01 for Mongolian oak; 16.01, 19.41, and 16.39 for black locust; and 20.54, 21.55, and 21.13 for white birch. The changes in color by natural light irradiation in Qingdao were slightly more noticeable than those in the other two locations. This difference could have been attributed to the difference in sunlight illumination intensity.

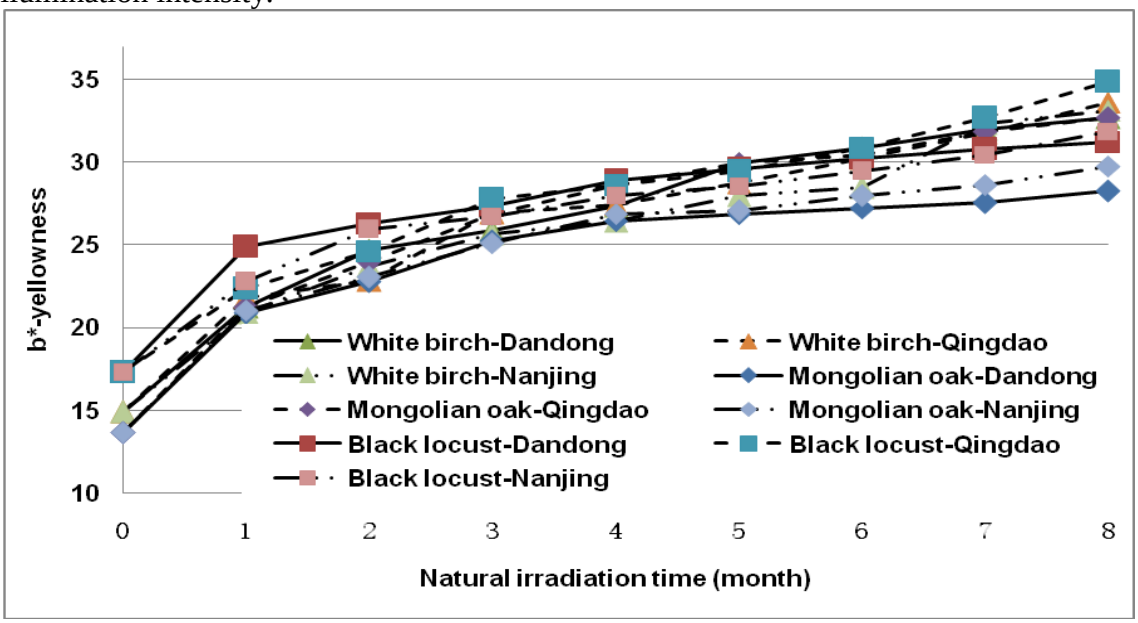

Fig. 3. Change in yellowness in the three wood species under natural light irradiation in three locations

Table 2. Results of Color Changes after the Last Irradiated Term for Three Investigated Wood Species: *L**, *a**, *b** Coordinates and Global Color Difference Δ*E* (average values and standard deviations - in brackets)

Color changes		Artificial irradiated (96h)	Natural irradiated (8 months)		
			Dandong	Qingdao	Nanjing
Mongolian oak	Δ*L**	-9.60 (*0.37*)	-11.96 (*0.29*)	-9.96 (*0.56*)	-10.06 (*0.42*)
	Δ*a**	0.18 (*0.03*)	0.06 (*0.04*)	0.05 (*0.06*)	0.05 (*0.04*)
	Δ*b**	23.65 (*0.87*)	14.63 (*0.51*)	19.03 (*0.79*)	16.13 (*0.92*)
	Δ*E*	25.52 (*1.02*)	18.90 (*0.74*)	21.48 (*0.51*)	19.01 (*0.58*)
Black locust	Δ*L**	-8.54 (*0.64*)	-7.87 (*0.24*)	-8.24 (*0.93*)	-7.54 (*0.29*)
	Δ*a**	0.04 (*0.02*)	-0.01 (*0.05*)	0.04 (*0.01*)	-0.01 (*0.06*)
	Δ*b**	22.94 (*0.83*)	13.94 (*1.18*)	17.57 (*0.88*)	14.55(*0.59*)
	Δ*E*	24.48 (*0.60*)	16.01 (*0.55*)	19.41 (*0.35*)	16.39(*0.46*)
White birch	Δ*L**	-9.96 (*0. 87*)	-10.31 (*0.74*)	-10.66 (*0.98*)	-10.66 (*0.73*)
	Δ*a**	0.10 (*0.03*)	-0.01 (*0.05*)	0.02 (*0.02*)	-0.01 (*0.07*)
	Δ*b**	23.55 (*0.52*)	17.77 (*0.70*)	18.73 (*0.63*)	18.24 (*0.89*
	Δ*E*	25.57 (*0.67*)	20.54 (*0.52*)	21.55 (*0.85*)	21.13 (*0.91*)

The graphs in Fig. 4 present the changes in color in the three wood species under xenon light irradiation. Under artificial xenon light irradiation, the three wood species exhibited changes in color as lightness decreased (with a maximum value of -9.96 units), yellowness increased (with a maximum of 23.65), and redness slightly increased at 40 °C. Similar to those under natural irradiation, the changes in color sharply changed for 12 h and then linearly changed. The rapid period of discoloration caused by xenon light contributed 55.7% to the total change for Mongolian oak, 51.6% for black locust, and 56.4% for white birch.

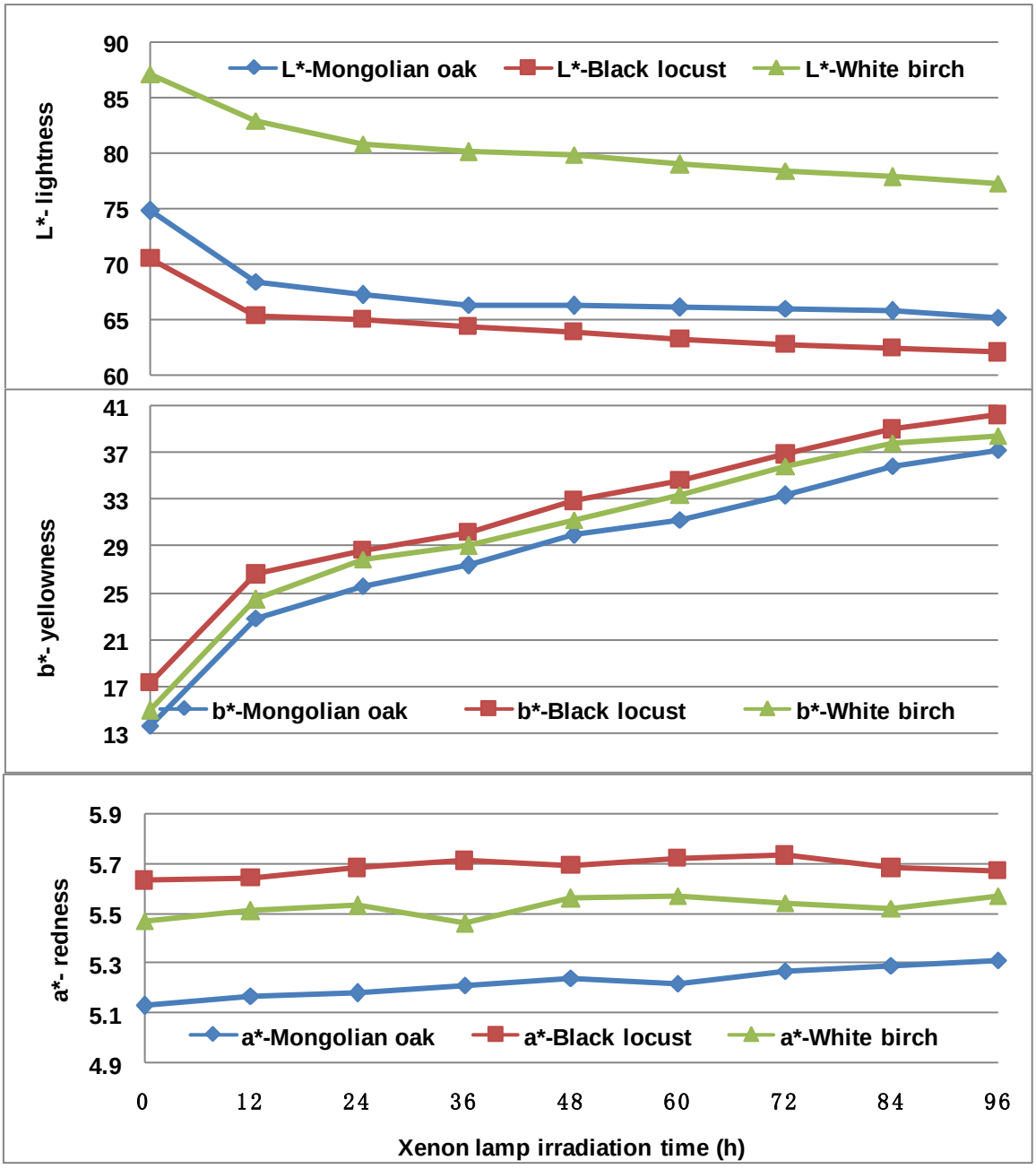

Fig. 4. Changes in color in the three wood species under xenon lamp irradiation

Comparison of Changes in Color between Natural and Artificial Irradiation

As previously presented, the changes in color caused by natural irradiation and conditions indoors at normal ambient temperature (approximately 20 °C ±5 °C) were more closely related to the color changes resulting from xenon lamp artificial lighting filtered using a quartz glass filter. The artificial and natural irradiation relation was effectively emphasized in the combined graph in Fig. 5, in which the in-time evolution of color changes (ΔL, Δb, and ΔE) was mirrored against the vertical axis crossing two opposite horizontal time axes (with different scale units) at the 0 common point. The x-axis on the left is referred to artificial irradiation, with each unit representing exposure for 12h. The x-axis on the right is referred to natural light irradiation filtered by glass at the Qingdao site, with each unit representing exposure for 1 month. Horizontal lines with different values of color changes allowed the interpolation of corresponding irradiation times for approximately similar color changes.

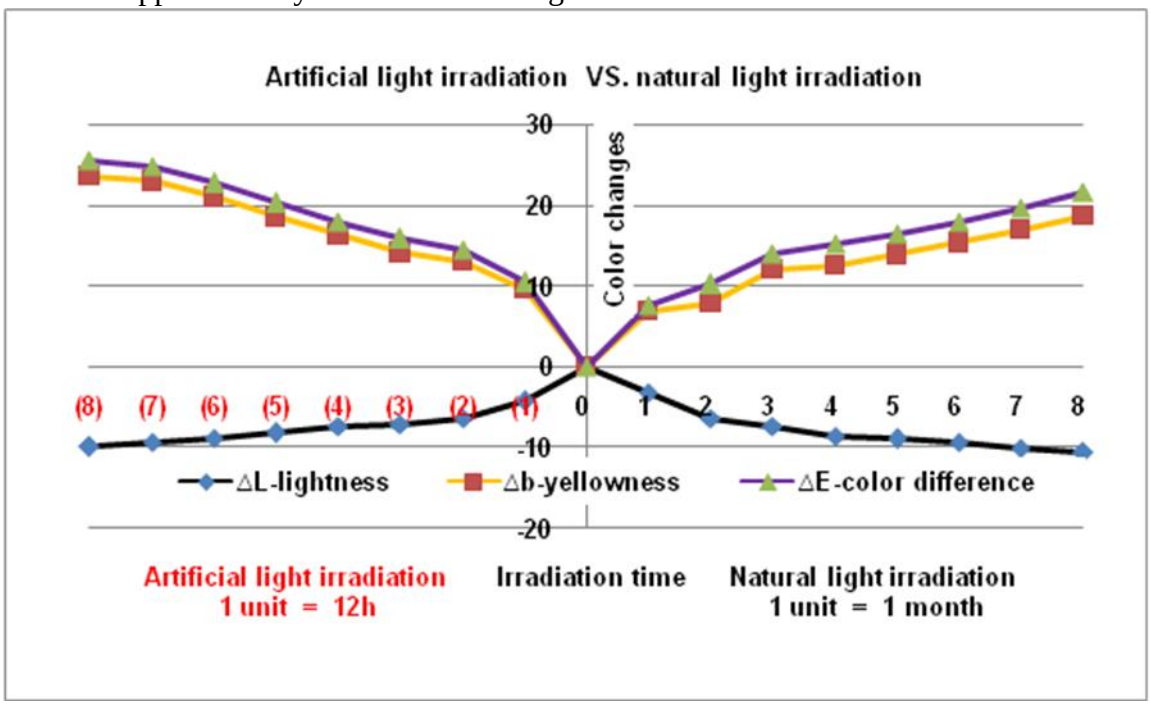

Fig. 5. Comparative in-time evolution of color changes in white birch wood under artificial light irradiation and natural light irradiation in Qingdao

For instance, a color difference of 10 units resulting from natural exposure for almost 2.5 months was obtained from xenon lamp exposure for approximately 12 h. However, in this color difference, only the contribution of increased yellowness was similar, and the curves of ΔE and Δb were almost parallel. Similarly, an increase of 17 units in yellowness caused by natural light irradiation for 8 months was achieved by artificial light irradiation for approximately 60 h. This change led to an acceleration index of approximately 60× to 90× for xenon light exposure relative to that of natural sunlight exposure (12 h of light per 24 h day). The acceleration indexes were also calculated for the other two wood species, approximately 63× to 84× for black locust and approximately 70× to 87× for Mongolian oak.

FTIR Investigation of Surface Chemical Modifications

Color changes caused by natural and artificial light irradiation resulted from the various and specific chemical processes undergone by the main chemical components of both wood and extractives. The degradation of the main components (cellulose, hemicellulose, and lignin) of wood was monitored *via* FTIR spectroscopy. The different light source irradiation procedures in this research induced specific chemical changes as reflected by the comparative spectra of the control and irradiated samples (after the last irradiation term and the spectrum of artificial irradiated sample is an average spectrum of the spectra from three locations)of three wood species (Fig. 6).

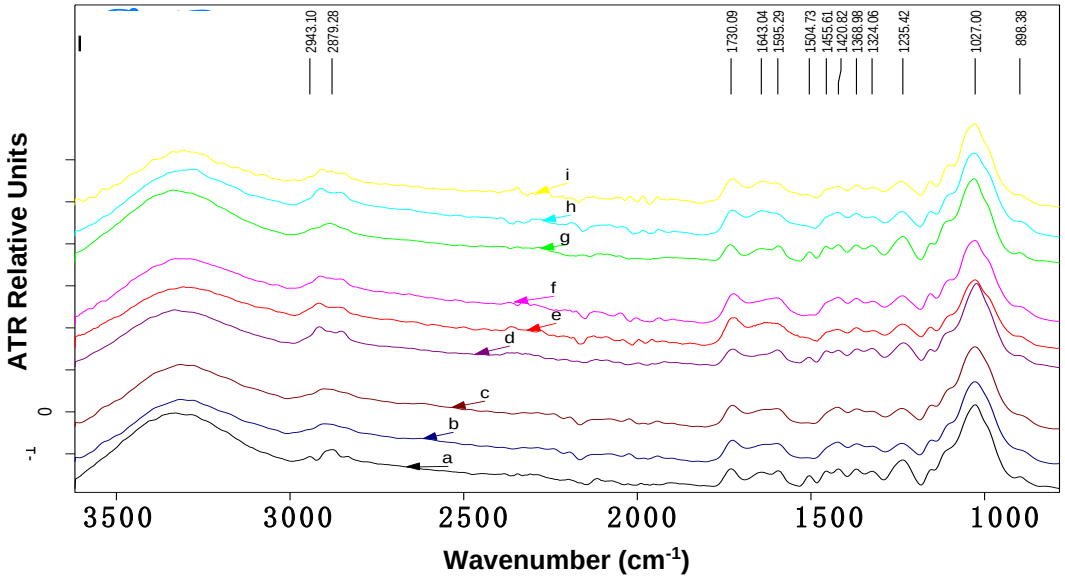

Fig. 6. Comparative spectra of control and irradiated samples for three wood species: a, b, and c for Mongolian oak (control, artificially irradiated, and naturally irradiated); d, e, and f for black locust (control, artificially irradiated and naturally irradiated); g, h, and i for white birch (control, artificially irradiated, and naturally irradiated)

Within the 3500 to 2000 cm^{-1} range, all spectra showed strong bands of H-bonded O-H stretching absorption within the 3300 to 3400 cm^{-1} range and C-H asymmetric and symmetric stretching of methylene groups within 2943 to 2880 cm^{-1}, usually appearing as two sharp absorption peaks. Comparison of naturally irradiated spectra (average variation of three locations) with artificially irradiated spectra indicated the appearance of similar alternate peaks, such as 1595 and 1505 cm^{-1}.

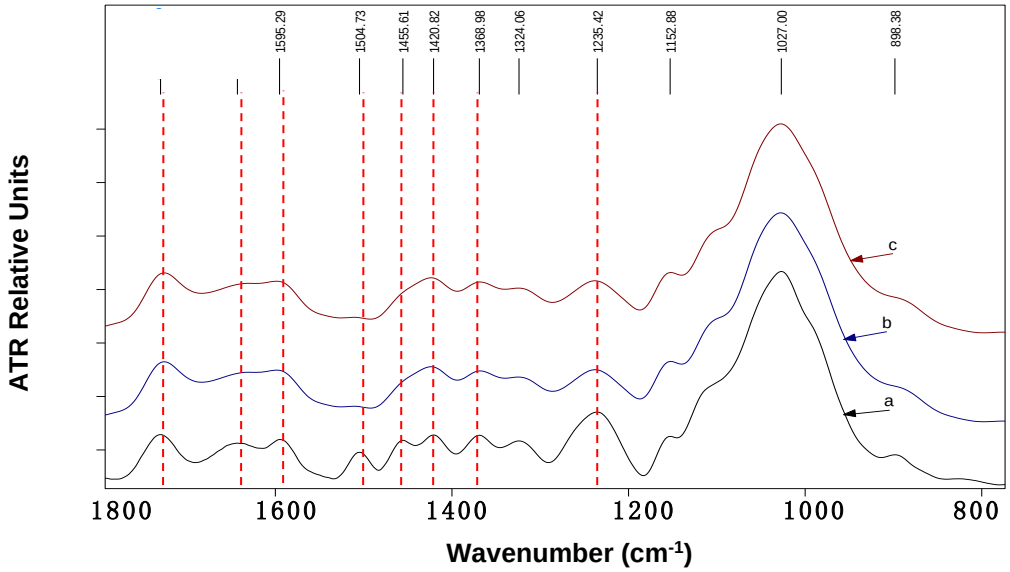

Fig. 7. Comparative spectra (the fingerprint region 1800 to 800 cm^{-1}) of control and irradiated Mongolian oak samples: a. control, b. artificial irradiated, c. natural irradiated

In Fig. 7, the more obvious comparative spectra of control and irradiation Mongolian oak samples indicate that lignin is the main UV absorber in wood and also the most sensitive to photodegradation. The most important lignin-related absorption bands are 1595 cm^{-1} (aromatic skeletal vibration of lignin plus C=O stretch), 1505 cm^{-1} (aromatic skeletal vibration of lignin), 1455 cm^{-1} (aromatic CH deformation and asymmetric bending of CH_3 in lignin), and 1324 cm^{-1} (syringyl ring breathing with CO stretch and C_1–O vibration in syringyl derivatives of lignin and C–H vibration in cellulose). Lignin also contributes to other peaks that can be identified in the spectra, as follows: 1422 cm^{-1} (C–H deformation in lignin and carbohydrates; CH_2 bending in cellulose), 1240 cm^{-1} (C–O stretch in lignin and xylan, syringyl ring). Following the natural and artificial irradiation, all these bands decreased, with the largest decrease (almost disappearing) being clearly visible for the skeletal vibration at 1505 cm^{-1}, which is mostly specific and frequently employed in following lignin degradation (*e.g.* Pandey and Vuorinen 2008; Tolvaj *et al.* 2013; Timar *et al.* 2016). The band at 1595 cm^{-1} also decreased, becoming less evident. Thus, there was a gradual increasing trend of the absorption band at 1643 cm^{-1} (conjugated C=O and C=C, aromatic ketones). The increase in number of absorption bands for conjugated (1643cm^{-1}) and unconjugated carbonyl (1730 cm^{-1}) groups is related to the formation of different carbonyl groups containing chromophores by oxidative processes. The absorption band at about 1370 cm^{-1} can be assigned to C-H deformation in cellulose and hemicelluloses, which mostly represent these compounds, remained practically unchanged following light-induced aging. This band is thus often employed as an internal standard (*e.g.* Pandey 2005). Therefore, variations in the ratios A1506/A1370 (lignin/ holocellulose) and A1730/A1370 (carbonyl/ holocellulose) are most often employed to evaluate chemical changes associated with photodegradation (*e.g.* Pandey and Vuorinen 2008; Chang *et al.* 2010).

Table 3. Variations in Selected FTIR Ratios Following the Irradiation of Three Wood Species (values reported relative to those of the control)

FTIR Ratios	Control samples	Irradiation samples					
		Artificially irradiated (96h)			Naturally irradiated (8 months)		
		Mongolian oak	Black locust	White birch	Mongolian oak	Black locust	White birch
A1505/A1370	1.00	0.07	0.05	0.02	0.05	0.03	0.04
A1505/A3400	1.00	0.22	0.18	0.15	0.21	0.16	0.19
A1730/A1370	1.00	1.55	1.46	1.51	1.37	1.42	1.56

Table 3 lists the ratios of the integrated areas of some relevant absorption bands, as values relative to the corresponding values of the control samples. These values highlight and allow the comparison of chemical changes following irradiation under different conditions. They present strong similarities in chemical changes occurring under the effect of natural and artificial light and slight differences in ratios among the three wood species.

Light from natural or artificial sources primarily led to lignin degradation, as reflected by decreases in the relative ratios of lignin/carbohydrates (A1505/A1370) and lignin/hydroxyl (A1505/A3400) from 1 (the value of the control) to 0.02–0.07 and in the relative ratios of lignin/hydroxyl (A1505/A3400) from 1 (the value of the control) to 0.15–0.22. These reductions roughly suggest lignin degradation in the proportion of about 90% on the surface of the samples exposed to light. Oxidative processes following lignin degradation result in the formation of carbonyl-containing chromophores, as reflected by the increase in the relative ratio A1730/A1370 from 1.00 (the value of the control) to the range 1.37 to 1.56.

CONCLUSIONS

1. Both artificial light and natural irradiation caused wood to undergo color changes such as yellowing and darkening. However, the examined wood species varied because of the difference in quantity of extractives.
2. Comparison of the color changes caused by natural light irradiation in three locations showed that the color changes produced in Qingdao had a slightly higher value because of almost similar temperature and humidity but higher illumination intensity under three indoor conditions.
3. Comparative in-time evolution of the changes in color for three wood species by artificial light irradiation and natural light irradiation under indoor conditions led to the estimation of an acceleration index that ranged from 60× to 90×.
4. Fourier-transform infrared spectroscopy differences highlighted the changes in specific surface chemistry occurring during these irradiation tests. Ultraviolet light from natural or artificial sources primarily caused lignin degradation, as reflected by decreases in lignin and carbohydrates, followed by oxidative processes that led to the formation of carbonyl-containing chromophores.

ACKNOWLEDGMENTS

This work was supported by Nanjing Forestry University Foundation for Basic Research (Grant No. 163104127).

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Article submitted: May 10, 2019; Peer review completed: June 29, 2019; Revised version received and accepted: July 5, 2019; Published: July 10, 2019.
DOI: 10.15376/biores.14.3.6909-6922