ORIGINAL ARTICLE

Effect of temperature on thermal softening of black sweet-bamboo culms (*Dendrocalamus asper* Backer) in linseed oil

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Abstract

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The aim of this research is to study the effect of temperature on thermal softening behavior of black sweet-bamboo culms in linseed oil. Pressing test rig with a length scale and a pointer indicating specimen height was constructed. This apparatus was used to apply a compressive force to bamboo specimen immersed in hot linseed oil in a boiler. Half circular cross-section specimens with thickness of 3 mm and length of 150 mm were dipped into water at room temperature to attain water saturated condition prior to immersing into linseed oil at various temperatures under the load of 20 N. Specimen height, used to calculate the degree of flatness, was measured as a function of time. The values of the final degree of flatness and the rate of degree of flatness were used for the analysis of thermal softening behavior of bamboo in linseed oil. It was found that thermal softening behavior of bamboo culms in linseed oil was divided into two temperature regimes with the glass transition temperature at 115ºC. At low temperature regime, deformation occurred slowly and showed only a single stage of deformation, corresponding to deformation in the glassy state. At high temperature regime, specimens deformed slowly in the first stage followed by a rapid deformation in the

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second stage, corresponding to deformations in the glassy and rubbery states, respectively. Effect of temperature on the rate of softening was well described by means of the Arrhenius equation with the activation energy ranging from 18 kJ/mole to 32 kJ/mole.

Key words : bamboo, thermal softening, linseed oil, glass transition temperature

บทคัดย่อ

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ผลของอุณหภูมิต่อการอ่อนตัวทางความร้อนของลำไม้ไผ่ตงดำ (*Dendrocalamus asper* Backer) ในน้ำมันลินสีด

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งานวิจัยนี้มีจุดมุ่งหมายเพื่อศึกษาผลของอุณหภูมิต่อการอ่อนตัวทางความร้อนของลำไม้ไผ่ตงดำในน้ำมันลินสีด ชุดกดชิ้นตัวอย่างซึ่งมีเข็มบอกระดับความสูงของตัวอย่างถูกสร้างขึ้นเพื่อใช้กดตัวอย่างซึ่งถูกจุ่มไว้ในน้ำมันลินสีดในหม้อต้ม ชิ้นตัวอย่างไม้ไผ่ซึ่งมีหน้าตัดเป็นครึ่งวงกลม หนา 3 มม. และยาว 150 มม. ถูกแช่ในน้ำที่อุณหภูมิห้องจนถึงจุดอิ่มตัว ก่อนที่จะถูกจุ่มลงในน้ำมันลินสีดที่อุณหภูมิต่าง ๆ ภายใต้แรงกด 20 นิวตัน ความสูงของไม้ไผ่ที่ลดลงจะถูกบันทึกที่ระยะเวลาต่าง ๆ เพื่อนำไปคำนวณหาค่าองศาความแบน ค่าองศาความแบนสุดท้ายและอัตราขององศาความแบนจะใช้ในการวิเคราะห์พฤติกรรมการอ่อนตัวทางความร้อนของไม้ไผ่ในน้ำมันลินสีด ผลการทดลองพบว่าพฤติกรรมการอ่อนตัวทางความร้อนของไม้ไผ่ในน้ำมันลินสีดแบ่งเป็นสองช่วงอุณหภูมิ โดยมีอุณหภูมิ glass transition ที่ 115°C ในช่วงอุณหภูมิต่ำการเปลี่ยนรูปของตัวอย่างเกิดขึ้นช้าและเกิดขึ้นเพียงกระบวนการเดียวซึ่งเป็นการเปลี่ยนรูปในสถานะ glass ส่วนในช่วงอุณหภูมิสูงการเปลี่ยนรูปจะเกิดขึ้นช้าในช่วงแรกซึ่งเป็นการเปลี่ยนรูปในสถานะ glass ตามด้วยการเปลี่ยนรูปที่เกิดขึ้นอย่างรวดเร็วซึ่งเป็นการเปลี่ยนรูปในสถานะ rubber ผลของอุณหภูมิต่ออัตราองศาความแบนสามารถอธิบายโดยอาศัยสมการ Arrhenius ซึ่งพบว่าพลังงานกระตุ้นสำหรับกระบวนการอ่อนตัวทางความร้อนของไม้ไผ่ในน้ำมันลินสีดมีค่าในช่วง 18 kJ/mole ถึง 32 kJ/mole

[1]สาขาวิชาฟิสิกส์ สำนักวิชาวิทยาศาสตร์และหน่วยวิจัยวิทยาศาสตร์และวิศวกรรมศาสตร์ไม้ [2]สำนักวิชาวิศวกรรมและทรัพยากรและหน่วยวิจัยวิทยาศาสตร์และวิศวกรรมศาสตร์ไม้ มหาวิทยาลัยวลัยลักษณ์ อำเภอท่าศาลา จังหวัดนครศรีธรรมราช 80160

While a demand for good quality timber has been increasing, government regulations and environmental restrictions to preserve the world's existing forests have mounted pressures on logging in many developing countries, especially in Thailand, where commercial logging in the forests has been prohibited since 1989 (Royal Forest Department of Thailand, 2000). These factors are major driving forces in searching for alternative wood materials to supplement domestic uses of timbers in the near future. Bamboo has potential to be a timber substitute material. Bamboo grows very fast to its mature size in less than one year with an average maturity of 3-8 years (Dransfield and Widjaja, 1995, Suzuki and Itoh, 2001). It is also easy to plant and cultivate at low cost. The bamboo culm which is cylindrical and hollow is divided at intervals by nodes. The culm is comprised of exodermis (bark which is heavily overlaid with a waxy covering called cutin to prevent loss of water from the culms), parenchyma cells, vascular bundles and endodermis (inner surface layer). The vascular bundle is made up of vessels (transporting water), sieve tubes (transporting nutrition) and thick-walled fibers (Grosser and Liese, 1971). The strength of bamboo, especially in the longitudinal direction, mainly arises from the thick-walled fibers which have comparable mechanical strength with steel (Amada *et al.*, 1996). The value of fracture toughness of bamboo is also higher than that of Al-alloy (Amada and Untao, 2001). Biological structure of bamboo on microscopic scale (e.g. microstructure of fibers) and macroscopic scale (e.g. distribution of vascular bundles from the inner surface to the outer surface of the culm) was considered to be a good and smart model of the advanced composite materials (Amada *et al.*, 1996). Recently, microstructure of bamboo fibers was used as guidance from nature to improve performance of engineering composite materials in the field of biomimetics (Li *et al.*, 1995a).

For the past few years, research on efficient utilization of bamboo of low-cost resources into high value-added products has increased tremendously. These projects include development of structural materials (e.g. structural boards, trusses and concrete reinforcement) and development of reinforcement of composite materials (e.g. plybamboo, bamboo-epoxy composites, bamboo-fiber reinforced polymer, and reformed bamboo/aluminium composites) (Ghavami, 1995; Li *et al.*, 1994; Li *et al.*, 1995b; Ismail *et al.*, 2002 and Chen *et al.*, 1998). However, the manufacturing techniques of these products can be very complicated and time consuming because of the tubular shape with transverse diaphragms of bamboo culms. To maximize the utilization of bamboo and to overcome constraint due to the shape, the process of softening the bamboo culms is quite essential. Various techniques of softening bamboo culms have been reported by many researchers e.g. in China Guisheng (1987) dipped pieces of bamboo strips (*Phyllostachys pubesens*) into boiling water for several hours followed by paraffin at the temperature of 130ºC for ten minutes. Li and co-workers (1994) heated bamboo strips (*Bambusa pervaribilis*) in a container to adjust the moisture content to a certain value and then compressed them into plates. For bamboo species grown in Thailand, Kyokong and coworkers (2000) have successfully softened black sweet-bamboo culms (*Dendrocalamus asper* Backer) by immersion of bamboo strips in boiling water for 18 hours followed by dipping into boiling linseed oil for 45 seconds. Linseed oil is natural oil that has been long used as wood surface coating. Recently, impregnation of linseed oil into wood to prolong the service life has been achieved (Olsson, 1999). The main objective of this research was to study the effect of temperature of linseed oil on thermal softening of black sweet-bamboo culms. Particular attention was given to determining the glass transition temperature, Tg of bamboo in linseed oil. This information can be used in the design of bamboo softening process in the manufacture of bamboo structural composite products.

Background

Wood is a polymeric material consisting of crystalline and amorphous constituents (Haygreen and Bowyer, 1989). Amorphous wood constituents, such as hemicellulose and lignin, exhibit a visco-elastic behaviour influenced by temperature and moisture content (Glasser *et al.*, 1998). At low temperature, amorphous wood constituents are in the "glassy state" exhibiting high strength and modulus. As temperature increases, values of strength and modulus drop very sharply within small temperature range called the glass transition temperature, Tg. Amorphous wood constituents attain another softer state called a "rubbery state". Glass transition temperature in wood, ranging from 60ºC to 235ºC, depends on moisture content, wood chemical composition and method of testing (Lenth, 1999). Lower moisture content trends to increase the glass transition temperature. Upon further increasing temperature, amorphous wood constituents become viscous fluid. However, this state is not reached in wood because of total thermal degradation of amorphous wood constituents at high temperature (Wolcott *et al.*, 1990).

Materials and Methods

Half circular cross-section bamboo specimens with thickness of 3 mm and length of 150 mm were prepared from approximately 3-4 years old black sweet-bamboo culms (*Dendrocalamus* *asper* Backer), taken from Thasala district, Nakhon Si Thammarat province (Figure 1a). After removing the outer and the inner surfaces, specimens were touch sanded with SiC papers to ensure that the surface finish was in good condition (Figure 1b). Twenty-four specimens were prepared and allocated randomly among the six temperature treatments of linseed oil (80ºC, 100ºC, 115ºC, 130ºC, 150ºC and 180ºC). Prior to testing, all specimens were dipped into water at room temperature of 29ºC to adjust the moisture content to water saturated condition. Average moisture content of specimens at water saturated condition was 46±8%. To study thermal softening of bamboo culm in linseed oil, a novel and simple technique was developed. The apparatus shown in Figure 2 was constructed and arranged. A pressing test rig with a length scale and a pointer indicating

(a)

(b)

Figure 1. Photographs showing (a) black sweet bamboo culms and (b) half circular cross-section bamboo specimens used in this study.

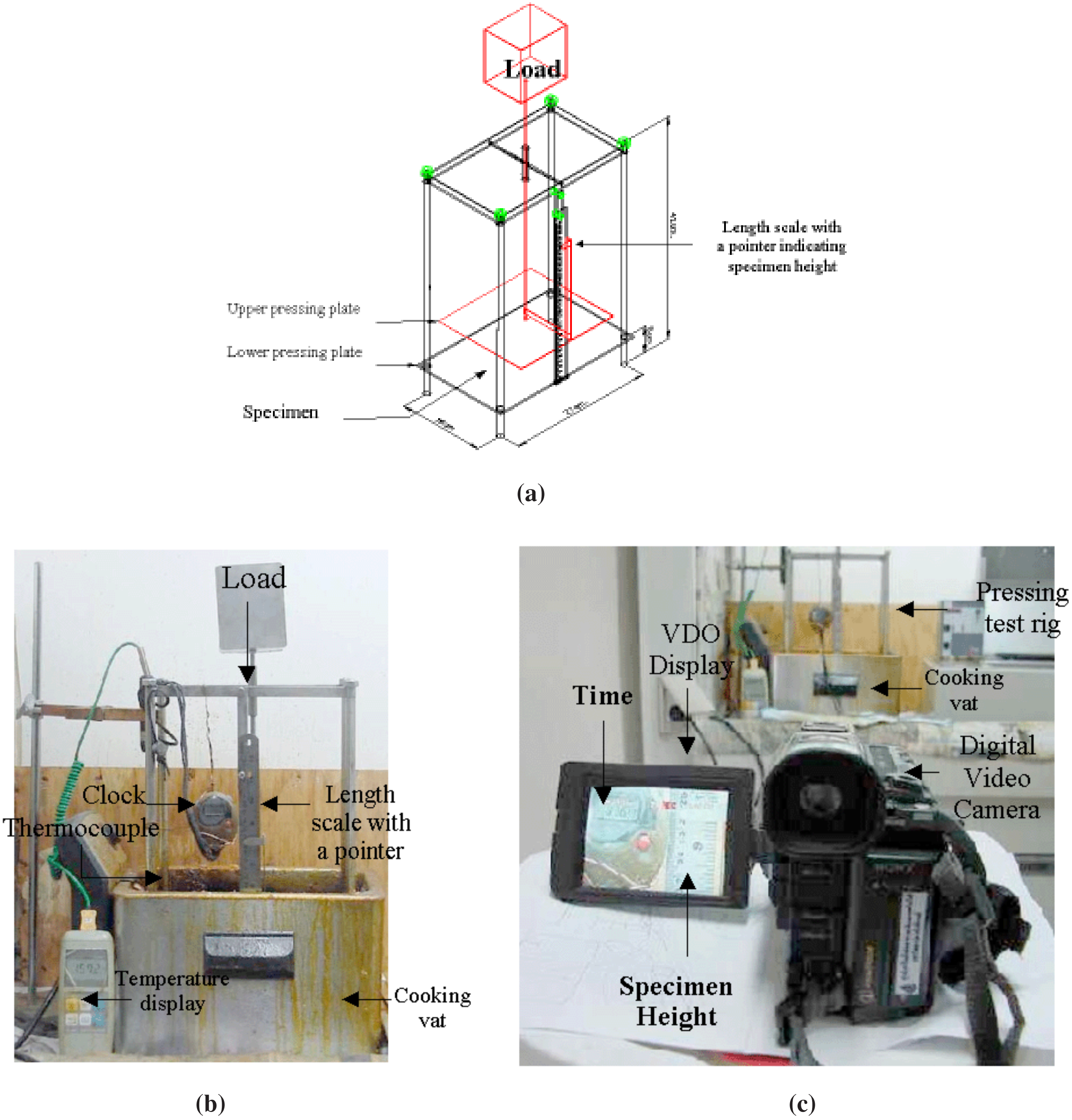


Figure 2. (a) Schematic diagram showing pressing test rig and photographs showing (b) pressing test rig immersed in linseed oil, heated to specified temperature in a boiler and (c) digital video camera used to record specimen height and time during testing.

specimen height was used to apply a compressive force to bamboo specimen (Figure 2a). The pressing test rig was immersed into hot linseed oil, heated to the required temperatures in a cooking vat (Figure 2b). A thermocouple was used to measure the temperature of the linseed oil. The cooking vat was capable of heating linseed oil up to 190ºC with an accuracy of ±2ºC (increased up to ±5ºC during testing). A force of 20 N was applied to the bamboo specimen immediately after immersing the specimen into linseed oil (Figure 2b). This force generated a compressive stress at the outer surface and a tensile stress at the inner surface. These stresses caused the specimen to deform into a flatter shape (Figure 3). From our preliminary work, it was found that the applied force of 20 N was sufficient to deform a half circular bamboo specimen with the thickness of 3 mm into a plate at linseed oil temperature of 180ºC without any cracks developing within the specimen. A digital video camera was used to record specimen height during testing (Figure 2c). Specimen height at any given time, d, was then later extracted from the video files. The degree of flatness, Φ, was calculated from specimen height using the equation $\Phi = (r_0-d)/(r_0-h)$ where r_0 is the initial height and h is the specimen thickness (Figure 3). It should be noted that at the initial condition where $d = r_0$, $\Phi = 0$, and after testing if the specimen is completely deformed into a plate where $d = h$ then $\Phi = 1$. The final value of degree of flatness, Φ_F, and the rate of degree of flatness, K, were used for the analysis of thermal softening behavior of bamboo in linseed oil.

Results and Discussion

Figure 4 shows typical bamboo specimens after testing at three different temperatures of linseed oil. Specimen deformed very little with

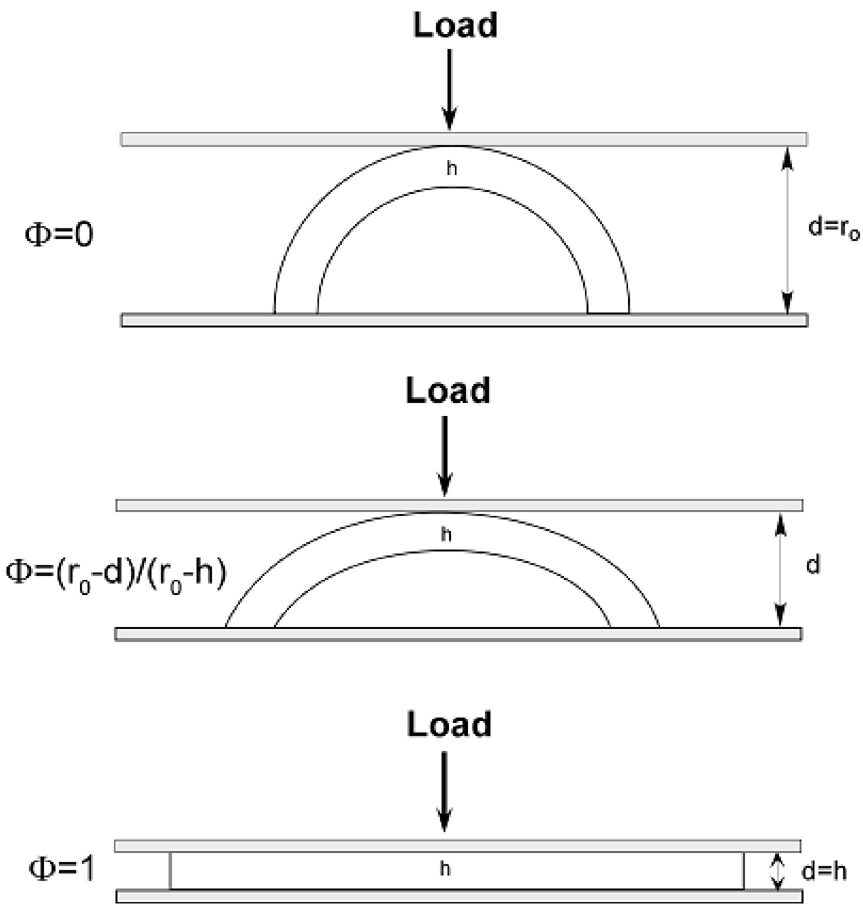


Figure 3. Schematic representation of the experimental method and the definition of the degree of flatness, Φ.

(a)
(b)
(c)

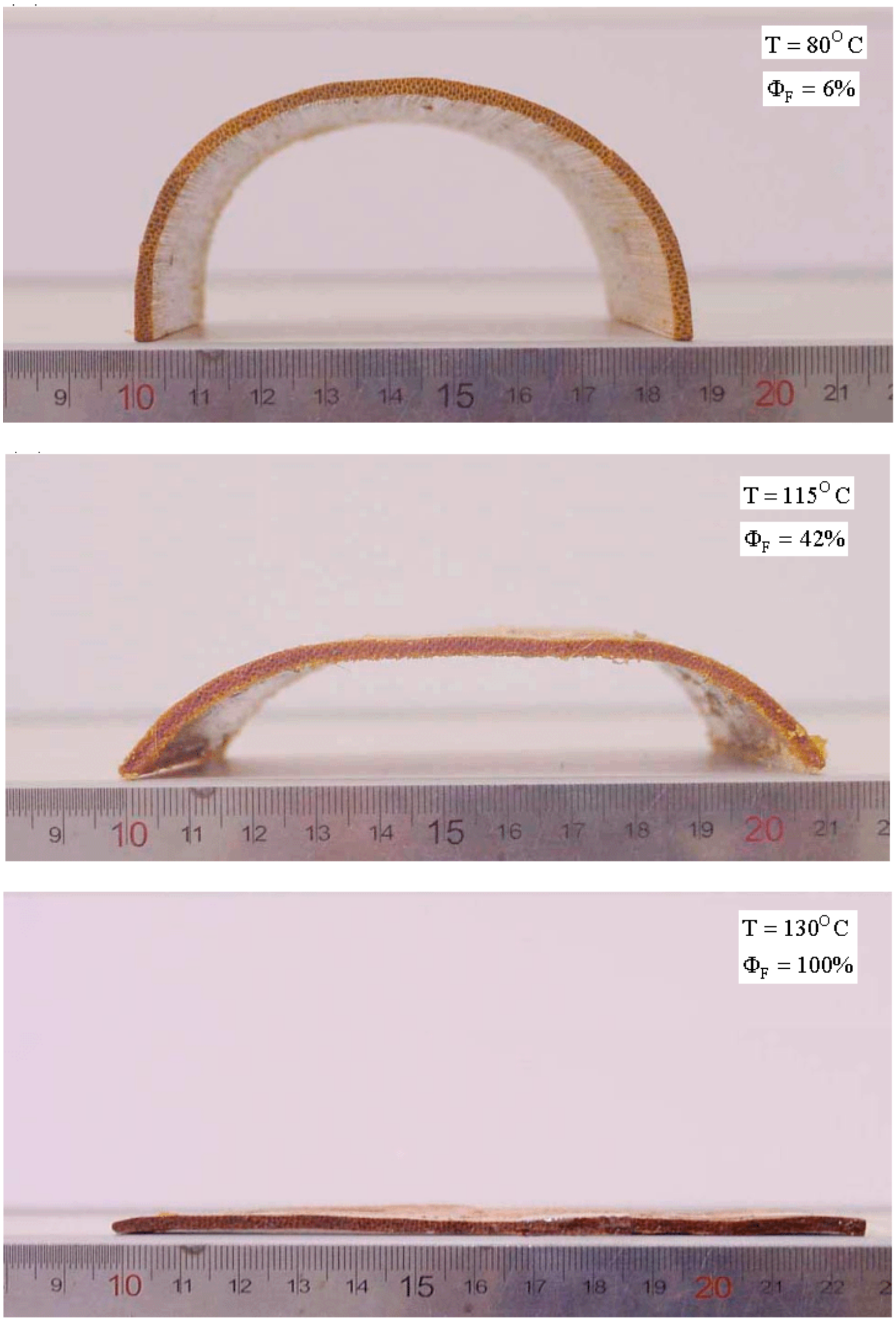


Figure 4. Photographs showing half circular cross-section specimens after pressing with constant load of 20N at various temperatures of linseed oil (a) 80ºC, (b) 115ºC and (c) 130ºC.

$\Phi_F = 6\%$ at linseed oil temperature of 80ºC (Figure 4a), while it completely deformed into a plate with $\Phi_F = 100\%$ (without cracking) at linseed oil temperature of 130ºC (Figure 4c). Although the internal stress within the specimen was not constant during testing owing to specimen shape change,

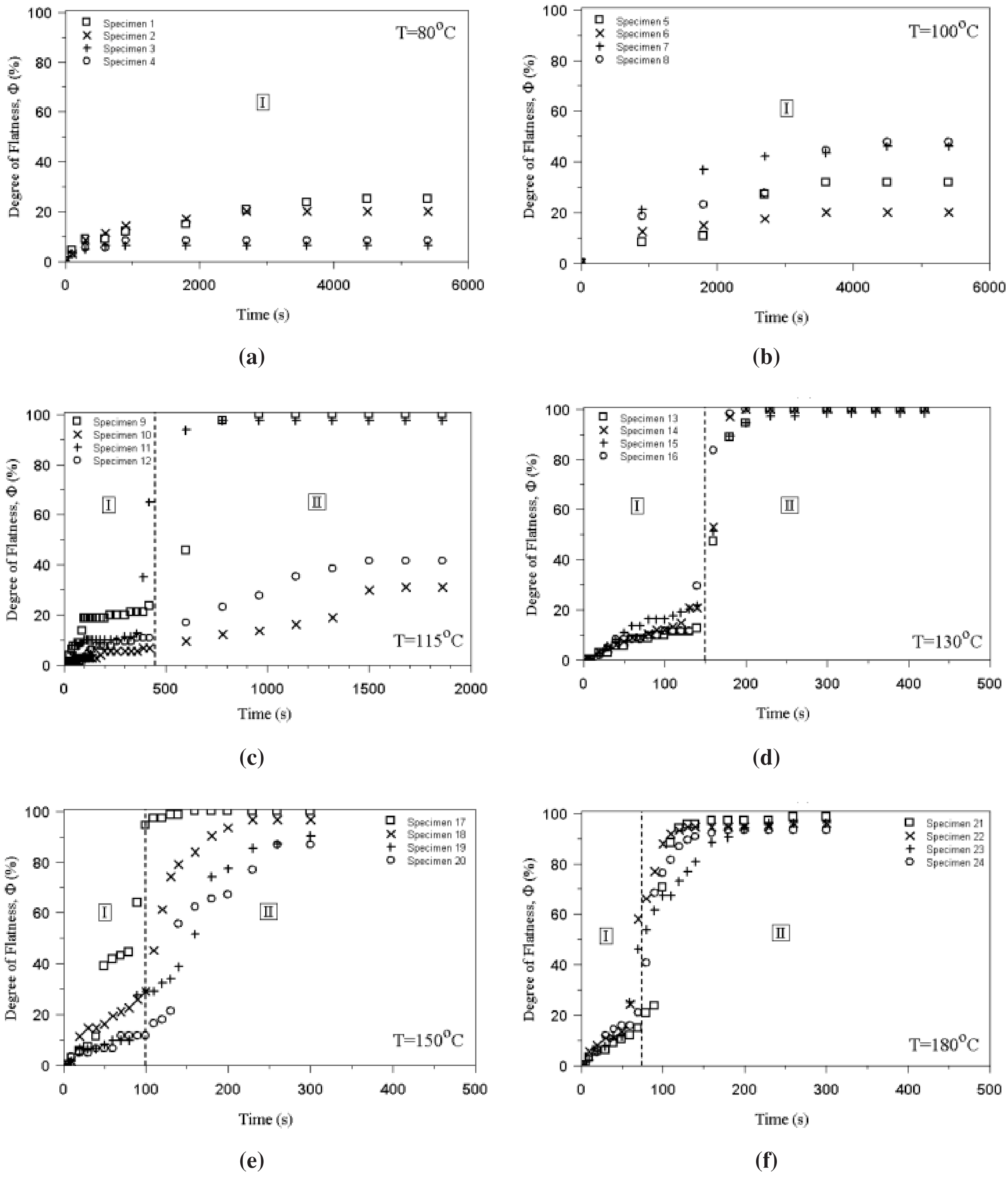


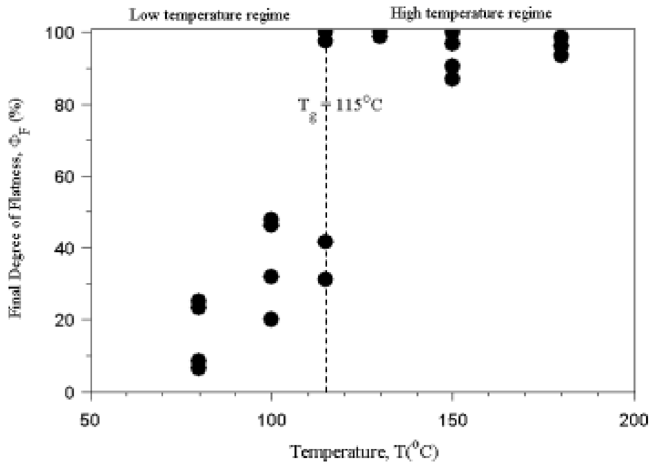
Figure 5. Deformation profiles, plots of degree of flatness against time, of bamboo specimens at various temperatures of linseed oil (a) 80ºC, (b) 100ºC, (c) 115ºC, (d) 130ºC, (e) 150ºC and (f) 180ºC. State I and state II are referred to deformation in the glassy and rubbery state, respectively. Vertical dot lines indicate time required for the transition from state I to state II.

the final value of degree of flatness still reflected a marked change in degree of softening of bamboo specimens at two different temperature regimes.

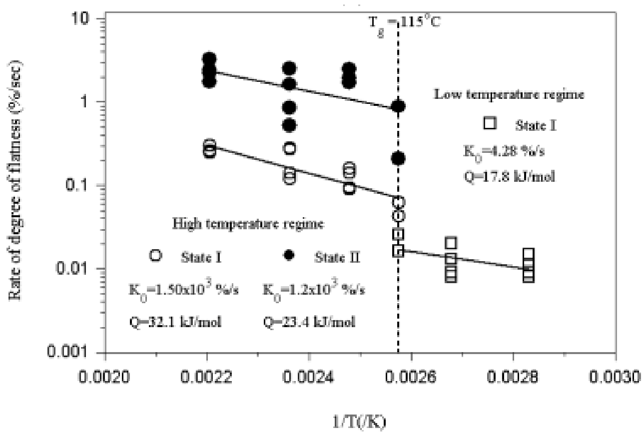
Figure 5 shows the deformation profiles, which are plots of degree of flatness against time, of bamboo specimens at six temperature treatments of linseed oil. At high temperature regime i.e. 130ºC, 150ºC and 180ºC, deformation profiles are clearly divided into two stages (Figures 5d, 5e and 5f). Bamboo specimens deformed very slowly in the first stage and then quickly deformed up to almost $\Phi_F = 100\%$ in the second stage. The transition from the first stage to the second stage occurred at 150, 100 and 75 seconds for temperatures of linseed oil at 130ºC, 150ºC and 180ºC, respectively. At low temperature regime i.e. 80ºC and 100ºC, deformations occurred only in a single stage which is similar to deformation in the first stage at high temperature regime (Figures 5a and 5b). At this low temperature regime, the final value of degree of flatness and the rate of deformation were much less than those at high temperature regime. The behavior was more complex at a temperature of linseed oil of 115ºC (Figure 5c). Some specimens deformed in a similar way to deformation at a high temperature regime while other specimens deformed like ones at a low temperature regime.

After dipping a specimen into hot linseed oil, the temperature of the specimen gradually increased from room temperature to reach the temperature of linseed oil. At low temperature of linseed oil i.e. 80ºC and 100ºC, amorphous wood constituents are in the glassy state so deformation profiles show only a single stage of deformation (state I in Figures 5a and 5b). At this condition, specimens had high strength and modulus so they were difficult to deform. At high temperature of linseed oil i.e. 130ºC, 150ºC and 180ºC, the specimen temperature was initially lower than the glass transition temperature. Because amorphous wood constituents were in the glassy state, deformation in the first stage was slow (state I in Figures 5d, 5e and 5f). When specimen temperature reached the glass transition temperature, wood materials became very much softer and much easier to deform. Within this second stage, corresponding to the rubbery state, specimens quickly deformed at a much higher rate to attain the higher value of degree of flatness (state II in Figures 5d, 5e and 5f). Time required for the transition from the first to the second stages was also found to decrease with increasing temperature. This is because higher temperature of linseed oil raised the specimen temperature to reach the glass transition temperature more quickly than lower temperature of linseed oil. A large variation in deformation behavior at 115ºC reflects a small temperature range of the glass transition temperature and also the effect of initial moisture content on the glass transition temperature. After dipping specimens into hot linseed oil and as specimen temperature increased, moisture within the specimens continuously diffused out of the wood materials. Moisture content of specimens gradually decreased with time. This increased the glass transition temperature to a higher temperature (Glasser *et al.*, 1998). As a result, a large variation of deformation behavior especially at the temperature close to the glass transition temperature (Figure 5c) could be observed.

Final degree of flatness plotted against temperature of linseed oil is shown in Figure 6a. Specimen behavior is clearly separated into two temperature regimes with the softening temperature at 115ºC. Softening temperature of bamboo in linseed oil obtained in this work is within a range of glass transition temperature reported elsewhere for various species of wood, ranging from 60ºC to 235ºC (Lenth, 1999). At the low temperature regime, all values of final degree of flatness, Φ_F, are less than 50%. The values of Φ_F increase slightly as temperature increases. At high temperature regime, all values of final degree of flatness, Φ_F, are close to 100%. Effect of temperature on the rate of degree of flatness, K, was described well using the Arrhenius equation which takes the form $K = K_0 e^{(\frac{-Q}{RT})}$, where K_0 is the frequency factor, Q is the activation energy of the process, T is absolute temperature and R is the universal gas constant. This equation has been successfully applied to



(a)



(b)

Figure 6. Plots of (a) final degree of flatness, Φ_F, against temperature of linseed oil and (b) reciprocal absolute temperature of linseed oil against log rate of degree of flatness of deformations in state I and state II.

describe softening behavior of potato cell wall structure (Alvarez and Canet, 2001). Rate of degree of flatness plotted against the reciprocal of absolute temperature is shown in Figure 6b. Values of K_0 and Q derived from curve fitting are also shown in Figure 6b. Rate of degree of flatness in state I at low temperature and high temperature regimes almost falls onto the same curve which implies that deformation in state I at low temperature and high temperature regimes undergo the same temperature dependent process. Small increment of rate of degree of flatness of state I deformation at high temperature regime might be a result of the presence of temperature gradient within the specimen during the process of heating bamboo the specimen in linseed oil. The outer shell of the specimen might reach the glass transition temperature, and therefore be in the rubbery state, while the inner core of the specimen (lower temperature) is still in the glassy state. This makes an overall observed deformation in state I at the high temperature regime slightly higher than that at the low temperature regime, where only deformation in the glassy state occurs. At high temperature regime, rate of degree of flatness of state II is about one order of magnitude higher than that of state I. This indicates that bamboo specimen in the rubbery state is about 10 times easier to deform than that in the glassy state. Values of Q obtained, ranging from 18 kJ/mole to 32 kJ/mole for bamboo specimens at water saturated condition (moisture content 46±8%), are in similar trend with data reported elsewhere. Lenth (1999) reported the activation energy for thermal softening of southern pine at moisture content 0%, 5%, 12% and 20% to be 109 kJ/mole, 88 kJ/mole, 84 kJ/mole and 63 kJ/mole, respectively. Further investigation should be made on the effect of moisture content on the glass transition temperature and deformation behavior of bamboo culms, the movement of moisture out of bamboo specimens after dipping specimens into linseed oil and the thermal heat transfer between linseed oil and bamboo specimens.

Conclusions

The following conclusions can be drawn from this work:

1. The simple technique developed in this work was successfully employed to study the thermal softening of bamboo culms in linseed oil.
2. The proposed parameter, the degree of flatness, was proved to be capable of describing the deformation behavior of bamboo culms in linseed oil under loading condition.
3. Thermal softening behavior of bamboo culms in linseed oil was divided into two temperature regimes with the glass transition temperature at 115ºC.
4. At the low temperature regime, deformation occurred slowly and showed only a single stage of deformation corresponding to deformation in the glassy state.
5. At the high temperature regime, specimens deformed slowly in the first stage and then rapidly deformed in the second stage when specimen temperature reached the glass transition temperature, corresponding to deformations in the glassy and rubbery states, respectively.
6. Effect of temperature on the rate of softening was well described by means of the Arrhenius equation with the activation energy ranging from 18 kJ/mole to 32 kJ/mole.

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