TIME-COURSE CHANGES OF CHEMICAL AND PHYSICAL PROPERTIES IN SUGI (*CRYPTOMERIA JAPONICA* D. DON) LOGS DURING SMOKE HEATING

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ABSTRACT

Kiln-drying of sugi (Japanese cedar, *Cryptomeria japonica* D. Don) wood is a very difficult process. This is because the moisture content (MC) of the green log is very high and varies considerably between heartwood and sapwood. To investigate the time-course changes of chemical and physical properties during smoke heating, sugi green logs were smoke-heated with different treatment times at a temperature inside the log of 80°C using a modified food smoker. After smoke heating, the amounts of chemical components and some physical properties, such as MC, relative degree of crystallinity (RDC), equilibrium moisture content (EMC), and bending strength, were examined. The distribution of MC within the log became uniform after smoke heating for 60 h. Almost no differences in the amounts of chemical components were recognized between the control and smoke-heated woods. The relative degree of crystallinity increased with smoke heating for 40 h, corresponding to the decrease in EMC. The modulus of elasticity (MOE) in static bending increased with smoke heating, whereas the modulus of rupture and the absorbed energy in impact bending did not change significantly. The increase in MOE was considered to be due to the increase in RDC. The results obtained here indicated that no significant thermal degradation of wood occurred by smoke heating for 60 h at a temperature inside the log of 80°C. However, smoke heating affected the physical properties, such as RDC, EMC, and MOE.

Keywords: Sugi, smoke heating.

INTRODUCTION

Kiln-drying of sugi (Japanese cedar, *Cryptomeria japonica* D. Don) wood is a very difficult process. This is because the moisture content (MC) of the green log is very high and varies considerably between heartwood and sapwood. Smoke heating is a technique of heating under a wet condition. Past research has shown drying wood with smoke heating

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resulted in improved quality and more uniform decrease of MC within a log. In the present study, we investigated the changes in MOE and RDC for smoke-heating periods of various lengths at 80°C.

Recently, several researchers have attempted smoke heating of sugi logs to improve the wood quality (Okuyama et al. 1988, 1990; Nomura 1995; Andoh et al. 1996; Tejada et al. 1997; Ishiguri et al. 1998; Yoshizawa et al. 1999). The following advantages of smoke heating for the utilization and processing of wood have been reported: reduction of growth stress, improvement of sawing yield, and predrying effect by uniform decrease of MC within a log. However, some kinds of damage, such as surface checks, carbonization, and thermal deterioration of the wood's chemical components, also occurred frequently when logs were excessively smoke-heated. To avoid this, it has been pointed out that logs have to be smoke-heated within 40 h at a temperature inside the log of 80 to 100°C (Okuyama et al. 1988, 1990; Nomura 1995; Andoh et al. 1996; Ishiguri et al. 1998; Yoshizawa et al. 1999). This schedule has been mainly determined by a relationship between the degree of reduction of growth stress and the thermal deterioration of hemicelluloses (Okuyama et al. 1988, 1990).

In general, it is thought that if wood is dried at a low temperature below 100°C, no significant decreases in mechanical properties occur (Kadita et al. 1961). It is also well known that heating greatly affects the impact bending strength of wood, compared to the static bending strength (Kitahara and Chuganji 1951; Davis and Thompson 1964; Thompson 1969). Furthermore, it is well known that heating wood under a wet condition leads to the thermal deterioration of wood due to degradation of hemicellulose (Hillis 1984).

Smoke heating is also considered to be a technique of heating under a wet condition. Recently, Tejada et al. (1997) have reported that almost no reduction of the modulus of rupture (MOR) in static bending occurs even after prolonged smoke heating for 70 h at a temperature inside the log of 80 to 100°C. However, these authors did not examine the time-course changes of the relationship between the chemical components and the static and impact bending properties.

On the other hand, it is well known that heat treatment slightly increases the modulus of elasticity (MOE), which is related to the increase in degree of cellulose crystallinity (Hirai et al. 1972; Kubojima et al. 1998; Ishiguri et al. 1998). Ishiguri et al. (1998) reported that smoke heating of a sugi log for about 35 h at a temperature inside the log of 80°C increases the MOE in the bending of sapwood, corresponding with the increase in the relative degree of crystallinity (RDC). However, these time-course changes during smoke heating have not yet been clarified. It would be of interest to determine when the increase of MOE and RDC occurs during smoke heating and whether or not the changes depend on the heating temperature and time.

In the present study, we investigated the changes in MOE and RDC for smoke-heating periods of various lengths at 80°C. Sugi (*Cryptomeria japonica* D. Don) green logs were smoke-heated for various time periods using a modified food smoker that can easily control the treatment temperature and time. After smoke heating, the time-course changes of the chemical and physical properties were examined. From the results obtained, the effects of smoke heating on the chemical and physical properties of sugi logs were determined.

EXPERIMENTAL

Materials

A sugi (*Cryptomeria japonica* D. Don) tree, growing in the experimental forest of the Forest Research Center of Tochigi Prefecture, Utsunomiya, was harvested on 22 May 1996. A green log (220 mm in diameter and 3.2 m in length) was cut into 8 parts of 400 mm in length. Of the logs end-matched for testing, the first log was used for the control, while the others were used for smoke heating.

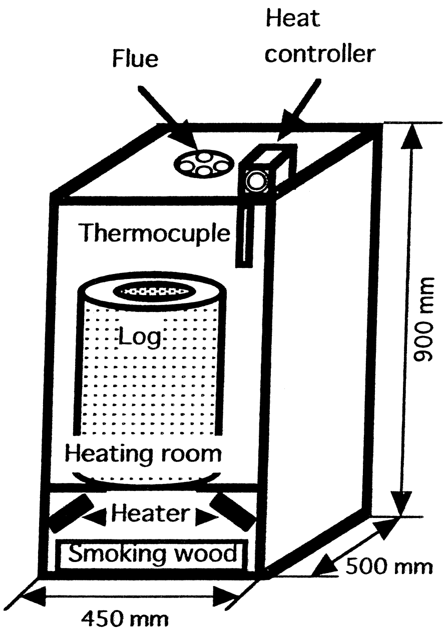

FIG. 1. Illustration of smoke-heating system (Ishiguri et al. 2000).

Smoke heating

Smoke heating of logs was carried out using a modified food smoker (Shinsei Sangyo, FS-50N) for 10–70 h according to procedures outlined in our previous study (Ishiguri et al. 2000) (Fig. 1). The changes of temperature inside the log during treatment were recorded with a thermocouple (Chino, Type T) inserted 100 mm deep from the log surface and a multi-channel digital recorder (Advnatest, TR2724). The temperature inside the log was controlled at 80 to 90°C during smoke heating. The internal temperatures were constant after about 10 h (Fig. 2). Treatment times reported in this paper include the 10 h of rising temperatures.

Moisture content (MC)

After smoke heating, disks of about 20 mm in thickness were collected from the middle logs for measuring the MC. Small blocks were taken from heartwood (HW), transition wood (TW: intermediate wood between heartwood and sapwood), and sapwood (SW). Moisture content was determined by the oven-drying method.

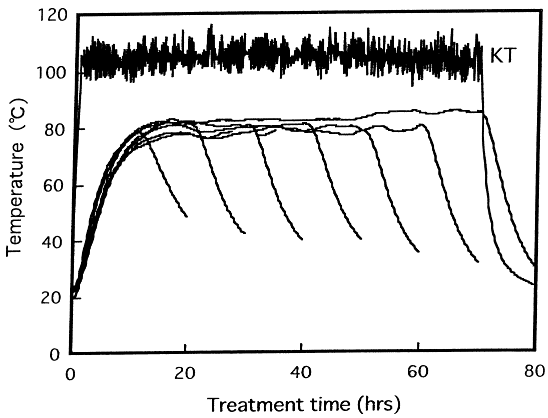

FIG. 2. Changes of temperature inside the logs during smoke heating. Note: The uppermost line (KT) in the figure indicates the kiln temperature at smoke heating for 70 h.

For measuring the equilibrium moisture content (EMC), wood meal (42–60 mesh) was taken from the sapwood of the control and smoke-heated woods. One gram wood meal in a weighing bottle was placed in a desiccator containing saturated sodium chloride at 25°C for 2 months. After reaching constant weight, EMC was determined by the oven-drying method.

Chemical components

Amounts of extractives (hot water, 1% sodium hydroxide, and ethanol-toluene) and main wood components (holocellulose and α-cellulose) were determined by the procedure known as TAPPI test methods (1991).

Static and impact bending strength

Ten specimens for each test, the static bending test (10 (R) × 10 (T) × 170 (L) mm) and the impact bending test (10 (R) × 10 (T) × 100 (L) mm), were taken from the sapwood of the control and smoke-heated woods. Specimens were conditioned to about 12% MC by placement in a desiccator containing 35% sulfuric acid solutions (65% R.H.) for two months.

The static bending test was conducted in an

TABLE 1. *Conditions for X-ray diffraction analysis.*

Characteristic of X-ray	CuKα
Slit div. angle	½ deg.
scatter. slit	½ deg.
receiv. slit	0.2 mm
Tube voltage	40 kV
Tube current	100 mA
Time const.	1 sec
Scanning speed	2 deg./min

Instron-type material test machine (Toyobaldwin, Tensilon UTM-III-500). The load was applied to the center of the longitudinal-radial surface of the specimen. The span and cross-head speed were 140 mm and 2 mm/min, respectively.

The impact bending test was carried out using a Charpy-type impact test machine (Toyo-kougyo-seisakusyo). Conditions for the impact bending test were as follows: span, 65 mm; pendulum weight, 16.6 N; distance from the center of the supporting axis to the center of gravity of the pendulum, 400 mm; and initial angle of the pendulum, 125°. The impact was applied to the center of the longitudinal-radial surface of the specimen.

Relative degree of crystallinity (RDC)

After the static bending test, wood meal (60–120 mesh) was taken from the bending test specimens and conditioned at 20°C and 65% RH for two weeks. An X-ray diffraction apparatus (JEOL JDX-12VA) was used for obtaining the X-ray diffraction intensity curve. Measuring conditions for the X-ray diffraction analysis are shown in Table 1. X-ray diffraction analysis was carried out at 20°C and 65% RH. The RDC was determined according to the following equation (Segal et al. 1959):

$$RDC(\%) = [(I_{002} - I_{am})/I_{002}] \times 100 \qquad (1)$$

where I_{002} is the maximum intensity of the $_{002}$ lattice diffraction and Iam is the intensity of diffraction in the same units at $2\theta = 18°$.

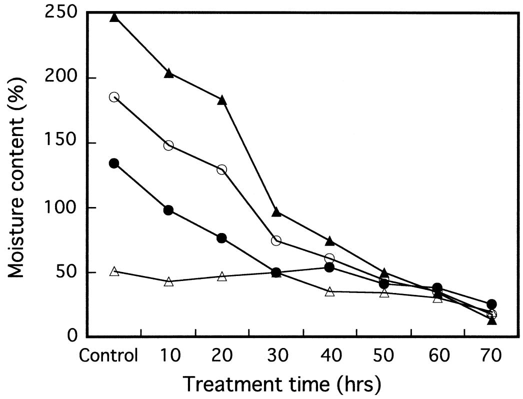

FIG. 3. Changes of moisture content by smoke heating. Symbols: Open circle, average; closed circle, heartwood; open triangle, transition wood; closed triangle, sapwood.

RESULTS AND DISCUSSION

MC

Figure 3 shows the changes of MC in logs by smoke heating with different treatment times. In the control wood, the MC of HW, TW, and SW showed 134.4, 51.5, and 247.5%, respectively. The MC decreased with increasing treatment time, finally showing 25.5, 19.7, and 13.4% for HW, TW, and SW, respectively, after smoke heating for 70 h.

In general, the initial MC gradient in the log is made more uniform as smoke heating dries the log (Okuyama et al. 1988, 1990; Nomura 1995; Andoh et al. 1996; Tejada et al. 1997). However, when the MC decreased below the fiber saturation point, many surface checks frequently occurred because of drying stress generated by the unequal distribution of MC within a log (Okuyama et al. 1988). In the present study, an almost uniform distribution of MC within a log was obtained by smoke heating for 60 h (MC: HW, 38.4%; TW, 30.8%; SW, 34.5%) without causing severe surface checks. In the log that was smoke-heated for 70 h, however, the MC of SW largely decreased below the fiber saturation point, causing many surface checks. These results suggest that the limit of smoke-heating time is 60 h at this temperature from the re-

TABLE 2. *Amounts of chemical components in smoke-heated woods.*

Treatment times (h)	Main components		Extractives		
	Holocellulose	A-cellulose	Hot water	1% NaOH	Ethanol-toluene
Control	70.4	43.0	1.5	9.1	1.1
10	68.4	41.1	2.7	10.7	1.5
20	73.5	45.5	1.2	8.5	1.1
30	72.7	45.4	2.2	9.2	1.3
40	69.9	45.0	4.4	12.1	1.7
50	71.5	45.6	3.5	10.8	1.5
60	70.6	44.0	2.2	10.5	1.5
70	69.3	43.4	1.3	10.9	1.7

lationship between MC and frequency of occurrence of surface checks.

Chemical components

Table 2 shows changes of chemical components by smoke heating. Almost no change of chemical components was found between the control and treated woods.

Okuyama et al. (1990) reported that smoke heating within 40 h at a temperature inside the log of 80–100°C effectively decreases growth stress in the trunk without any thermal degradation of chemical components, but thermal deterioration or degradation of hemicellulose rapidly occurs over 40 h of smoke heating. Similarly, Ishiguri et al. (1998) reported that almost no decrease in the amounts of chemical components is recognized in sugi logs smoke-heated for 35 h the same temperature. Furthermore, Tejada et al. (1997) reported that nonsignificant declines in the strength properties occurred even when some softwoods (karamatsu; *Larix kaemperi* Carriere, todomatsu; *Abies sachalinensis* Fr. Schm, and sugi; *Cryptomeria japonica* D. Don) were smoke-heated for a long time, 70 h, at a temperature inside the log of 80–100°C. Unfortunately, these authors did not research the changes of chemical components that accompanied the prolonged smoke-heating treatment.

In the present study, almost no change of chemical components was found between the control and smoke-heated woods. These results indicate that significant thermal degradation of chemical components does not always occur with smoke heating of more than 40 h if the temperature inside the log is kept low. It would be of interest to determine when higher temperatures and longer times in smoke heating of logs cause significant decreases of holocellulose. Further research is needed concerning the treatment time at which chemical components degrade, leading to a decrease in the mechanical properties, during smoke heating of logs.

TABLE 3. *Changes of RDC and EMC by smoke heating with different treatment times.*

Treatment times (h)	RDC (%)		EMC (%)	
	Mean ± SD	IR (%)	Mean ± SD	IR (%)
Control	40.7 ± 2.0	—	13.7 ± 0.4	—
10	40.5 ± 2.4	−0.5	13.7 ± 0.7	0.0
20	38.0 ± 2.3	−6.6	13.5 ± 0.5	−1.5
30	42.9 ± 2.4	+5.4	13.8 ± 0.5	+0.7
40	45.6 ± 1.9	+12.0*	13.6 ± 0.5	−0.7
50	44.2 ± 2.3	+8.6*	13.8 ± 0.5	+0.7
60	44.2 ± 1.1	+8.6*	13.1 ± 0.3	−4.4*
70	44.6 ± 1.1	+9.6*	12.8 ± 0.5	−6.6*

RDC, relative degree of cellulose crystallinity; EMC, equilibrium moisture content; IR, increased ratio; *, significance (5% level).

RDC and EMC

Table 3 shows changes of RDC by smoke heating with different times. The relative degree of crystallinity was significantly increased by smoke heating for over 30–40 h. It has been reported that, when wood was heated at a high temperature over 100°C, RDC increased at the initial stage of heating (Hirai et al. 1972; Inoue and Norimoto 1991; Kubojima et al. 1998). On the contrary, it has also been reported that RDC tends to decrease with increased heating temperature and duration of the heating time (Hirai et al. 1972; Dwiant et al. 1997).

The increase of RDC has been observed in smoke heating of logs (Okuyama 1996; Tejada et al. 1997; Ishiguri et al. 1998). Ishiguri et al. (1998) reported that when sugi logs were smoke-heated for about 35 h at a temperature inside the log of 80°C, RDC increased about 2% for heartwood and 8% for sapwood. In addition, the increase of RDC was also found in karamatsu, todomatsu, and sugi logs which were smoke-heated for 70 h at a temperature inside the log of 80–100°C (Tejada et al. 1997). Furthermore, the present study also revealed that RDC significantly increased by smoke heating for over 30–40 h. However, the mechanism involved in the crystallization of cellulose by heating, which probably leads to the increase of RDC, is not yet fully understood.

On the other hand, it is well known that EMC decreases as RDC increases (Kubojima et al. 1998). Okuyama (1996) reported that EMC decreases with the increase of RDC, this fact being evaluated as one of the effects of smoke heating. A similar tendency was confirmed also in karamatsu, todomatsu, and sugi, which were smoke-heated for about 70 h at a temperature inside the log of 80–100°C (Tejada et al. 1997). As shown in Table 3, EMC remarkably decreased in the present study as well when smoke heating exceeded 50 h. It is interesting to note that the increase of RDC is ahead of the decrease in EMC. The decrease of EMC is considered to be due to the increase of RDC occurring with the crystallization of the cellulose amorphous region by smoke heating (Hirai et al. 1972). In addition, the formation of intermolecular cross-linking or the aggregation of cellulose molecule accompanied with the decomposition of cell-wall components by heating might have occurred (Inoue and Norimoto 1991). It has also been reported, however, that RDC tends to decrease depending on the heating temperature and duration of heating time (Hirai et al. 1972; Dwiant et al. 1997). In general, the water molecule can penetrate into microfibrillar spaces of the non-crystalline region but not into the crystalline region (Fujita 1994). Therefore, the decrease of EMC is considered to be mainly due to the crystallization of cellulose in the non-crystalline region.

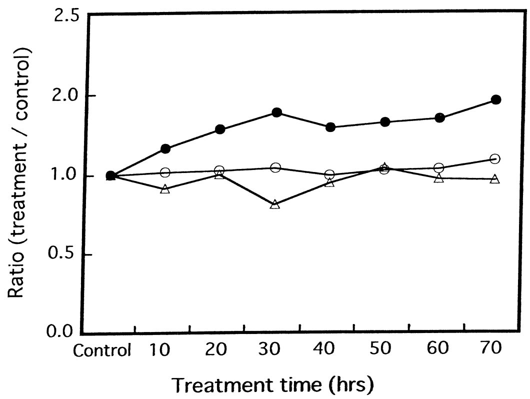

FIG. 4. Changes of bending properties by smoke heating with different treatment times. Symbols: Open circle, specific MOR; closed circle, specific MOE; open triangle, specific U.

Static bending properties

Figure 4 shows changes of the modulus of rupture (MOR) and the modulus of elasticity (MOE) in static bending of the sapwood samples by smoke heating with different treating times. The modulus of elasticity gradually increased with the increase of treating time, whereas MOR did not show great changes.

It has been considered that smoke heating for more than 40 h at a temperature inside the log of 80–100°C does not greatly reduce the mechanical properties (Okuyama et al. 1987; Nomura 1995; Ishiguri et al. 1998). Nomura (1995) reported that when a sugi log was smoke-heated at the same temperature inside the log, a slight reduction in both MOR and MOE occurred. On the other hand, Ishiguri et al. (1998) have demonstrated that both the MOR and MOE decrease in the heartwood of the smoke-heated sugi wood but increase in sapwood. However, Tejada et al. (1997) reported that smoke heating for 70 h at a temperature inside the logs of 80–100°C does not reduce the MOR or the MOE in the static bending of sugi wood, indicating that mechanical properties are not reduced by smoke heating over 40 h at a temperature inside the logs below 100°C. On the other hand, the results of the present study showed an apparent increase of MOE in static bending; interestingly, this increase corresponds to the increases of RDC.

In general, it has been recognized that MOE tends to increase at first and then decrease with the increase of heating time when the wood specimen is heated at the temperatures of 100–200°C (Hirai et al. 1972; Inoue and Norimoto 1991; Kubojima et al. 1998). Kadita et al. (1961) found that when Hinoki (*Chamaecyparis obtusa* Endle) wood was heated at 70, 100, 140, 170, 185, and 200°C for up to 100 h, the dynamic Young's modulus in the longitudinal direction increased in the initial stage of heating at a relatively low temperature (below 170°C) and then gradually decreased with the increase of heating time, whereas it decreased monotonically from the initiation of heating at the high temperatures of 185 and 200°C. From these results, they concluded that the effect of the heating temperature on the dynamic Young's modulus can be classified into two groups. In one, the effect is caused by lower temperatures from 70 to 170°C, and, in the other, by high temperatures above 185°C. Furthermore, Hirai et al. (1972) found that the dynamic Young's modulus slightly increases up to 100 h of heating at the relatively low temperature of 100°C. The increase of MOE in the initial stage is considered to be due to the increase of RDC by heating. This can be supported by our results showing a significant increase in the RDC by smoke heating.

On the other hand, Inoue and Norimoto (1991) found that heating treatment at 180, 200, and 220°C greatly decreases the MOR in static bending, whereas MOE slightly increases in the initial stage of heating at 180°C and then gradually decreases with the increase of heating time. They pointed out that the decrease ratio from heating is larger in MOR than in MOE. This decrease of MOR seems to be mainly due to the decrease in degree of polymerization (DP) of cellulose by heating (Inoue and Norimoto 1991). Differing from the results that have been obtained so far, however, MOR showed almost no changes by smoke heating at the relatively low temperature used in the present study (Fig. 4). This fact suggests that the decrease in the DP of cellulose does not occur by low-temperature heating, at least up to 70 h.

The results obtained with the static bending test indicate that thermal degradation of wood components did not occur by smoke heating for 70 h at a temperature inside the log of 80°C. However, a full explanation of the increase in the MOE by smoke heating has not been obtained yet. Further research is needed to clarify the relationship between MOR, MOE, and the DP and RDC of cellulose in the smoke-heated wood.

Absorbed energy in impact bending (U)

As shown in Fig. 4, U showed slight variation by smoke heating. However, the *t*-test (5% level) showed almost no significant difference in the absorbed energy in impact bending between the control and smoke-heated woods.

It has been reported that heating greatly affects the impact strength of wood compared to the static strength (Kitahara and Chuganji 1951; Davis and Thompson 1964; Thompson 1969). For example, the U of hinoki wood decreased to about one half by heating at 150°C for 20 h (Kitahara and Chuganji 1951). Furthermore, it has been found that heating in the wet condition largely decreases the impact strength due to the degradation of carbohydrate, such as hemicelluloses, compared to heating in the dry condition (Davis and Thompson 1964; Thompson 1969). Okauchi et al. (1997) reported that when wood was heated at 130°C under the wet condition, U decreased with the increase in treatment time. Thompson (1969) also found that chemical analysis of steamed specimens shows a highly significant relationship between the toughness and changes in the carbohydrate fraction of wood. Hillis (1984) also reported that heating wet wood leads to degradation, loss of strength, and other changes, probably due to the increase of acetic acid derived from the degradation of hemicelluloses during heating. These findings indicate that the loss of mechanical properties by heating under the wet condition is due to the thermal deterioration or degradation of hemicelluloses.

Smoke heating is the heating of wood under a wet condition (Okuyama 1996). In general, smoke heating within 40 h at a low temperature inside the log of 80–100°C does not cause thermal degradation of chemical components (Okuyama et al. 1990; Ishiguri et al. 1998). However, Okuyama et al. (1990) recognized that the thermal deterioration of hemicelluloses occurred even at a low temperature when smoke heating exceeded 40 h. In the present study, as shown in Table 2, there were almost no differences in the amounts of chemical components between the control and smoke-heated wood despite the prolonged smoke heating. This suggests that the thermal degradation of chemical components did not occur in the treated woods. Therefore, it is concluded that low-temperature heating under wet conditions does not affect the absorbed energy in impact bending.

CONCLUSIONS

(1) Smoke heating for 60 h resulted in a uniform distribution of MC within the log.
(2) Almost no differences in the amounts of holocellulose, α-cellulose, and extractives were found between the control and smoke-heated woods.
(3) The EMC decreased in smoke heating for 30–40 h, corresponding to the increase of RDC.
(4) The MOE increased as the smoke heating time increased, whereas MOR did not show great changes. The absorbed energy in impact bending did not change significantly.

These results indicate that smoke heating at lower temperature and for longer time periods of up to at least 70 h does not cause thermal degradation of wood components. However, smoke heating does affect some physical properties, such as RDC, EMC, and MOE.

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