

STUDY OF THE DISCOLORATION BEHAVIOUR OF TEAK
WOOD (*TECTONA GRANDIS* LINN. FIL.) CAUSED BY
SIMULATED SUNLIGHT

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ABSTRACT

To investigate the discoloration behaviour of teak (*Tectona grandis* Linn. fil.) during irradiation, teak veneer specimens were exposed to xenon lamp light simulating sunlight for 80 h and subsequently analysed with a colorimeter. The colour changes were explained by recording Fourier transform infrared (FTIR) and gas chromatography-mass spectrometry (GC-MS) spectra at different exposure times. Forty hours of simulated irradiation marked a key point in the colorimetric analysis. During irradiation of 40 h, the increase in the CIELAB parameters Δa^* and Δb^* originated from the generation of carbonyl derivatives that were determined by FTIR analysis. Besides that, by combining GC-MS and microscopic analyses, the decrease in ΔL^* in the first 20 h resulted from extractives migrating from the interior to the surface, and that the subsequent increase in ΔL^* mainly arose from volatilization of the extractives. Additionally, the decrease in Δb^* after another 40 h of irradiation was mainly due to decreasing volatile extractive contents. Our conclusion reveals the causes of photo-induced discoloration in teak.

KEYWORDS: Teak wood (*Tectona grandis* Linn. fil.), discoloration, FTIR, GC-MS.

INTRODUCTION

Teak wood (*Tectona grandis* Linn. fil.), as a furniture and indoor decorative material, is commonly found attractive for its beautiful colour and unique texture. Wood colour and its fastness are some of the most important aesthetic aspects of wooden products used in interiors (Baar and Gryc 2011). Wood colour is important for routine actions like wood identification and aesthetic considerations, and it sometimes determines a wood's market value (Zhao et al. 2010).

Wood colour can change dramatically in response to various factors (sunlight, moisture, temperature, microorganisms, contact with metals, etc.). From the perspective of wood atmospheric degradation, the most important influence on colour change is the ultra violet (UV) component of sunlight, which induces photochemical reactions in wood (Hon and Chang 1985, Kataoka et al. 2007, Müller et al. 2003, Tolvaj et al. 2011). This study addresses the teak veneer surface discoloration caused by simulated sunlight. Teak wood, as one of the most valuable wood types, is different from other wood species. Upon irradiation with sunlight, not only will teak boards not become dry and deformed, but their colour will change to unattractive golden yellow from its initial brown. The unique colour of teak wood has increased its market value. Therefore, understanding the factors that affect wood colour is a primary challenge in increasing the added value of wood (Duan 2005).

Numerous investigations of the surface changes of commercial woods during weathering have been reported. According to these studies, lignin and extractives are the most sensitive components to sunlight because they contain many chromophoric groups, including aromatic skeletons, carbonyls, biphenyls, and ring-conjugated double-bond structures (Huang et al. 2012b, Pandey 2005b, Timar et al. 2016). During irradiation, these chromophoric groups can absorb sunlight to form new aromatic compounds and free radicals, which can react with oxygen and lead to surface colour changes in the wood (Oltean et al. 2008, Pandey and Vuorinen 2008). Additionally, extractives play an essential role in the colour changes of wood. Variations in the contents and types of extractives of wood species can cause different colour changes during irradiation (Baar and Gryc 2011, Baar et al. 2014, Chang et al. 2010a, Pandey 2005a). Furthermore, research has indicated that extractives function as antioxidants and protect the wood against photodegradation, thereby reducing surface colour changes. Researchers observed that, after extraction of water-extractable compounds, the exposed wood surface became darker than that of unextracted samples (Chang et al. 2010a, Nzokou and Kamdem 2006).

Based on the aforementioned supposition, the aim of this paper was to investigate the possible reason behind the change in colour of teak wood to golden yellow during irradiation. Additionally, the intensity of chemical changes at different stages of exposure was analysed. The results of our studies can enhance understanding of the photodegradation processes in teak wood and facilitate the development of an economical, simple, effective and regulatable technology for wood discoloration.

MATERIALS AND METHODS

Materials

All samples were teak wood veneers acquired from Guangdong Yihua Wood Incorporated Company. Wood specimens measuring 60 mm (length) × 20 mm (width) × 0.06 mm (thickness) were prepared and exposed to artificial sunlight in an LP/SN-500 (Shanghai) xenon-lamp test chamber. The temperature of the chamber was $(50 \pm 2)^{\circ}\text{C}$, and the irradiation durations were 10 h, 20 h, 30 h, 40 h, 50 h, 60 h, 70 h and 80 h. After their colour was measured, all of the veneer samples were shattered and sieved through a 40-mesh screen to remove the larger particles and through a 60-mesh screen to remove the fine particles for analysis.

The wood powder was extracted with methanol solution in an ultrasonic cleaner for 30 min. The liquid extractives obtained after filtration were analysed directly by gas chromatography-mass spectrometry (GC-MS). All teak powder was stored in a dark room at ambient temperature until required for further experimentation.

Colorimetric analysis

The surface colour of wood samples exposed to xenon light for different periods was measured with a colorimeter (WSC-S, Shanghai). According to the CIELAB colour system, negative and positive values of Δa^* indicate a tendency of the surface to become greenish and reddish, respectively, and negative and positive values of Δb^* indicate a tendency of the surface to become bluish and yellowish, respectively. A negative lightness stability, ΔL^* , indicates a darker surface. The colour difference ΔE^* was calculated according to Eq. 1.

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

FTIR spectral analysis

The surface chemical changes in teak wood during irradiation were monitored with a Nicolet Impact 400 spectrometer (Avatar 330, USA) using the KBr method. All spectra were measured at a resolution of 8 cm^{-1} , using 64 scans and a $4000\text{-}400 \text{ cm}^{-1}$ scan range per specimen. The peak intensities of the IR peaks were calculated relative to the baselines.

Chemical composition analysis

Changes in the chemical composition of irradiated teak extractives were analysed with Thermo Trace 1300 ISQ QD GC-MS system (USA) equipped with a TG-5MS capillary column ($30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \text{ }\mu\text{m}$) from Thermo.

Extractives were purged into the GC injector with a constant helium gas flow of $1.0 \text{ ml}\cdot\text{min}^{-1}$. The conditions for GC-MS were as follows: the injector temperature was 250°C ; the GC-MS interface temperature was 240°C ; the column temperature programme was an initial temperature of 50°C (for 3 min), an increase from 50°C to 240°C (heating rate $10^\circ\text{C}\cdot\text{min}^{-1}$) and 240°C for 10 min. For mass spectra analysis, electronic ionization at 70 eV and a 230°C ion source temperature was used.

For identification of analytes, the National Institute of Standards and Technology (NIST) software was used.

Microscopic analysis

A teak wood tangential section of $20 \text{ }\mu\text{m}$ thickness was cut on a sliding microtome, and the specimens were extracted with methanol in an ultrasonic cleaner for 30 min. Sections were then stained in 0.5% safranin prepared in 50% ethanol. Stained sections were dehydrated and mounted in DPX mountant for microscopic examination. The characteristics of the oils were also observed on a stereoscopic microscope.

RESULTS AND DISCUSSION

Visual observation

Stereoscopic microscope photos of teak veneer treated for different irradiation times are shown in Fig. 1. The colour of the wood samples changed greatly after xenon irradiation. The teak samples became yellower and redder as the irradiation time increased. A distinct change in colour to a golden yellow was observed after light exposure for 40 h. The brightness of the teak veneer increased slightly as the irradiation time reached 80 h, but the overall appearance tended to be more stable.

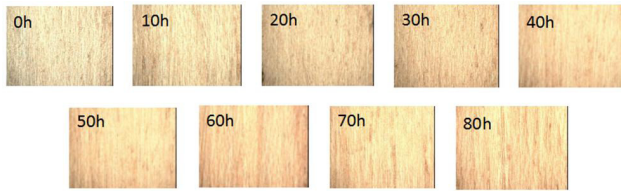


Fig. 1: Stereoscopic microscope photos of teak wood treated for different irradiation times.

The unique golden-yellow of teak wood is the most popular colour among consumers for teak wood furniture (Wen et al. 2017). This picture shows that after irradiation for 40 h, the colour that consumers prefer was no longer generated rapidly. Therefore, in this paper, irradiation for 40 h was considered the most effective and time-saving approach for producing golden yellow teak wood.

Surface colour changes

Changes in the colour parameters ΔL^* , Δa^* , Δb^* and ΔE^* of teak veneer after artificial sunlight exposure for different durations are presented in Fig. 2. Changes in the ΔL^* values imply that the surface of the teak veneer became brighter or darker during the exposure (Peng et al. 2014). The ΔL^* values slightly decreased during the first 20 h and then sharply increased. It is suggesting that the sample first darkened and then turned bright.

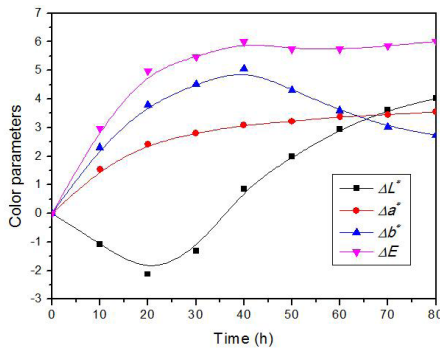


Fig. 2: Colour changes due to irradiation time of teak wood.

A positive change in Δa^* means the colour turned reddish, whereas a negative value indicates a shift toward green. The changes in Δa^* values in Fig. 2 imply that the colour of the teak veneer surface shifted towards red during the exposure. The Δa^* values increased rapidly within the first 40 h and then increased slightly thereafter. Generally, all wood samples became reddish after sunlight exposure, consistent with previous studies (Tolvaj et al. 2010, 2011).

A decrease in Δb^* indicates that the wood surface became bluish, whereas an increase means a shift towards yellowish. As a change in b^* is the main colour alteration induced by the degradation of lignin, all wood species showed considerable changes in their b^* parameter (Stark and Matuana 2004). The trends of Δb^* were similar to those of Δa^* except during the last 40 h, during which the Δb^* values decreased continuously. An exposure time of 40 h appears to be a key point for the change towards yellow.

Changes in ΔE^* values in Fig. 2 present the total colour differences (ΔE^*) in teak veneer during exposure. Caused by the fluctuations in the ΔL^* , Δa^* and Δb^* values, the ΔE^* of teak increased sharply within the first 40 h of irradiation and then tended to stabilize. The reasons for the increase in ΔE^* may be complicated. On the one hand, the increase is likely attributable to the generation of new derivatives related to the extractives and lignin, containing new carbonyl and carboxyl chromophore groups (Colom et al. 2003, Huang et al. 2012a, Huang et al. 2012b). On the other hand, the increase was also effected by volatilization of the original compounds in the extractives. A specific discussion is presented further.

Surface chemical changes

The colour changes on the wood surface reflect the chemical structural changes during light irradiation caused by photo-chemical reactions (Kataoka et al. 2007, Müller et al. 2003, Pandey 2005a, Pandey 2005b). Fig. 3 shows the FTIR spectra of teak powder after 0 h, 10 h, 20 h, 40 h and 80 h of irradiation under xenon light. Obvious transmittance changes are observed in the fingerprint region from 800 cm^{-1} to 1800 cm^{-1} . The light sensitivity of the carbonyl structure (C=O) and aromatic ring (C=C) are particularly noticeable.

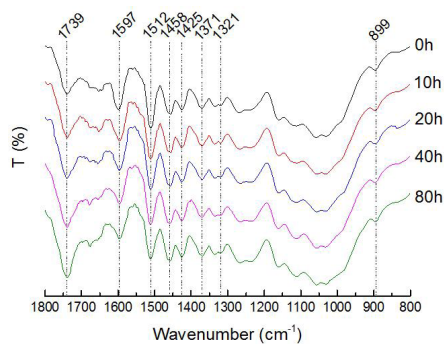


Fig. 3: ATR-FTIR spectra of teak wood during irradiation with xenon light.

The transmittance bands at 1597 cm^{-1} and 1512 cm^{-1} were assigned to the C=C aromatic skeletal vibration of lignin (Liu et al. 2017). Additionally, a substantial increase was observed in the intensity of the band at 1739 cm^{-1} for the C=O groups of unconjugated ketones in derivatives of lignin. The transmittance bands at 1458 cm^{-1} and 1425 cm^{-1} represents $-\text{CH}_2$ and $-\text{CH}_3$ asymmetric bending vibrations, and the bands at 1371 cm^{-1} and 899 cm^{-1} are assigned to the $-\text{CH}$ in-plane and out-of-plane bending vibrations of carbohydrates (Liu et al. 2015) reported that the changes in C=C and C=O bonds mainly signified destruction of the original chromophoric system of wood and the generation of new chromophores, inducing desirable discoloration.

To quantify the rate of C=C group decay and C=O group formation attributable to light irradiation, the relative changes in the ratio between the intensities of the C=C and C=O peaks in the FTIR spectra of teak samples irradiated for different durations were calculated by determining the ratio of C=C groups at the reference bands (1597 cm^{-1} and 1512 cm^{-1}) and the carbonyl absorption reference band (1739 cm^{-1}) against carbohydrates (1371 cm^{-1}). The peaks at 1371 cm^{-1} and 899 cm^{-1} were mainly due to carbohydrates, with no substantial contribution from lignin. Photo-irradiation did not substantially affect these carbohydrate peaks, especially irradiation over a short time. Therefore, we used the reference band (1371 cm^{-1}) as an internal reference, in accordance with previously reported methods (Pandey 2005b, Pandey and Pitman 2003).

The lignin/carbohydrate relative intensity ratios (I_{1512}/I_{1371} and I_{1597}/I_{1371}) decreased rapidly with increasing irradiation time, more obviously at the initial stages (Tab. 1).

Tab. 1: Changes in the relative intensity ratios of the absorption peaks in the FTIR spectra of teak wood during irradiation.

Irradiation time (h)	I_{1321}/I_{1371}	I_{1425}/I_{1371}	I_{1458}/I_{1371}	I_{1512}/I_{1371}	I_{1597}/I_{1371}	I_{1739}/I_{1371}
0	1.00	1.00	1.00	1.00	1.00	1.00
10	0.99	1.00	1.01	0.91	0.91	1.11
20	1.01	1.02	1.01	0.86	0.85	1.26
40	1.01	1.01	1.00	0.83	0.78	1.50
80	1.00	0.99	0.98	0.78	0.73	1.79

The relative intensity ratios of I_{1512}/I_{1371} and I_{1597}/I_{1371} decreased by 0.17 and 0.22, respectively, after the specimen was irradiated for 40 h, whereas they decreased by 0.05 after irradiation for another 40 h; thus, the degradation of lignin was faster during the first 40 h of irradiation. The relative concentration of the carbonyl groups (I_{1739}/I_{1371}) increased by 0.50 after irradiation for 40 h, whereas it rose by 0.29 after irradiation for another 40 h. This result may have been due to the rapid photo-degradation of lignin and the generation of chromophoric carbonyl groups during the first 40 h of irradiation, followed by a slight increase in the amount of chromophoric carbonyl groups. The relative intensity ratios of I_{1321}/I_{1371} , I_{1425}/I_{1371} and I_{1458}/I_{1371} remained almost unchanged for methylene, methyl group is insensitive to light.

Obviously, the colour changes of the teak wood surface during simulated exposure were closely related to lignin. The trends of Δa^* and Δb^* during irradiation for 40 h were completely consistent with decreasing lignin concentrations and with an increase in the number of C=O groups. Thus, the degradation of lignin was accompanied by the generation of carbonyl derivatives, which could change the total light-absorbing wavelength of the teak veneer and alter the colour towards red and yellow. Moreover, the rapid changes in chromophore concentrations during irradiation for 40 h further evidence that 40 h is the most desirable time for colour change.

Chemical composition analysis

Photo-induced changes in wood colour can indicate changes in the chemical composition of the extractives in the wood (Chang et al. 2010b). In this study, the chemical constituents and

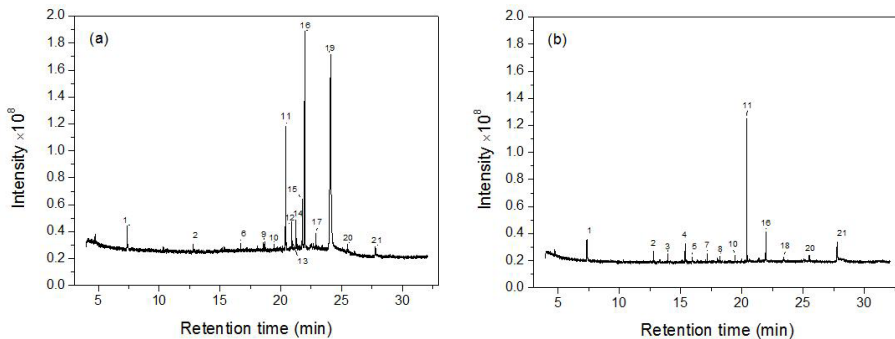


Fig. 4: GC-MS chromatograms of the extractives of teak before irradiation (a) and after 80 h of irradiation (b).

relative contents of extractives were determined by GC-MS to study the relationship between the change in colour and the change in composition of extractives under light irradiation.

The GC-MS chromatograms of the extractives of teak before and after 80 h of irradiation are presented in Fig. 4. The composition and content of extractives changed greatly after 80 h of exposure compared with those of unexposed samples. The chromatographic analysis results are presented in Tab. 2, which includes a list of compounds with the similarities greater than 70%, their retention times and their mean peak intensities in the chromatograms of the three samples.

Tab. 2: GC-MS analysis of extracts under different durations of light radiation.

Peak no.	Retention time (min)	Compounds	Intensity	
			Before irradiation	After irradiation
1	7.35	Phenol	280442121	222793281
2	12.80	Phthalic anhydride	2847843	8695775
3	13.95	Vanillin	-	8911979
4	15.38	Benzaldehyde, 3,4-dimethoxy-, methylmonoacetal	-	131791797
5	15.94	3-Hydroxy-4-methoxybenzoic acid	-	2013823
6	16.68	Cedrol	2366491	-
7	17.18	4-Hydroxy-3,5-dimethoxy benzaldehyde	-	7767438
8	18.03	Coniferyl aldehyde	-	1065851
9	18.67	Anthracene	4015369	-
10	19.44	Diisobutyl phthalate	1076459	3481348
11	20.40	Dibutyl phthalate	135395371	156383800
12	20.89	4a-Methyl-1-methylene-1,2,3,4,4a,9,10,10a-octahydrophenanthren	25657345	-
13	20.95	6-Hydroxy-6-phenyl-tricyclo[3.3.1.0(3,7)]nonan-2-one	2201625	-
14	21.23	4'-Ethyl-4-biphenylcarboxylic acid	37723729	-
15	21.77	Tricyclo[5.2.0.0(2,5)]nona-3,8-dien-6-ol, 6-phenyl-, (1 α 2 β 5 β 6 β 7 α)-	56938432	-
16	21.96	2-methyl-9,10-Anthracenedione	252958334	28682319
17	22.90	2-(1,1-dimethylethyl)-9,10-Anthracenedione,	14903199	-
18	23.40	1-formylanthraquinone	-	1340492
19	24.10	Squalene	656204903	-
20	25.51	2,2'-methylenebis[6-(1,1-dimethylethyl)]-4-methyl-Phenol	4064930	4323896
21	27.78	Diisooctyl phthalate	11010589	38903167

- indicates undetected.

A total of 21 compounds were identified, as shown in Tab. 2. A total of 15 compounds were identified in teak wood before irradiation, whereas 13 compounds were identified in teak wood after 80 h of irradiation. Additionally, 8 compounds were detected in teak extractives from unexposed samples that were not detected in teak extractives from exposed samples, whereas 6 additional substances were generated in teak extractives after irradiation for 80 h.

As shown in Fig. 5 and Fig. 6, after the irradiation time reached 80 h, 10 compounds exhibited increased intensities and 11 compounds exhibited decreased intensities. Before

irradiation, the compounds with higher contents were squalene; 2-methyl-9,10-anthracenedione and phenol; respectively. However, the contents of phenol; dibutylphthalate and benzaldehyde; 3,4-dimethoxy-, methylmonoacetal were higher after exposure for 80 h. Wood discoloration has been shown to be mainly caused by the degradation of lignin and changes in pigments, tannins, resins, aromatic compounds and other compounds in the extractives (Živković et al. 2016). In this study, the intensities of squalene; 2-methyl-9,10-anthracenedione and phenol decreased substantially, and the intensities of benzaldehyde; 3,4-dimethoxy-, methylmonoacetal; diisooctyl phthalate and dibutyl phthalate obviously increased during the exposure. In a previous paper, Chang et al. (2010a) reported that the generation of quinonoids and carbonyl derivatives from lignin degradation is the main process behind the surface discoloration of wood. In contrast, the concentrations of squalene and 2-methyl-9,10-anthracenedione in this study were greatly reduced. This result is attributed to the speed at which the compounds volatilized on the surface far outweighing the speed at which they were generated. Additionally, both the main reduced compounds, squalene and 2-methyl-9,10-anthracenedione, are yellow-coloured substances. The reduction of coloured compounds makes the overall colour of teak veneer change from dark to light. Squalene and 2-methyl-9,10-anthracenedione contain double bonds or carbonyl groups related to wood discoloration, which indicates that the change in the shade of yellow during irradiation of the teak wood surface may have been directly related to the content of the coloured chemical components squalene and 2-methyl-9,10-anthracenedione.

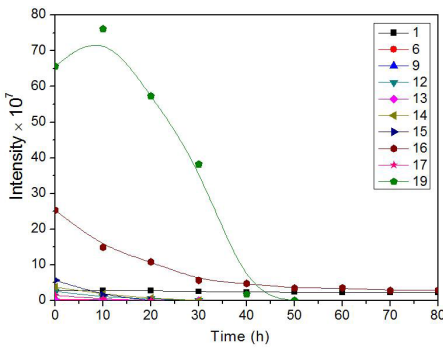


Fig. 5: A list of compounds whose concentrations decreased at different stages of irradiation.

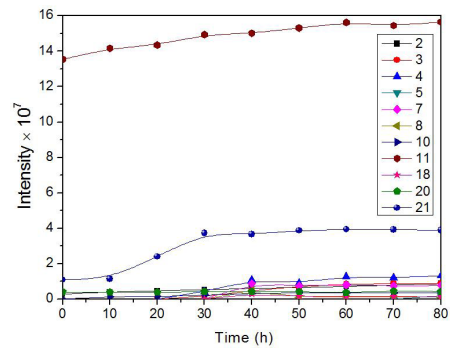


Fig. 6: A list of compounds whose concentration increased at different stages of irradiation.

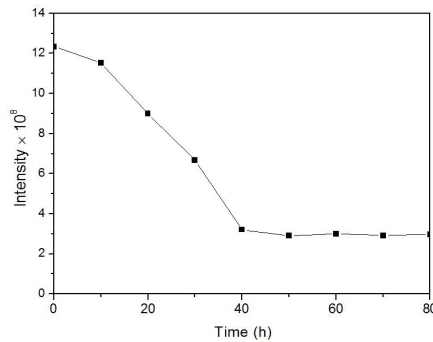


Fig. 7: The total ion current of extracts at different stages of exposure.

Fig. 7 shows the changes in the total content of extractives (TCE) during simulated exposure. With increasing time, the TCE rapidly decreased in the first 40 h of irradiation and then tended to stabilize. This behaviour demonstrates that some compounds in the extractives easily volatilized before the irradiation time of 40 h. This volatilization caused the teak veneer to undergo obvious colour changes, consistent with the results of the colour analysis. The decrease in ΔL^* during the first 20 h might have been due to the migration of extractives from the interior to the surface, which led to dark staining on the teak veneer surface because the teak extractives were brown polymers. With increasing irradiation time, the ΔL^* values were mainly influenced by the volatilization of extractives, which increased the lightness of the teak. After exposure for 40 h, the decrease in Δb^* was mainly due to the decreased contents of volatile extractives. Finally, these multifaceted factors led to the changes in ΔE .

Microscopic analysis of oil stain

Fig. 8 shows the changes of the oil stains on the teak veneer surface before irradiation and after 80 h of irradiation. Some striped oil stains were observed in the wood fibre and cells before irradiation; these stains were formed by resin fillers. After 80 h of irradiation, the oil stains decreased significantly. This observation indicates that the oil stains on the teak surface volatilized during irradiation, which led to an increase in lightness. We also investigated the distribution of oily liquids in the teak tissue (Fig. 9).

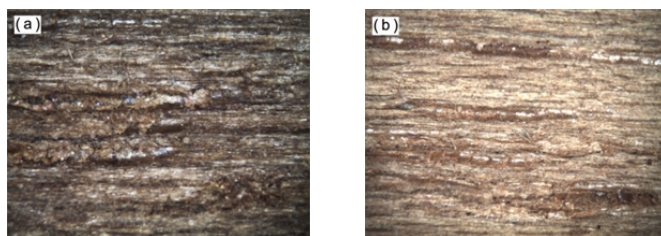


Fig. 8: Stereoscopic microscope photos of teak wood before irradiation (a) and after 80 h of irradiation (b).

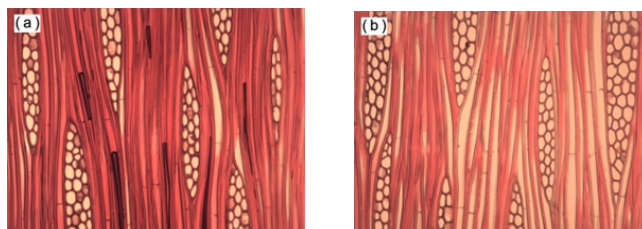


Fig. 9: Tangential sections of teak before extraction (a) and after extraction (b).

Teak slices of tangential sections were extracted and compared with those of untreated samples. The unextracted slices clearly contained black striped oil stains that did not exist after extraction. This result shows that extraction removed most of the oily organic components, producing the same effect as irradiation. In total, we concluded that the increase in ΔL^* was mainly caused by the volatilization of oil stains, which is consistent with the law of TCE, as determined by GC-MS analysis.

CONCLUSIONS

The colour of teak wood, during simulated sunlight exposure was changed towards a popular golden yellow from the initial brown. With analysis using a colorimeter we found that the ΔL^* values slightly decreased in the first 20 h and then sharply increased. The Δa^* values increased rapidly within 40 h and then increased slightly. In addition, the trends of Δb^* were similar to those of Δa^* , except for during the last 40 h, where Δb^* decreased continuously. Because of the synergetic effect of ΔL^* , Δa^* and Δb^* , the ΔE^* of teak wood increased sharply within the first 40 h of irradiation and then was tended to stabilize. Forty hours of simulated irradiation appears to be a key factor.

The FTIR and the quantitative results suggest that the trends of Δa^* and Δb^* during irradiation for 40 h changed completely, consistent with the decrease in lignin and the increase in C=O groups; thus, the generation of carbonyl derivatives caused the colour to shift towards red and yellow.

Additionally, the type and content of benzene-ethanol extractives was also closely related to the colour changes of teak wood by GC-MS analysis. With increasing irradiation time, the TCE rapidly decreased in the first 40 h of irradiation and then tended to stabilize. Combined with the microscopy analysis, the decrease in ΔL^* during the first 20 h might have been attributable to the migration of extractives from the interior to the surface. With increasing the irradiation time, the ΔL^* value was mainly being influenced by the volatilization of extractives, which increased the lightness of the teak. Additionally, after the teak was irradiated for 40 h, the decrease in Δb^* was mainly due to the decrease in the amount of volatile extractives. Thus, two main effects are responsible for the discoloration of teak wood. On the one hand, new chromophores related to lignin are generated. On the other hand, the original compounds in the extractives were volatilized.

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