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SURFACE CHARACTERISTICS OF SCOTS PINE WOOD HEATED AT HIGH TEMPERATURES AFTER WEATHERING

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ABSTRACT

This study was conducted to investigate some surface properties of wood specimens of heat treated Scots pine (*Pinus sylvestris* L.) after weathering such as surface hardness, surface roughness, gloss, and color changes. Heat treatment of Scots pine wood was carried out by hot air in an oven for 1, 2, and 3 hours at 210, 220, and 230°C.

The results showed that generally surface hardness losses of heat treated Scots pine wood were lower than that of un-heated Scots pine wood after weathering. Heat treated Scots pine wood gave smooth surface after weathering. Except for heat treatment at 230°C for 1 and 2 hours, heat treatment resulted in better glossiness compared to un-heated Scots pine wood after weathering. According to the test results, while heat treated Scots pine wood become lighter after weathering, un-heated wood become darken after weathering. In general, heat treated wood surface to become reddish and yellowish after weathering.

KEYWORDS: Scots pine, heat treatment, weathering, color, surface roughness, surface hardness, gloss.

INTRODUCTION

Wood and wood derived products have deficiencies such as hygroscopic behaviour based dimensional instability. There have been carried out many studies to increase this handicap of wood using different modification processes (Militz 2002, Hill 2006). Heat treatment is the oldest, most eco-friendly and the less expensive modification method which is widely in contemporary practices (Mburu et al. 2008, Ates et al. 2010). In a widely used heat treatment procedure, the material is exposed to temperatures between 120 and 250°C for 15 mins to 24 hours in where the exposure time is depended upon species, process, moisture content, sample size, and the planned target usage (Korkut and Guller 2008, Kocaefe et al. 2010). Since it doesn't include the use of chemicals and is not toxics it is is the most suitable method for wood. In last decade, utilization of heat treatment for wood modification has been increased (Bachle et al. 2010, Khalid et al. 2010).

Lignin and hemicelluloses are degraded after the treatment of wood at temperatures between 180 and 260°C. This aforementioned process reduced the woods hygroscopicity and alters the woods chemical structure. Therefore, thermally modified wood has more dimensionally stability than the unmodified wood (Shi et al. 2007). Because of its enhanced biological resistance against microorganisms and fungi, dimensional stability and discoloration, thermally processed wood can be used as expensive tropical species substitute.

Thermally processes wood is typically employed for the production of sauna and kitchen ceiling panels, parquets and wooden floor units, furniture units and garden fences and window frames (Boonstra 2008, Nemeth and Bak 2012, Zivkovic et al. 2008, Tuong and Li 2010). Heat treatment creates negative effects on woods hardness. Hardness of the sample woods can decrease with respect to increments in temperature and duration of process which can be assumed to be connected to degradation of cell wall structure with aforementioned processes. It can be seen that with alternative thermal processes loss of strength can be limited. In other respects with applied heat treatment, surface quality of the samples increased (Salca and Hiziroglu 2014). Ozgenc et al. (2017) reported that heat treated wood at high temperatures caused the extermination of hemicelluloses, which caused a reduce in free hydroxyl groups, which are responsible for wood wetting and the results of ATR-FTIR spectroscopy indicated that the destruction of hemicelluloses results in the formation of organic acids such as acetic acid and formic acid, which cause a decrease in the polymerization degree and the degradation of polysaccharides. They also explained that high temperature during the heat treatment affects the amorphous structures of cellulose, leading to an increase in the crystalline part of cellulose. Xianjun et al. (2011) was investigated the effect of heat treatment on some physical properties of Douglas fir (*Pseudotsuga menziesii*). They observed that heat treatment resulted in a darkened color, decreased moisture performance, and increased dimensional stability of wood. They also explained that equilibrium moisture content (EMC), water absorption (WA), and volume swelling (VS) for heated wood decreased up 42.63%, 34.93% and 67.47%, repectively. Increasing heat treatment duration and higher applied temperature might decrease WA, VS, and EM. Volume swelling of treated samples decreases more significantly than equilibrium moisture content and water absorption. If the samples are treated under more than 180°C, more distinct visual color changes are observed. It is proved that heat treatment gave rises to smooth surfaces and made color of the material stable but on the other hand under longer weather exposure, heat treatments protection effect become weaker (Yildiz et al. 2013). There is no sufficient research on the heat treatment of Scots pine wood at temperatures above 200°C.

This research was conducted to investigate the effects of heat treatment of Scots pine wood over 200°C and putting it to the weathering for 6 months exposure on surface properties.

MATERIAL AND METHODS

Preparation of test specimens

The specimens with dimensions of 10 x 100 x 150 mm (by radial by tangential by longitudinal) were collected from air-dried sapwood of Scots pine (*Pinus sylvestris* L.) lumber. Scots pine timber was kindly provided by Yucel Wood Products which is located at Mugla, South West of Turkey. Before the commencement of experiments all collected specimens are exposed to 20°C under 65% relative humidity for the duration of two weeks.

Heat treatment

A temperature-controlled laboratory oven is used at heat treatment at Mugla Sitki Kocman University. With air presence and under atmospheric pressure three heat treatment periods (1, 2, and 3 hours) and three different temperatures (210, 220, and 230°C) were applied to wood specimens.

Surface hardness test

Surface hardness of wood specimens was measured as the ASTM D4366-14 (2013) based Konig hardness. First the wood specimens were placed on a panel table, and then a pendulum was placed on the panel surface. Then, the pendulum was deflected through 6° and released and during this period a timekeeper was started. Amplitude time decreased 6° to 3° was recorded as Konig hardness.

Surface roughness test

Surface roughness of specimens was measured by the Mitutoyo Surftest SJ-301 (Mitutoyo Corporation, Tokyo, Japan) according to DIN 4768 (1990). The surface roughness measurement instrument includes a pick-up unit which includes a 5 µm tip radius containing diamond stylus and tip detector of conical taper angle of 90° and a main unit. The stylus scans the surface with the constant speed of 0.5 $mm \cdot s^{-1}$ over 8 mm sampling length (Zhong et al. 2013). Three roughness parameters which are typically used in previous studies for evaluation of wood and wood based materials surface characteristics: mean arithmetic deviation of profile (*Ra*), mean peak-to-valley height (*Rz*), and root mean square (*Rq*) (Hiziroglu 1996, Hiziroglu and Graham 1998).

Color test

CIELAB method is utilized in measuring the a^*, b^*, and L^* color test parameters. Lightness is represented by L^* axis and a^* and b^* represents the chromaticity coordinates. Red and green colors are represented by + a^* and $-a^*$ parameters, respectively. Yellow is represented by $+b^*$ parameter, and blue is represented with $-b^*$. L^* value can change between 100 (white) to zero (black) (Zhang 2003). Before and after the weathering a color meter is used for test specimens color measurement (X-Rite SP Series Spectrophotometer, X-rite, MI, USA). The measuring spot will be arranged as equal to not more than one third of the distance from this areas center and where the receptor field stops. The color difference, (ΔE^*) for specimens was determined with respect to ASTM D1536-58T (1964). The color changes were calculated using Eqs. 1 to 4,

$$\Delta a^* = a_f^* - a_i^* \quad (1)$$

$$\Delta b^* = b_f^* - b_i^* \quad (2)$$

$$\Delta L^* = L_f^* - L_i^* \quad (3)$$

$$(\Delta E^*) = [(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2]^{1/2} \quad (4)$$

where: the difference between the initial and final values are represented as Δa^*, Δb^*, and ΔL^*.

Gloss test

Glossiness of wood specimens was measured with a ASTM D523-08 (2008) based gloss meter (MicroTRI-Gloss, BYK Gardner, MD, USA). 60° incidence angle is chosen as the preferred geometry. Specular gloss value of 100 is selected as a basis of results. The results were based on a specular gloss value of 100 which perfectly reflects the conditions of wood sample under identical illumination and upon the highly polished, plane, black glass surface.

Weathering test

Each groups consisted of 10 individual wood specimens. In total, 10 wood specimen groups for every type were exposed to weathering conditions during from May 2016 to October 2016 in 2016. Wood panels were prepared for weathering exposure according to ASTM D 358-55 (1970).

Tab. 1: Details of the climate condition of Mugla city during weathering.

Months	May	June	July	August	September	October
Average temperature (°C)	16.7	24.8	28.0	27.6	21.4	17.3
Highest temperature (°C)	30.2	39.9	39.0	39.5	37.1	30.7
Lowest temperature (°C)	6.1	10.5	18.0	17.1	8.1	6.2
Sunbathing time per month (h)	7.0	10.2	10.4	8.6	6.8	4.0
No. of rainy days	11	5	2	2	4	1
Total rainfall per month ($kg \cdot m^{-2}$)	92.8	4.8	46.6	3.4	42.6	0.0
Moisture content (%)	62	45	41	46	50	55

A test site in the vicinity of Regional Meteorological Observation Station, Mugla, which is in Southern Aegean Region, was established for enabling practical assessments. The details of the climate condition of Mugla city in this period are given in Tab. 1 (Turkish State Meteorological Service Database 2016).

RESULTS AND DISCUSSION

Surface hardness changes

Surface hardness values of the heat treated Scots pine before and after weathering process were indicated in Tab. 2 and Fig. 1. The heat treatment at 210 and 220°C for 1, 2, and 3 hours and the heat treatment at 230°C for 1 hour were found to increase the surface hardness of Scots pine wood specimens compared to the un-heated (control) specimen.

Tab. 2: The surface hardness values measurements of Scots pine before and after the weathering.

Temp.(C°)	Duration (Hours)	Before weathering	After weathering	Differences
Control		23.2 (1.81)*	13.2 (2.49)	-43.10
210	1	23.8 (2.82)	12.4 (2.01)	-47.90
	2	24.8 (2.82)	14.8 (1.99)	-40.32
	3	24.1 (1.79)	17.0 (2.26)	-29.46
220	1	26.7 (2.71)	15.4 (2.63)	-42.32
	2	24.4 (2.67)	15.9 (2.23)	-34.84
	3	24.0 (2.87)	16.4 (2.72)	-31.67
230	1	23.5 (2.32)	16.0 (2.58)	-31.91
	2	23.0 (2.58)	16.4 (2.8)	-28.70
	3	20.1 (2.51)	15.7 (1.7)	-21.89

* : Standard deviations are shown in parenthesis.

Note: Ten replicates were made for each group.

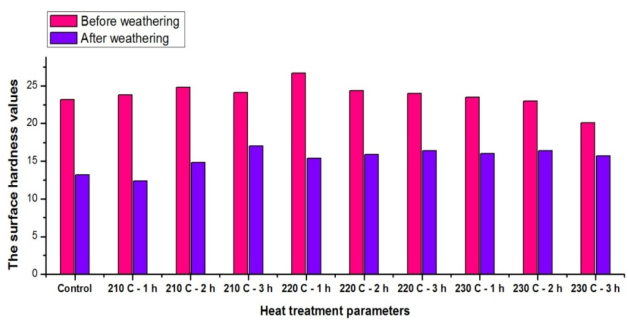

Fig. 1: The surface hardness of heat treated Scots pine before and after weathering.

Heat treatment of Scots pine at 220°C for 1 hour gave the highest surface hardness result before weathering. The lowest surface hardness of Scots pine was observed as a result of the heat treatment conducted at 230°C for 3 hours. The experimental results showed that weathering caused to decrease surface hardness of Scots pine in some extent. It can be explained that combined effect of moisture, UV light, and temperature could destroy the lignocellulosic network of the wood. Therefore, the degradation products become water-soluble and are leached out resulting in erosion of the wood surface (De Meijer 2001). In a similar studies, Baysal (2008) and Yalinkilic et al. (1999) studied surface hardness values of some weathered wood species. They found that that weathering softened wood surfaces and caused decrease in surface hardness values of test samples. Our results are in good agreement with data Baysal (2008) and Yalinkilic et al. (1999). After weathering while surface hardness of un-heated Scots pine decreased as 43.10%, it decreased 21.89 to 47.90% for heat treated Scots pine. Except for heat treatment at 210°C for 1 hour, decreasing of surface hardness of all heat treated Scots pine were lower than that of un-heated Scots pine after weathering. Therefore, heat treatments generally caused to lower decreases of surface hardness compared to un-heated Scots pine wood after weathering. Moreover, higher temperature and durations resulted in lower surface hardness decreases after weathering.

Surface roughness changes

Surface roughness parameters like *Ra, Rz,* and *Rq* values of the heat treated Scots pine wood are provided in Tab. 3. Un-heated wood (control) sample is measured in average as *Ra, Rz*, and *Rq* values, 2.50 µm, 16.51 µm, and 3.19 µm, respectively before weathering. According to test results, it can be said that the heat treatments caused lower surface roughness of Scots pine compared to un-heated Scots pine after weathering. For example, while the increase of *Ra, Rz*, and *Rq* were by 121.16, 66.33, and 117.89% respectively, for un-heated Scots pine after weathering, the increase range of *Ra* was from 54.37 to 107.36 %, *Rz* was from 32.96 to 51.06 %, and *Rq* was from 49.58 to 86.24 %, for the heat treated Scots pine after weathering. Turkoglu et al. (2015) investigated surface roughness of heat treated Oriental beech (*Fagus orientalis* L.) wood specimens after weathering. Heat treatment of Oriental beech wood samples was performed by using hot air in an oven for the treatment duration of 1, 4, and 8 hours at 140, 170, and 200°C. According to test results, average *Ra, Rz* and *Rq* values of un-heated specimens were increased 204.7%, 139.3%, and 131.3%, respectively.

Tab. 3: The roughness values of Scots pine before and after the weathering.

Temp.	Hours	Before weathering			After weathering			Differences (%)		
		Ra	***Rz***	***Rq***	***Ra***	***Rz***	***Rq***	***Ra***	***Rz***	***Rq***
Control		2.50 (0.8)*	16.51 (2.17)	3.19 (1.08)	5.52 (1.94)	27.46 (2.83)	6.95 (2.38)	121.16	66.33	117.89
210	1	2.47 (0.58)	16.00 (2.82)	3.20 (0.84)	5.13 (1.71)	23.70 (2.29)	5.97 (1.01)	107.36	48.17	86.24
	2	3.13 (1.24)	14.88 (1.64)	4.06 (1.67)	4.89 (1.19)	22.07 (2.72)	6.07 (1.47)	56.45	48.28	49.58
	3	2.87 (1.11)	15.15 (1.1)	3.56 (1.3)	4.43 (0.91)	22.89 (2.31)	5.57 (1.24)	54.37	51.06	56.72
220	1	2.44 (0.56)	16.16 (1.8)	3.16 (0.73)	4.72 (0.96)	22.65 (2.78)	5.77 (1.12)	93.49	40.22	82.67
	2	2.88 (0.54)	15.95 (2.19)	3.66 (0.69)	4.86 (1.56)	21.21 (2.92)	5.98 (1.87)	68.94	32.96	63.46
	3	3.12 (1.42)	15.55 (2.52)	3.88 (1.72)	5.13 (1.03)	22.61 (2.33)	6.31 (1.16)	64.05	45.40	62.61
230	1	2.79 (1.01)	13.74 (1.58)	3.56 (1.36)	4.38 (1.34)	19.91 (2.49)	5.45 (1.64)	57.25	44.95	53.07
	2	2.87 (1.17)	16.03 (2.48)	3.59 (1.37)	4.84 (1.27)	23.74 (2.59)	6.04 (1.59)	68.59	48.13	68.18
	3	3.26 (0.95)	16.40 (1.88)	4.09 (1.08)	5.47 (1.72)	21.89 (2.18)	6.82 (2.1)	67.62	33.48	66.66

* Standard deviations are shown in parenthesis.

Note: Ten replicates were made for each treatment group.

The lowest increases surface roughness of *Ra, Rz* and *Rq* values were observed by 21.4 %, 67.7 %, and 24.1 %, respectively, on the heat treated Oriental beech specimens at 200°C for 8 h. Yildiz et al. (2013) studied change in surface characteristic of heat treated four wood species (Ash, Iroko, Spruce, and Scots pine) under artificial weathering with the durations of 400 hours to 1600 hours with regard to their surface roughness. As a result of heat treatment, it is observed that enhanced wood specimens and smooth surfaces are relatively more resistant against factors of weathering. Baysal et al. (2014a) determined that surface roughness of un-heated Scots pine wood has a higher value with respect to heat treated Scots pine wood after accelerated weathering.

The experimental results are in good agreement with these researchers' findings. The industry expects smooth surface in solid wood for many applications. In addition, losses observed at the planer are decreased and qualities of the finishes are increased (Korkut et al. 2009). Moreover, the rough surface wood requires many times more sandpaper than the smooth surface wood, in the thickness of the material is reduced and losses caused by the sanding process are increased (Dundar et al. 2008).

Gloss changes

Glossiness which means the material's property of reflecting light like a mirror is extremely important for coated wood surfaces decorative and aesthetic appearance (Cakicier et al. 2011). Gloss values of the heat treated Scots pine wood before and after weathering were displayed in Tab. 4 and Fig. 2. Heat treatments caused gloss loss of Scots pine wood before weathering. While the gloss value of un-heated Scots pine was 3.30, it changed from 1.73 to 2.03 for the heat treated Scots pine.

This result is compatible with Aytin et al. (2015), Korkut et al. (2013), Aksoy et al. (2011 Baysal et al. (2014a, b), Turkoglu et al. 2015, Toker et al. 2016 who studied the effects of heat treatment on gloss of wood decreased with heat treatment. The weathering decreased 16.97 % gloss of the un-heated Oriental beech. Also, heat treatments at 230°C cause 1.07 to 33.16 % gloss loss of Oriental beech after weathering.

Tab. 4: The gloss changes values of Scots pine before and after the weathering.

Temp.	Hours	Before weathering (60°)	After weathering (60°)	Differences (%)
Control		3.30 (0.27)*	2.74 (0.34)	-16.97
210	1	2.03 (0.18)	3.17 (0.44)	56.16
	2	1.97 (0.26)	2.78 (0.27)	41.12
	3	1.73 (0.16)	2.39 (0.17)	38.15
220	1	1.83 (0.22)	2.42 (0.40)	32.24
	2	1.83 (0.25)	2.08 (0.33)	13.66
	3	1.85 (0.24)	1.90 (0.37)	2.70
230	1	1.87 (0.13)	1.85 (0.21)	-1.07
	2	1.97 (0.25)	1.50 (0.30)	-23.86
	3	1.93 (0.28)	1.29 (0.18)	-33.16

* : Standard deviations are shown in parenthesis.

Note: Ten replicates were made for each group.

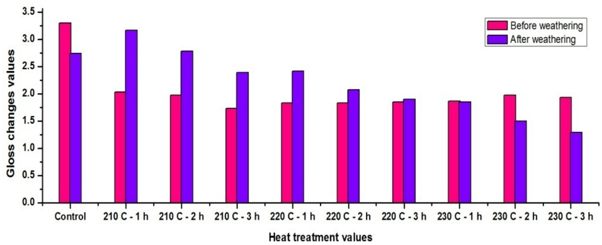

Fig. 2: The gloss changes of heat treated Scots pine before and after weathering.

Gloss degradation is occurred as a result of wood surfaces abrasion and accompanying erosion (Yalinkilic et al. 1999). However, all the other heat treatments increased gloss of Scots pine after weathering. At the publication of Turkoglu et al. (2015), it is stated that gloss losses of heat treated Oriental beech were lower than un-heated Oriental beech after weathering. Baysal et al. (2014a) investigated gloss changes of heat treated Scots pine wood after accelerated weathering. They found that heat treated Scots pine's gloss change was lower than un-heated Scots pine's after artificial weathering.

Except for heat treatments at 230°C, our results are compatible with these researchers' findings. Therefore, except for heat treatments at 230°C, all other heat treatments increased surface gloss of Scots pine after weathering.

Color changes

*L**, *a**, *b** values of un-heated and heat treated Scots pine before weathering and the alterations in *ΔL**, *Δa**, *Δb**, and *ΔE** of un-heated and heat treated Scots pine after weathering are given in Tab. 5.

Tab. 5: The color changes values of Scots pine before and after the weathering.

Heat treatment		Before weathering			After weathering			Color change			
		L_i^*	a_i^*	b_i^*	L_f^*	a_f^*	b_f^*	ΔL^*	Δa^*	Δb^*	ΔE^*
Control		66.34	11.83	20.28	51.89	3.27	18.08	-14.45	-8.56	-2.20	16.94
210	1	45.41	9.46	15.24	55.06	6.54	21.08	9.65	-2.92	5.84	11.65
	2	40.07	8.46	14.18	51.73	7.49	21.68	11.65	-0.97	7.50	13.89
	3	38.16	7.65	13.58	47.83	8.21	21.27	9.68	0.56	7.68	12.37
220	1	37.83	7.56	13.13	48.98	7.81	20.83	11.15	0.25	7.69	13.55
	2	32.51	6.13	9.40	44.10	8.26	18.83	11.59	2.13	9.43	15.09
	3	31.11	5.80	8.90	41.58	8.46	18.38	10.47	2.65	9.48	14.37
230	1	29.66	4.46	6.55	40.54	8.00	16.65	10.88	3.54	10.10	15.26
	2	29.06	4.16	5.95	36.39	7.63	14.93	7.33	3.47	8.98	12.10
	3	28.76	3.93	5.75	33.01	7.21	12.60	4.25	3.28	6.85	8.71

Note: Ten replicates were made for each group.

Before weathering, *L** of heat treated Scots pine wood was lower with respect to un-heated wood. The lower *L** values shows that the test examples become darker on the effect of heat treatment. Previous studies have shown that the treatment temperature and the duration make darker the test specimens (Militz 2002, Akgul and Korkut 2012, Esteves et al. 2008, Mitsui et al. 2003). The degradation of lignin and other noncellulosic polysaccharides may increase the darkening of heat treated Scots pine wood (Grelier et al 2000, Hon and Chang 1985, Petric et al. 2004). While positive ΔL^* indicates that heat treated Scots pine wood specimens become lighter after weathering, negative ΔL^* of un-heated Scots pine wood specimens become darker after weathering. ΔL^* of un-heated Scots pine was measured as -14.45, the values are changed for heat treated Scots pine after weathering from 4.25 to 11.65. Olărescu et al. (2014) studied ΔL^* of heat treated wood panels after three months of natural weathering. They found that while untreated samples become dark (negative ΔL^* measurements), the heat treated panels have a disposition to become light (positive ΔL^* measurements).

Our results are compatible with data Olărescu et al. (2014). While the positive values of Δa^* indicate a tendency of wood surface to become reddish, the negative values of Δa^* indicate a tendency of wood surface to become greenish. The results showed that except for the un-heated and heat treated at 210°C for 1 and 2 hours, all heat treatments gave positive Δa^* values after weathering. While positive Δb^* measurements show wood surface's disposition to appear yellowish, negative Δb^* measurements shows wood surfaces disposition to appear as bluish after weathering. Except for un-heated (control), all heat treated wood specimens showed positive Δb^* values after weathering. Our results showed that the heat treated Scots pine wood showed better color stability compared to the un-heated Scots pine. While, the total color changes ΔE^* of un-heated Scots pine wood was 16.94 it was changed from 8.71 to 15.26 for heat treated Scots pine. Total color changes (ΔE^*) at the end of the weathering were higher for the un-heated wood than that of the heat treated wood. Tomak et al. (2014) were investigated the color changes observed during two year long natural weathering in heat treated Iroko, Ash, Scots pine, and Spruce wood species. They found that after weathering heat treatment has an effect in improving the stability of color. Turkoglu et al. (2015) investigated color characteristics of heat treated Oriental beech. They found that ΔE^* of un-heated Oriental beech were higher than that of heat treated Oriental beech. Our results are compatible with these researchers' findings.

CONCLUSIONS

This study is attended on heat treated Scots pine wood specimens surface hardness, surface roughness, glossiness, and color after weathering. The results of study showed that weathering softened wood surface and increased surface roughness of Scots pine wood. However, heat treated wood showed better surface hardness and surface roughness than un-heated wood after weathering. Gloss loss were observed un-heated and heat treated Scots pine wood specimens at 230°C. While gloss value decreased for un-heated Scots pine specimens, gloss values increased heated Scots pine wood at 210 and 220°C after weathering. It can be said that heat treatment at 210 and 220°C prevented gloss loss of pine despite weathering. According to color change results, after weathering, while un-heated Scots pine wood become darken, heat treated Scots pine wood specimens become lighter. After weathering, wood surfaces generally indicate to become reddish and yellowish. Generally, higher temperature and duration of treatment improved surface characteristics of Scots pine after weathering.

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