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Colour stability of oil-heat treated black locust and poplar wood during short-term UV radiation

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ABSTRACT

Black locust (*Robinia pseudoacacia* L) samples with high extractive content and poplar (*Populus* × *euramericana cv.* Pannónia) samples with low extractive content were chosen for the test. The specimens were thermally treated at 160 and 200 °C in sunflower oil for 2, 4, and 6 h then irradiated by a strong UV emitter mercury lamp up to 36 h. The effects of thermal treatment and UV radiation were monitored by colour measurement. The results indicated that the extractives play an important role in the photodegradation of oil-heat treated wood. Thermal treatments reduced the lightness change effect of photodegradation. The oil-heat treated black locust samples showed similar photodegradation properties independently on the thermal pre-treatment time and temperature. The redness of oil-heat treated black locust samples hardly changed during UV radiation proving the photostability of the thermally modified extractives. The redness change of poplar samples caused by UV radiation was partly determined by the temperature of thermal treatment (darkening and lightening also happened). The yellow colour change of the investigated samples showed that the lignin of thermally modified wood undergoes similar photodegradation as that of the untreated natural wood.

1. Introduction

The thermal treatment of wood has a long history, and the different methods have been continuously modified and developed in European countries and worldwide as well. The main advantages of thermal treatment are: reduced hygroscopicity, improved dimensional stability, and better resistance to degradation due to insects and micro-organisms. The improvement of these properties gives the possibility to use thermally modified wood for outdoor applications.

During thermal treatments the hemicelluloses are the most affected compounds of wood. "The degradation starts by deacetylation, and the released acetic acid acts as depolymerisation catalyst, which further increases polysaccharide decomposition. Acid-catalysed degradation leads to the formation of formaldehyde, furfural and other aldehydes. Furfural and hydroxymethylfurfural are degradation products of pentoses and hexoses, respectively. At the same time hemicelluloses undergo dehydration reactions with a decrease of hydroxyl groups" [1]. Extractives also undergo degradation and new extractable compounds are created during thermal treatments. The oxidation of hydroxyl groups in flavonol molecules can be a reason of the formation for new colour substances during heat treatment [2].

The colour stability of thermally modified wood under outdoor conditions is also an important parameter for the costumers. Thermal treatments of wood create dark and attractive brown colour, which is highly determined by the applied temperature and treatment time [3–11]. The colour modification by thermal treatment is important for those species that have naturally unattractive light grey colour, such as poplar, or those that have highly inhomogeneous colour, such as black locust.

The colour change caused by the sunshine and the rain for natural wood is a well investigated phenomenon [12–19]. But the colour change caused by the photodegradation for thermally treated wood is not well researched, and the published results are sometimes contradictory. The real comparison is difficult because of the different conditions applied.

Ayadi et al. [20] tested the colour stability of heat-treated ash (*Fraxinus* sp.), beech (*Fagus sylvatica* L.), maritime pine (*Pinus pinaster*), and poplar (*Populus sp.*) wood samples. The heat treatment was done at 240 °C for 2 h, under nitrogen atmosphere. The heat-treated samples were exposed to UV light for 835 h. The

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total colour change was determined representing the changes. The results showed that the colour stability of the heat-treated wood was better than that of the untreated control samples.

Yildiz et al. [21] exposed outside heat-treated alder (*Alnus glutinosa* L.) wood in Turkey for 3 years. The treatment parameters were: 150 °C, 180 °C, and 200 °C, for periods of 2, 6, and 10 h. The results showed that heat treatments delayed and decreased the rate of colour change caused by the weathering but did not completely prevent it. The most advantageous treatment parameters were 200 °C for 10 h. Similar results were found during artificial weathering (UV + water spray) of Scots pine (*Pinus sylvestris* L.), spruce (*Picea orientalis* L.), iroko (*Chlorophora excelsa*) and ash (*Fraxinus excelsior* L.) as well [22].

The artificial photodegradation properties of heat-treated jack pine (*Pinus banksiana*) were studied using xenon lamp [9,10]. The total irradiation time was 1500 h. There was no difference in the a^* and b^* colour coordinate values between thermally treated and the untreated wood after 400 h light irradiation. There were slight differences at shorter irradiation time, but the authors did not measure the colour parameters before 72 h of treatment. The same authors stated in other paper: "The heat treatment increases the lignin and crystallised cellulose contents, which to some extent protects heat-treated birch against degradation due to weathering" [23].

Noupponen et al. [24] reported that heat treated wood was more resistant to natural weathering mainly because some of its lignin degradation products are less leachable than those of untreated wood.

Tolvaj et al. [25] monitored the short-therm photodegradation properties of black locust, poplar, spruce and larch samples pre-treated at 160 and 200 °C for 2, 4 and 6 h. The short term UV irradiation was carried out for up to 36 h. "Results showed that the extractive content of the wood played an important role in the colour change not only during thermal treatment but also during light irradiation. It was found that, compared to the thermally untreated samples; the thermal treatment at 200 °C reduced the red colour change caused by photodegradation. The yellow colour change of photodegradation was hardly affected by the applied thermal treatments, showing that thermal treatments were not able to reduce the degradation of lignin. The applied treatments slightly stabilized the wood against the degrading effect of light."

Similar results were found by Miklečić and his coworkers [26]. "In the first half of exposure to UV light, the surface of uncoated thermally modified (at 190 and 212 °C) ash, beech and hornbeam wood samples discolored slowly compared to uncoated unmodified wood samples. FTIR spectra of thermally modified ash, beech and hornbeam wood samples exposed to UV light showed similar chemical changes as unmodified wood samples exposed to UV light, but less pronounced." The duration of UV treatment was 32 days.

Srinivas and Pandey [27] investigated the photodegradation behaviour of thermally treated rubber wood (*Hevea brasiliensis*). The thermal treatment was carried out in vacuum atmosphere at 225 °C for 2, 4, and 6 h. The IR spectra showed significant lignin degradation in thermally modified wood within few hours of exposure. Results of colour changes and FTIR spectroscopy revealed that thermal modification of wood does not induce resistance against UV radiation.

The light wavelength dependence of photodegradation for thermally modified and unmodified aspen samples were also investigated [28,29]. The total irradiation time was 100 h. It was found, that wavelength longer than 600 nm did not generate degradation of wood. "Greater changes in IR spectra were observed for thermally modified wood compared to unmodified wood for all analysed wavebands and irradiation systems, which suggests that thermally modified wood was more chemically transformed by irradiation."

The literature review shows that the behaviour of thermally modified wood during UV light exposure is still not clearly understood. There are only a few papers dealing with the oil-heat treated wood [30,31].

The purpose of this study was to monitor the colour change of oil-heat treated black locust and poplar wood during short term UV radiation. Strong UV emitter mercury lamp was used. The colour change was compared to that of thermally treated samples in dry condition. The treatment parameters were identical in both cases for the correct comparison.

The main difference between the applied two types of pre-treatments is that oil excludes the oxidation of thermally degraded chemical components.

2. Materials and methods

2.1. Materials

Black locust (*Robinia pseudoacacia* L.) and Pannonia poplar (*Populus* × *euramericana cv.* Pannónia) wood were oil-heat treated (OHT) in sunflower oil. These species were chosen because black locust has high while poplar has low extractive content. The dimensions of the samples were $100 \times 20 \times 10\,mm^3$ (L;R;T directions).

2.2. Treatments

The thermal treatments were performed at two different temperatures: 160 °C and 200 °C, the applied durations were 2 h, 4 h and 6 h. In all 6 schedules 4 samples were treated with an initial moisture content of 13%. The samples were immersed directly into the hot oil bath without preheating. At the end of the oil-heat treatment the samples were taken out from the oil bath (without cooling schedule). Untreated samples with same dimensions served as control (4/species).

The thermally treated samples underwent photodegradation together with the untreated control samples. A strong UV light emitter, mercury vapour lamp provided the light irradiation. The UV radiation was 80% of the total emission (31% UV-A, 24% UV-B and 25% UV-C). The total electric power of the applied double mercury lamps was 800 W and the distance between the samples and the light source was 64 cm. An irradiation chamber set for 70 °C ensured ambient temperature conditions. The total irradiation time was 36 h. The irradiation was interrupted after 3, 7, 16, and 36 h for measuring the colour change, based on previous experiences.

2.3. Colour measurement

Colour measurements were carried out with a colorimeter (Konica-Minolta 2600d). The CIE L^*, a^*, b^* colour coordinates were calculated based on the D_{65} illuminant and 10° standard observer with a test-window diameter of 8 mm. The relatively large window was chosen to measure the average colour of earlywood and latewood regions combined. The radial surface of the sample was used for colour measurement. The colour of randomly chosen 10 points were measured on each sample. Measurements on thermally treated samples served as control values for comparison purposes for the photodegradation.

The colour stability of OHT samples was compared to the results of dry heat-treated samples published in Tolvaj et al. [25]. Some of these data are presented in this paper as well. For proper comparison, the related graphs have the same vertical scale magnitude.

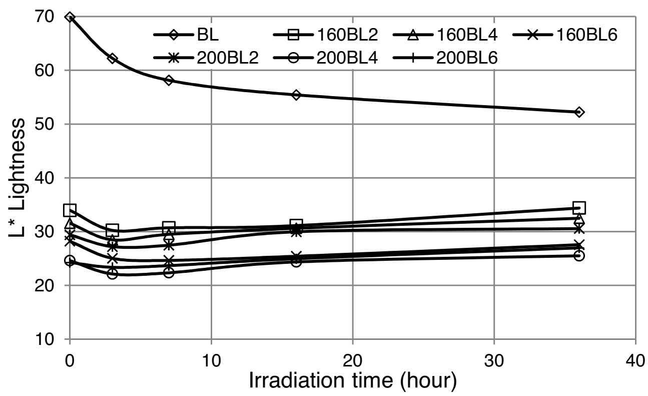

Fig. 1. Lightness change of OHT black locust caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

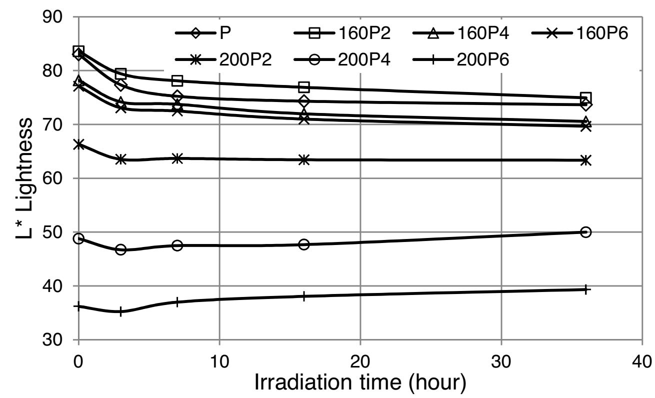

Fig. 3. Lightness change of OHT poplar caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

3. Results and discussion

The colour of a material is created by the conjugated double bond (chromophore) chemical systems. Such chromophores are in lignin and in certain sort of extractives for natural wood. The natural colour of wood is mostly determined by the extractives. Thermally modified wood has additional chromophores produced by the degradation of the hemicelluloses.

The investigated samples suffered intensive colour change during thermal treatment. The new lightness data generated by thermal treatment are visible in Figs. 1–10 as the left side starting points of the curves. The lightness intensity of black locust (Fig. 1) decreased from 70 down to 24, and varied between 24 and 34 depending on the heating temperature and time. The dry thermal treatment generated twice the diversity in lightness than OHT (Fig. 2), applying the same temperature and time conditions [25]. The reason is that oil-heat treatment prevents the oxidation. The dry thermal treatment reduced the lightness of black locust with up to 11 units because of oxidation of the heat generated chemical radicals. The variation was large between 42 and 11 units. It is 3 times larger interval than in case of oil-heat treatment. The lightness alteration behaviour of oxidation process was more time and temperature dependent for dry thermal treatment than for the oil-heat treatment. The oil-heat treatment of poplar samples at 160 °C hardly modified the lightness (Fig. 3). But the treatment at 200 °C generated large lightness decrease depending on the treatment time. The dry thermal treatment produced larger lightness decrease (the minimum was 31 units) than the oil-heat treatment (here the minimum was 36 units) in all examined cases for poplar. This difference can be interpreted by the lack of oxidation processes for OHT samples. The results indicate that the extractives play important role in darkening during thermal treatment. Especially the additional darkening effect of oxidation process is more pronounced for black locust (having high extractive content) than for poplar.

The UV radiation for natural (untreated) species produced intensive lightness decrease during the first 7 h of irradiation (Figs. 1 and 3). This change was followed by slow lightness decrease up to the end of the irradiation. All of the OHT black locust samples showed small lightness decrease during the first 3 h followed by very slow increase (Fig. 1). The changes were independent of the thermal treatment time and temperature. In contrast, the degradation behaviour of OHT poplar samples depended on the treatment temperature (Fig. 3). The poplar samples treated at 160 °C followed the change of natural poplar samples; the others (treated at 200 °C) followed the lightness alteration of OHT black locust samples. The lightness value of both OHT treated species decreased during the first 3 h of UV treatment independently of the heat treatment parameters. In contrast, samples treated thermally in dry air condition showed continuous but slow lightening (Fig. 2, the curves of poplar are not presented here). The values of these changes were considerably less than those of the untreated samples. These results suggest that thermal treatments (dry and OHT) reduced the lightness change effect of photodegradation.

The value of red colour coordinate for black locust increased more than double during oil-heat treatment (Fig. 4, See the left side starting points of the curves). It changed from 4.3 to 11.8 but the

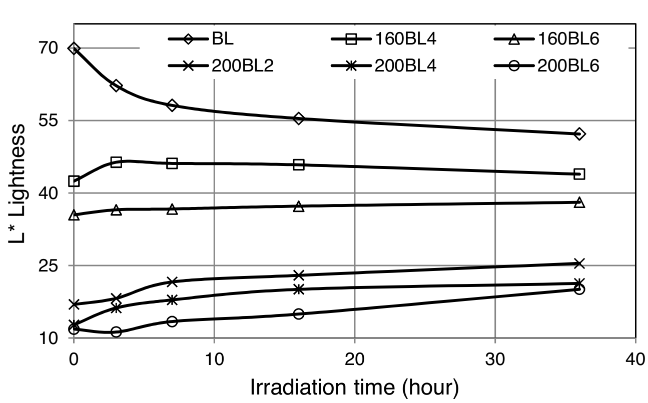

Fig. 2. Lightness change of dry thermal-treated black locust caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour (Tolvaj et al. [25]).

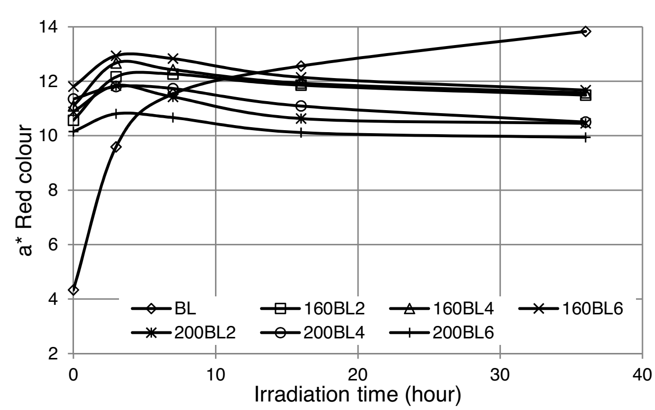

Fig. 4. Redness change of OHT black locust caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

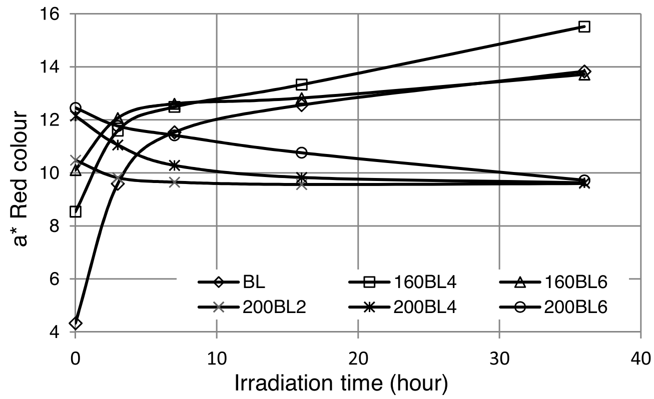

Fig. 5. Redness change of dry thermal-treated black locust caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

red colour coordinates of thermally treated black locust samples are in the narrow 10–12 interval. The heating time and temperature hardly affected the redness alteration. The dry thermal treatment generated more diverse redness change (Fig. 5, See the left side starting points of the curves) than the oil-heat treatment for black locust. The redness value of poplar samples also produced great increase during oil-heat treatment, but the changes were highly temperature and time dependent (Fig. 6, See the left side starting points of the curves). The redness coordinate increased from 3.6 to 13.7 for poplar. The dry thermal treatment in air generated similar redness coordinate increase as the oil-heat treatment (Fig. 7, See the left side starting points of the curves).

The red colour of untreated black locust samples rapidly increased during the first 7 h of UV exposure (Fig. 4). After 7 h of radiation, the trend line became straight, showing a slow increase. The red colour of untreated poplar shows almost equally continuous change during the whole irradiation period (Fig. 6). Red hue change of poplar was much less intensive than that of the black locust. The degradation products of extractives mainly determine the alteration of red colour coordinate during photodegradation [13,32,33]. This is the reason for the great difference between the photodegradation behaviour of black locust and poplar. The OHT black locust samples showed similar photodegradation properties. The changes were not affected by the pre-treatment time and temperature. The samples become a little more reddish during the first 3 h of UV exposure and the redness values slowly returned to the starting values during the further exposure (Fig. 4). It means that the OHT black locust samples were fairly

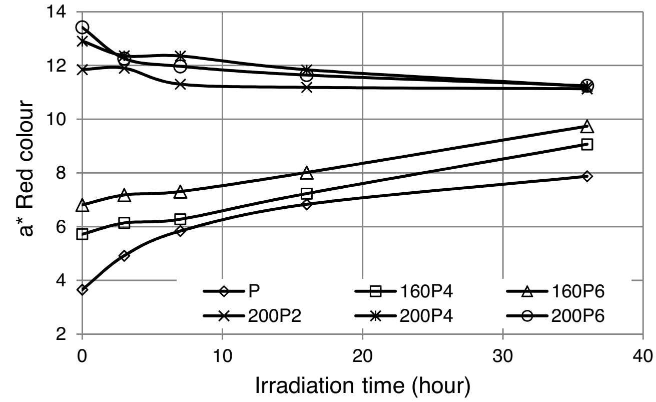

Fig. 7. Redness change of dry thermal-treated poplar caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

stable in terms of red hue change caused by photodegradation. The black locust samples pre-treated at 200 °C in air showed similar photodegradation behaviour than the OHT samples. Samples pre-treated at 160 °C in the air, however, followed the redness alteration of the natural samples (Fig. 5). It means that this type of thermal pre-treatment does not improve the photostability of black locust wood.

The starting points of poplar samples for UV exposure were highly different generated by the OHT pre-treatments (Fig. 5). The photodegradation behaviours of poplar samples pre-treated at 160 °C were similar as the behaviour of natural poplar samples. (Samples pre-treated for 2 h showed the same red colour shift as natural samples.) The redness coordinate of poplar samples pre-treated at 200 °C decreased a little during the UV exposure. The poplar samples pre-treated in air (Fig. 7) presented similar changes as OHT samples, only the starting points were slightly different. The results show that the heat treatment of poplar at 160 °C (neither oil- nor dry heat treatments) is not suitable to protect against photodegradation. Only the OHT black locust was an exception. It showed good resistance to photodegradation independently of the temperature of the pre-treatment. Thermal degradation products of black locust extractives seem to be less stabile during photodegradation if they undergo oxidation right after the thermal degradation. The pre-treatments at 200 °C generated protection against photodegradation in all examined cases. The samples treated at 200 °C for 2 h showed the most stable red hue values during photodegradation.

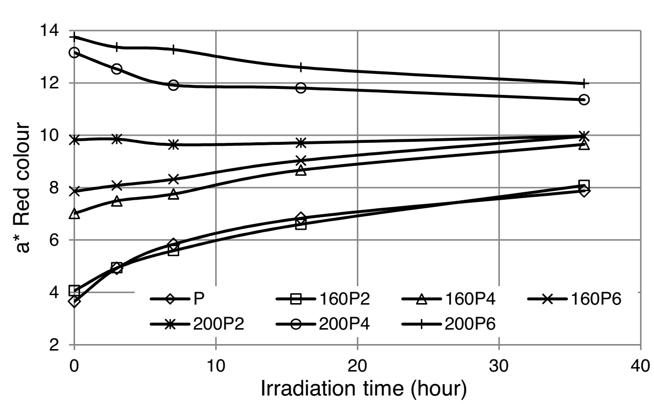

Fig. 6. Redness change of OHT poplar caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

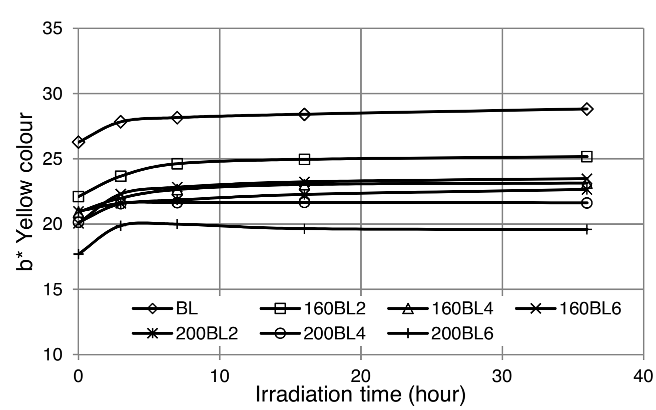

Fig. 8. Yellowness change of OHT black locust caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

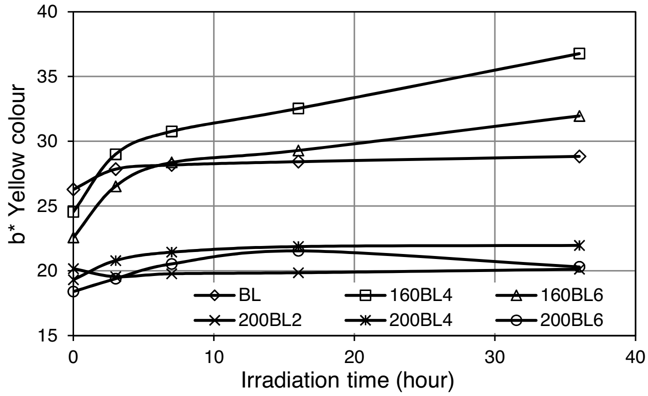

Fig. 9. Yellowness change of dry thermal-treated black locust caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

The yellow colour of black locust is naturally different compared to the other European species. This originally high yellow colour value decreased due to the oil heat treatment (Fig. 8, See the left side starting points of the curves) following the same tendency that the lightness change produced. At the same time, the low yellow colour coordinate of poplar increased following the tendency of red colour change (Fig. 10, See the left side starting points of the curves). The intensive yellow colour of black locust is generated by its high robinetin content. The degradation of robinetin produces the decrease of the yellow colour coordinate during thermal treatment.

The yellowing of wood during photodegradation is mainly generated by the degradation products of lignin [32]. The yellow colour of natural black locust changed only a little due to the UV exposure. A previous study demonstrated that the high extractive content of black locust partly protects its lignin content during light irradiation [34]. That is the reason why natural poplar samples revealed a more intensive yellow hue shift than black locust (Fig. 10). All of OHT black locust samples showed the same photodegradation behaviour as natural samples (Fig. 8). It means that in case of black locust the heat treatment in oil does not increase the protection of lignin against photodegradation. Moreover, black locust samples pre-treated in air at 160 °C showed even less protection against photodegradation than the natural wood. The natural samples produced 4 and 3 times less yellowness increase than those dry heat-treated at 160 °C for 4 and 6 h, respectively. It means that the extractives of black locust were not able to protect the lignin after these treatments. The degradation behaviour of OHT poplar at 160 °C was similar to the degradation of the natural poplar samples. Samples treated at 200 °C showed only moderate yellowing, however. Moreover, samples treated for 4 and 6 h produced small decrease in yellow coordinate during the first 3 h of UV exposure. Poplar samples pre-treated in dry condition showed the same yellowness change as OHT poplar samples (that is why this figure is not presented here). The yellow colour change in the samples treated at 200 °C for 2 h seemed to be the most stable, as it was found for red colour as well. The yellow colour changes of the investigated samples showed that the lignin of thermally modified wood undergoes a similar photodegradation as the untreated natural wood. It was found in a previous study as well [27].

The total colour change (ΔE^*) is a widely used parameter for monitoring the colour change. However, that the ΔE^* is not always reliable by itself. That is why the total colour change is not examined here. "In some cases ΔE^* values do not match to the visual observations. Human eyes are highly sensitive to the changes of hue determined by the alterations of a* and b*. The total colour change is mainly determined by the change of the dominant component. In the photodegradation of wood, the yellowing is the dominant change since the red colour shift is usually much smaller" [32]. The dominant parameter can be the lightness in other cases [35].

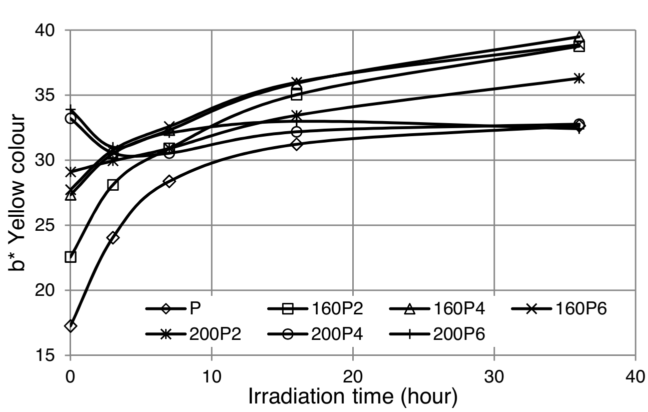

Fig. 10. Yellowness change of OHT poplar caused by light irradiation. Abbreviation: thermal treatment temperature/sample name/thermal treatment time in hour.

4. Conclusions

Thermally pre-treated black locust and poplar samples were irradiated by mercury lamp up to 36 h. 80% of total emission of the lamp was UV radiation. The colour measurement results indicate that the extractives play an important role in darkening during thermal treatment. Especially the additional darkening effect of oxidation process (following the thermal degradation) is more pronounced for black locust than for poplar. The thermal treatments reduced the lightness change effect of photodegradation. The oil-heat treated black locust samples showed similar photodegradation properties independently on the thermal pre-treatment time and temperature. The redness of OHT black locust samples hardly changed during UV radiation showing the photostability of the thermally modified extractives. The redness of poplar samples pre-treated at 200 °C was fairly stable to photodegradation, but the samples pre-treated at 160 °C showed same photodegradation properties as natural poplar samples. All of OHT black locust samples showed the same photodegradation behaviour as natural samples. The yellow colour changes of the investigated samples showed that thermal treatments were unable to protect the lignin during photodegradation.

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