

# Properties of wood plasticization with octanoyl chloride in a solvent-free system

J.-H. Wu, T.-Y. Hsieh, H.-Y. Lin, I.-L. Shiau, S.-T. Chang

**Abstract** Diffuse reflectance Fourier transform infrared spectroscopy (DRIFT), solid state cross-polarization/magic-angle-spinning  $^{13}\text{C}$ -nuclei magnetic resonance spectroscopy (CP/MAS  $^{13}\text{C}$ -NMR), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and thermomechanical analysis (TMA) were used in this study to elucidate the characteristics of wood sawdust after esterification. Results revealed that thermoplastic wood was produced by solvent-free esterification using octanoyl chloride. Increasing the duration of treatment enhanced the extent of esterification. After octanoylation, the crystallinity of wood sawdust was decreased, whereas thermoplasticity, hydrophobicity, and thermal stability were enhanced. The complete flow of the octanoylated wood meal was achieved at around  $300^\circ\text{C}$  under a force of 0.01 N, indicating that octanoylated wood sawdust is a good thermoplastic material.

## Introduction

Owing to the gradual depletion of global forest resources, innovative use and recycling of natural materials is greatly needed for environmental conservation and sustainable management of natural resources. There are many by-products (wood shavings and sawdust) in the wood-processing industry. Therefore, one of

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the unprecedented challenges in the wood products industry is how to improve the properties and performance of the wood products using the woody residues and how to recycle them. Among the known methods of wood utilization, wood plasticization is capable of becoming one with potential.

Plasticization of wood sawdust can be obtained either with wood-plastic composites (WPC) or by chemical modifications. In the former method, however, a major problem is the incompatibility between a natural fiber and thermoplastic polymers. Many problems have been identified including poor dispersion of the fiber due to intermolecular hydrogen bonding (Raj et al. 1989), and wide polarity differences of the surfaces preventing efficient polymer-fiber bonding (Maldas et al. 1989). To date, several physical and chemical approaches have been used to overcome these problems. One of these approaches is chemical modification which not only improves the thermoplasticity of wood (Morita and Sakata 1986; Shiraishi 1991; Nakano 1994), but also increases dimensional stability, as well as fire and decay resistance (Rowell 1983; Engonga et al. 1999).

Shiraishi et al. (1979a) reported that the thermofluidity was observed when wood samples esterified with appropriate acid chlorides in  $N_2O_4$ -dimethylformamide-pyridine medium, and that increasing the chain length of the alkyl substituent led to a decrease in the apparent melting temperature. In general, the esterification agents must contain at least six atoms of carbon to improve the thermoplastic properties of wood (Shiraishi et al. 1979b). Besides conventional anhydrides, e.g., trifluoroacetic acid anhydride (Nakano 1994), maleic anhydride (Kazayawoko et al. 1998), maleic anhydride modified polypropylene (MAPP) (Takase and Shiraishi 1989), and styrene-maleic anhydride (SMA) (Simonsen 1998), recent progress has allowed the esterification of lignocellulosic materials with fatty acid chlorides without any base or solvent to remove gaseous acid chloride from the reaction system by applying a nitrogen stream (Thiebaud and Borredon 1995; Thiebaud et al. 1997) or vacuum (Kwatra et al. 1992). Those solvent-free reaction systems not only reduce the solvent used, but also lessen environmental pollution. Furthermore, the fatty acid cellulose esters (FACE) have been identified as potential biodegradable plastics (Kwatra et al. 1992). Therefore, it is worthy to investigate further the plasticization of woody residues using the long chain fatty acid chloride in a solvent-free system.

Sugi (*Cryptomeria japonica* D. Don), a fast growing Japanese cedar, is one of the important plantation tree species grown in Taiwan. Unfortunately, owing to the color variations of the different forms of sugi and the susceptibility of its heartwood to discoloration induced by environmental factors (Abe et al. 1994; Abe and Oda 1994; Takahashi 1996, 1998; Kubo and Ataka 1998; Chang et al. 2000), use of sugi solid wood in Taiwan is often restricted. Thus, to date, how to utilize sugi is a major challenge for the forestry and wood industry. Research breakthroughs in exploring the utilization methods of sugi wood of different forms, e.g., low-quality wood, wood shavings and even sawdust, are imperative.

The purpose of this study is to find appropriate treatment conditions to convert the sugi sawdust into thermally meltable materials by solvent-free octanoylation under a nitrogen stream. Furthermore, the properties of esterified wood are also investigated by using various spectrometric techniques.

## Experimental

### Materials

Sugi sawdust (*Cryptomeria japonica* D. Don) (60–80 mesh) was obtained from the sawmill of the experimental forest of the National Taiwan University. It was used after extraction of soluble fractions in a Soxhlet apparatus for 24 h with a 1:2 (v/v) mixture of ethanol and toluene followed by washing with distilled water and then dried at 105°C for 12 h.

Octanoyl chloride was purchased from Sigma Chemical (St. Louis, MO). The other chemicals and solvents used in this experiment were of the highest quality available.

### Octanoylation

Two grams of dried sugi sawdust and 0.3 mol of octanoyl chloride were successively introduced into the reactor. The reaction was then conducted at 130°C with stirring for 1, 2, and 4 h, respectively. To trap the hydrogen chloride formed during the reaction, a continuous nitrogen stream (200 ml/min) was applied into the reactor and a wash-bottle containing an aqueous saturated sodium hydroxide solution was placed at the outlet of the condenser. At the end of octanoylation, the esterified wood was washed with 50 ml of ethanol and then Soxhlet-extracted using ethanol for 6 h to remove the excess and residues of acid chloride. Finally, the esterified wood was dried at 105°C for 12 h, and weight percent gain (WPG) was then calculated.

### Color measurement

The color of wood sawdust was measured with a color and color difference meter (Dr. Lange Co., Germany) using a D<sub>65</sub> light source. The tristimulus values X, Y, and Z of all specimens were obtained directly from the colorimeter. Then the recommended CIE (Commission Internationale d'Eclairage) L\*, a\* and b\* color parameters were computed, followed by calculating Δa\* (difference of a\* component), Δb\* (difference of b\* component), and ΔC\* (chroma difference) with the following formulae (Chang 1986, 1997).

$$\Delta a^* = a_t^* - a_s^*$$

$$\Delta b^* = b_t^* - b_s^*$$

$$\Delta C^* = C_t^* - C_s^*$$

where  $a_t^*$ ,  $b_t^*$ , and  $C_t^* = a^*$ ,  $b^*$ , and  $C^*$  of the treated sample, respectively;  $a_s^*$ ,  $b_s^*$ , and  $C_s^* = a^*$ ,  $b^*$ , and  $C^*$  of the control reference, respectively;  $L^*$ =the value on the white/black axis,  $a^*$ = the value on the red/green axis,  $b^*$ = the value on the blue/yellow axis,  $C^*$ =chroma= $[(a^*)^2+(b^*)^2]^{1/2}$ .

### DRIFT spectroscopy analysis

The chemical structure analysis of esterified wood powder was carried out using diffuse reflectance Fourier transform infrared spectroscopy (DRIFT). The spectra were obtained on a Bio-rad FTS-40 (USA). Data were collected from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> with 64 scans for each sample. The resolution was 4 cm<sup>-1</sup>.

### X-ray diffraction measurement

X-ray diffractograms were obtained with powder samples. A MAC science M03XHF analyzer (Japan) was used. The diffraction patterns were measured from  $2\theta=2$  to  $35^\circ$  using  $\text{CuK}\alpha_1$  radiation at 40 kV and 30 mA.

### Solid state CP/MAS $^{13}\text{C}$ -NMR analysis

Solid state of the wood samples were examined by CP/MAS  $^{13}\text{C}$ -NMR. The spectra were recorded on a Bruker DMX-300 FT-NMR spectrometer (Germany), at a frequency of 75.5 MHz. Chemical shifts were calculated relative to TMS for NMR control.

### Thermal properties

A TA TGA-2950 thermogravimetric analyzer (USA) was used to study the thermal properties of esterified wood. The heating rate was set at  $20^\circ\text{C}/\text{min}$  and the temperature ranged from 25 to  $600^\circ\text{C}$ . Measurements on 5 mg samples were carried out in a nitrogen atmosphere (100 ml/min).

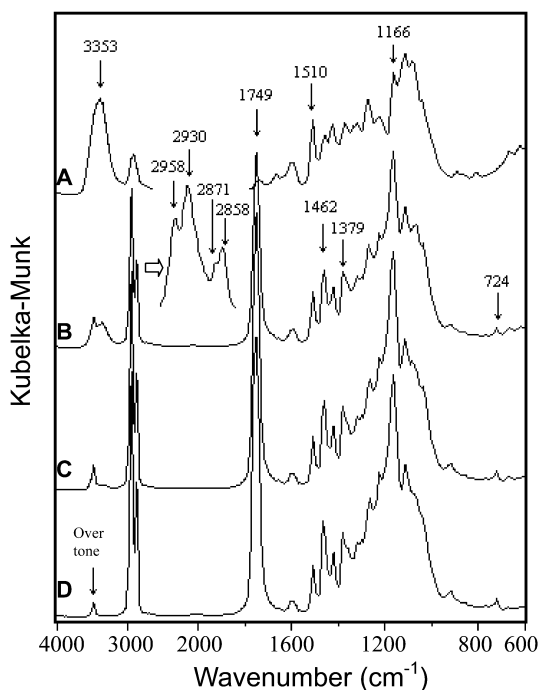
In addition, a TA TMA-2940 thermomechanical analyzer (USA) was used to evaluate the softening temperature of octanoylated wood sawdust. The wood samples (approximately 6–10 mm in size) were run in a quartz glass vial using the dilatometer probe. Nitrogen with a flow rate of 100 ml/min was used as a purge gas, and the experimental conditions included a temperature range of 25 to  $350^\circ\text{C}$ , a heating rate of  $1^\circ\text{C}/\text{min}$ , and a force of 0.01 N.

## Results and discussion

### Effects of octanoylation on the chemical structure of wood

To understand the chemical change of sugi wood sawdust before and after octanoylation, a DRIFT spectroscopy and a CP/MAS  $^{13}\text{C}$ -NMR spectrometer were used. The results showed that, after octanoylation, the intensity of the absorption band of O-H stretching vibration ( $3353\text{ cm}^{-1}$ ) was reduced by increasing the reaction time (Fig. 1), whereas those of C=O ( $1749\text{ cm}^{-1}$ ) and C-O ( $1166\text{ cm}^{-1}$ ) bands were increased. When the reaction duration was 4 h, the absorption peak of hydroxyl groups almost disappeared (Fig. 1D), elucidating that acyl groups had substituted for the hydrogen atoms of the hydroxyl groups of wood sawdust. In addition, the intensity of methyl and methylene groups at  $3000\text{--}2840\text{ cm}^{-1}$  increased significantly after octanoylation in which C-H symmetrical stretching ( $\nu_s$ ) and asymmetrical stretching ( $\nu_{as}$ ) bands including  $2958\text{ cm}^{-1}$  ( $\nu_s\text{ CH}_3$ ),  $2930\text{ cm}^{-1}$  ( $\nu_s\text{ CH}_2$ ),  $2871\text{ cm}^{-1}$  ( $\nu_{as}\text{ CH}_3$ ), and  $2858\text{ cm}^{-1}$  ( $\nu_{as}\text{ CH}_2$ ) could be clearly distinguished. Meanwhile, the intensities of C-H bending vibrations, such as  $1462\text{ cm}^{-1}$  ( $\delta_s\text{ CH}_2$ ),  $1379\text{ cm}^{-1}$  ( $\delta_s\text{ CH}_3$ ), and  $724\text{ cm}^{-1}$  ( $\rho\text{ CH}_2$ ), were also enhanced. In general, the band resulting from the methylene rocking vibration ( $\rho\text{ CH}_2$ ), in which all of the methylene groups in phase, appears at about  $720\text{ cm}^{-1}$  for straight-chain alkanes of seven or more carbon atoms (Silverstein et al. 1991). Thus, it indicated that octanoylation had indeed occurred when the sugi sawdust reacted with octanoyl chloride under a solvent-free system.

Furthermore, to understand the effects of reaction time on esterification, the absorption intensity of  $1510\text{ cm}^{-1}$  was used as an internal standard. The ratios of the intensity of peak at  $3353\text{ cm}^{-1}$  to  $1510\text{ cm}^{-1}$  calculated for 0, 1, 2, and 4 h of reaction were 1.8, 0.6, 0.1, and 0.0, respectively (Table 1). These data demonstrated that the contents of O-H of wood sawdust were decreased by prolonging



**Fig. 1.** DRIFT spectra of wood sawdust treated with octanoyl chloride for various time. A control; B 1 h; C 2 h; D 4 h

the reaction time (Table 1), whereas the relative intensities of  $2930\text{ cm}^{-1}$  (C-H) and  $1749\text{ cm}^{-1}$  (C=O) as well as the WPG were increased. It indicated that increasing reaction time enhanced the extent of octanoylation. The highest esterification (WPG=86.5) was achieved when the reaction time was 4 h.

On the other hand, the  $^{13}\text{C}$ -NMR spectra of wood sawdust showed that the peaks at 13.7, 22.6, 24.6, 29.3, 31.8, 32.8, and 33.6 ppm, all of which belong to straight-chain alkanes of seven carbons, were substantially observed after octanoylation (Fig. 2B). Moreover, a peak at 172.1 ppm, assigned to the octanoyl group ( $\text{CH}_3(\text{CH}_2)_6\text{CO}-$ ), was also found in Fig. 2B. These data confirmed further that the sugi sawdust was octanoylated after being treated with octanoyl chloride. In addition, Fig. 2A showed a characteristic carbohydrate pattern, namely C1 (104.7 ppm), C4 (88.6 ppm and 83.5 ppm), C2 (74.6 ppm), C3, C5 (71.9 ppm), and C6 (65.1 ppm and 62.1 ppm). However, after octanoylation, the intensities of

**Table 1.** Relative intensity of DRIFT absorption peaks and weight percent gain for octanoylated wood sawdust

Reaction time (h)	Relative intensity			WPG
	$A_{3353\text{ cm}^{-1}/1510\text{ cm}^{-1}}$	$A_{2930\text{ cm}^{-1}/1510\text{ cm}^{-1}}$	$A_{1749\text{ cm}^{-1}/1510\text{ cm}^{-1}}$	
Control	1.8	0.6	0.2	–
1	0.6	3.9	4.8	37.5
2	0.1	5.5	7.0	72.6
4	0.0	6.4	7.3	86.5

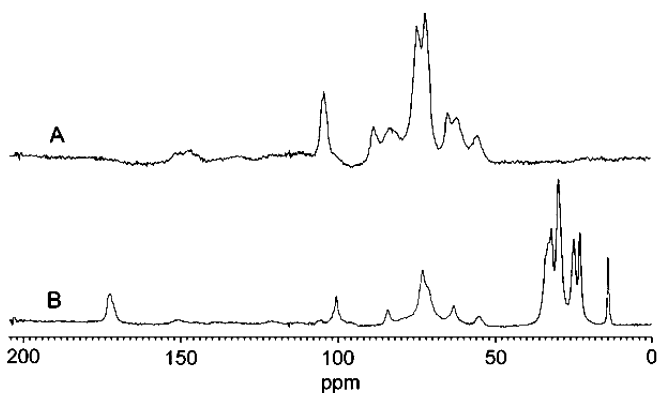


Fig. 2. Solid state CP/MAS  $^{13}\text{C}$ -NMR spectra of wood sawdust before (A) and after (B) esterification with octanoyl chloride for 2 h

peaks at 88.6 ppm (crystalline C4) and 65.1 ppm (crystalline C6) were decreased and shifted to 84.1 ppm (amorphous C4) and 63.0 ppm (amorphous C6), respectively. These results indicated that cellulose in wood was decrystallized after treatment with octanoyl chloride, a long-chain acid chloride. Furthermore, the diffraction patterns of wood sawdust before and after octanoylation were obtained by using an XRD analyzer. As shown in Fig. 3, there were two diffraction rays for  $2\theta=22^\circ$  and  $15^\circ$  before treatment, by which the calculated crystallinity index (CrI) was 43.3%. After octanoylation, the former ray due to the 002 plane of crystal lattice was shifted to the amorphous of  $19.6^\circ$ , and the other ray corresponding to the reflection of planes 101 and  $10\bar{1}$  of the native cellulose lattice vanished and a new peak appeared at  $2\theta=4.1^\circ$  (Fig. 3B, C, D). These results are similar to the spectra obtained by Thiebaud et al. (1997). The peak of crystalline phase could not be observed after octanoylation, and the extent of decrystallization was a function of ester content (or WPG). This indicated that prolonging

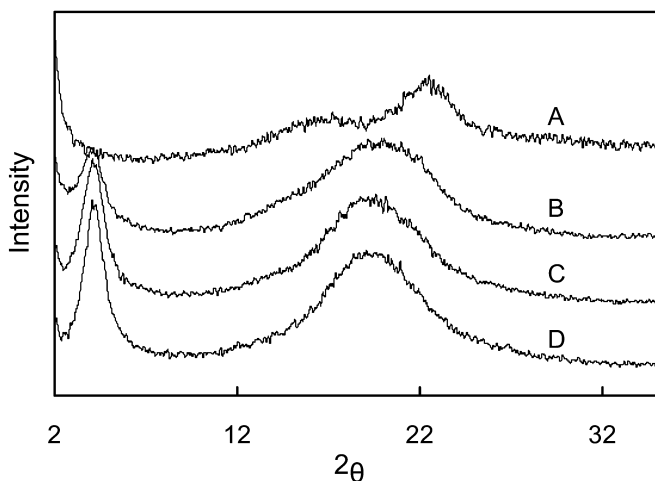


Fig. 3. X-ray diffraction diagrams of wood sawdust treated with octanoyl chloride for various time. A control; B 1 h; C 2 h; D 4 h

the reaction time increased the amorphous regions of cellulose, thus prompting reagents to react with new hydroxyl groups of those regions and enhance the accessibility.

### Effects of octanoylation on the color of wood sawdust

Table 2 shows the CIE  $L^*$ ,  $a^*$ , and  $b^*$  color parameters of wood sawdust before and after octanoylation. As can be seen,  $a^*$  and  $b^*$  values of wood sawdust were not significantly different from those of several octanoylated ones whose  $\Delta a^*$  and  $\Delta b^*$  values were in the range of 1.6–2.1 and 1.6–2.8, respectively. In other words, there was almost no influence on the hue of wood sawdust after treatment with octanoyl chloride. As for the lightness ( $L^*$ ) and chroma of wood sawdust, as shown in Table 2, the chroma increased slightly ( $\Delta C^* = 2.3$  to 3.1), whereas the  $L^*$  value reduced from 70.3 to 61.2–63.4 after octanoylation. These results indicate that the color of esterified wood sawdust becomes slightly less bright than that of untreated sawdust.

### Effects of octanoylation on the thermal properties of wood sawdust

To evaluate the thermal properties of wood sawdust after octanoylation, a thermogravimetric analyzer and a thermomechanical analyzer were used. The thermogravimetric analysis (TGA) of wood sawdust treated with octanoyl chloride for various durations is shown in Fig. 4. The TGA of untreated wood sawdust (Fig. 4A) indicated a water loss of 3.9% between 25 and 200°C. A water loss of less than 1% was obtained over the same temperature range for octanoylated ones.

Table 2. Color variations of wood sawdust after esterification with octanoyl chloride

Reaction time (h)	CIE LAB			$\Delta a^*$	$\Delta b^*$	$\Delta C^*$
	$L^*$	$a^*$	$b^*$			
Control	70.3	8.5	23.0	–	–	–
1	61.2	10.6	24.6	2.1	1.6	2.3
2	63.4	10.1	25.4	1.6	2.4	2.8
4	61.2	10.1	25.8	1.6	2.8	3.1

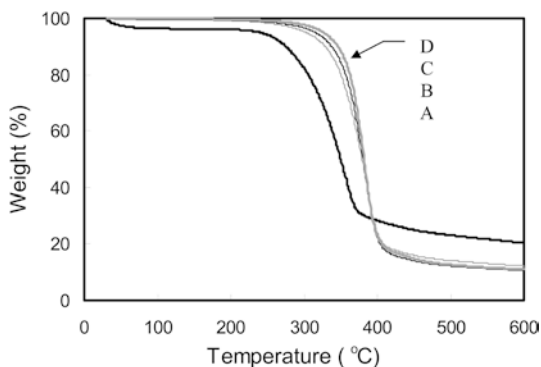


Fig. 4. Thermogravimetric analysis of wood sawdust treated with octanoyl chloride for various time. A control; B 1 h; C 2 h; D 4 h

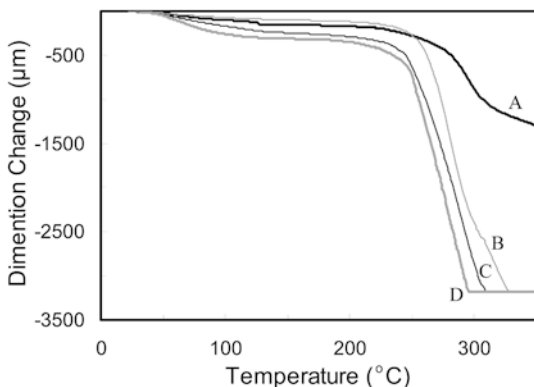
This could be accounted for by the increased hydrophobicity of wood sawdust after octanoylation because the hydroxyl groups of crystalline and amorphous regions of wood sawdust were replaced by the hydrophobic acyl groups ( $\text{CH}_3(\text{CH}_2)_6\text{CO}-$ ).

In general, the temperature of wood pyrolysis was over  $200^\circ\text{C}$  (Fengel and Wegener 1989). Indeed, on the TGA curve for the untreated wood (Fig. 4A), the weight loss increased gradually at temperatures above  $200^\circ\text{C}$ . A maximum weight loss was obtained when the temperature was around  $350^\circ\text{C}$ . In contrast, the temperature of maximum weight loss increased about  $30^\circ\text{C}$  after octanoylation (Table 3). These observations showed that octanoylation enhanced the thermostability of wood. In addition, comparisons of the amount of carbonized residue at  $600^\circ\text{C}$ , before and after octanoylation as shown in Table 3, indicated that 20.4% carbonized residue was obtained from the untreated sawdust, and was less than 13% for octanoylated ones. These results suggested that the acyl groups in the octanoylated wood were eliminated with the volatile products and did not contribute to the carbonized residue.

On the other hand, comparing the thermoplasticity of the octanoylated wood sawdust with that of the untreated one, we obtained TMA curves as shown in Fig. 5. It was obvious that no thermal softening was observed for the untreated sugi sawdust until it was heated to  $250^\circ\text{C}$ . In contrast, after octanoylation, the wood sawdust began softening at about  $250^\circ\text{C}$ , and showed a sharp drop due to the complete flow of the sample when the temperature reached about  $300^\circ\text{C}$ . Moreover, the temperature of thermal flow of octanoylated wood decreased

**Table 3.** Thermal properties of wood sawdust esterified with octanoyl chloride

Reaction time (h)	Weight % at		DTG Max. Peak
	$200^\circ\text{C}$	$600^\circ\text{C}$	
Control	96.1	20.4	$352.9^\circ\text{C}$
1	99.2	12.2	$384.0^\circ\text{C}$
2	99.4	10.6	$382.3^\circ\text{C}$
4	99.7	11.0	$381.8^\circ\text{C}$



**Fig. 5.** Thermomechanical analysis of wood sawdust treated with octanoyl chloride for various time. A control; B 1 h; C 2 h; D 4 h



gradually with increasing esterification time. Accordingly, the octanoylated wood sawdust possesses very good thermoplasticity indeed.

## Conclusions

Wood has almost no thermoplasticity and does not thermally flow or melt. However, wood can be converted into thermoplastic materials through some types of chemical modifications. Among the various plasticization methods, solvent-free octanoylation is a good chemical process. According to the data obtained from the analyses of DRIFT, solid state  $^{13}\text{C}$ -NMR, XRD, TGA, and TMA, prolonging the octanoylation time not only increases WPG of sugi sawdust, but also the acyl content. It is known that hydroxyl groups of wood are most often the reactive sites. When the reaction time was 4 h, the absorption characteristics of hydroxyl groups almost disappeared. This shows that, under a solvent-free system, an excellent reactivity exists between sugi sawdust and octanoyl chloride. In addition, the brightness of octanoylated sugi sawdust is slightly less than that of the untreated one, but the decrystallization of cellulose by acylation with octanoyl chloride enhances the thermoplastic properties and thermal stability of wood, while increasing its hydrophobicity. Furthermore, complete flow of the octanoylated sugi meal was achieved at about 300°C under a force of 0.01 N. Therefore, it is concluded that octanoylated wood sawdust is an excellent thermoplastic material.

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