A MICROWAVE-ASSISTED EXTRACTION METHOD FOR DETERMINING HOT WATER SOLUBILITY OF WOOD

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ABSTRACT

A microwave-assisted extraction method is proposed as an alternative to the conventional method for determining the hot water solubility of wood. In this alternative method, microwave heating substitutes for the boiling water to extract part of the extraneous components as well as starches in wood tissues. Experimental results indicate that 100 mL water can be heated to boiling in only 75 s under the microwave radiation. Hence, only 15–20 min are required to complete the extraction procedure for *Liquidambar formosana* Hance and *Swietenia mahagoni* Jacq., 10 min and 5 min for *Taiwania cryptomerioides* and *Cunninghamia lanceolata* (Lamb) Hook, respectively. In general, a 15-min microwave heating has the potential to be an alternative to the conventional method, which requires 3 h for the hot water solubility determination.

Keywords: Microwave-assisted extraction, hot water solubility, wood meal, irradiation, heat.

INTRODUCTION

The water-soluble materials in wood include inorganic salts, sugars, polysaccharides, cycloses and cyclitols, and some phenolics (Browning 1967). In the conventional method for determining cold water solubility, wood sawdust is extracted with water at 23 ± 2°C with stirring for 48 h to remove part of the extraneous components such as inorganic compounds, tannins, gums, sugars, and coloring matter present in wood (Browning 1967). For the determination of hot water solubility, wood meal is extracted with water under reflux in a boiling water bath for 3 h to remove the above-mentioned components plus starches. These procedures were revised in 1999 (TAPPI Standard T207, 1999). It takes 3 h to perform the extraction. It is tedious and time-consuming. In modern analytical practice, saving time is an essential target for most chemists. However, a new method should be proven in its applicability, including precision and accuracy, in addition to time-saving. Therefore, a time-saving procedure applicable to determine hot water solubility of wood is worthy of being investigated.

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In past decades, the application of microwave-assisted extraction was studied in a wide range of materials such as soils, plant and animal tissues, and a variety of manufactured products (Camel 2000; Carro et al. 1997; Kingston and Jassie 1998; Llompart et al. 1997; Pensado et al. 2000). The application is increasing. In general, the extractions assisted by microwave-irradiation are more selective and rapid with similar or better recoveries in comparison with the conventional extraction processes (Zuloaga et al. 1998). In addition, microwave-assisted extraction has the advantage of using less solvent and energy than conventional techniques. Thus, an application of microwave-assisted extraction has the potential to be an alternative to the conventional method of determining the hot water solubility of wood.

The objective of the present work was to compare hot water extraction for four Taiwan wood species (two hardwoods and two softwoods) with water under reflux in a microwave oven to the conventional method.

MATERIALS AND METHODS

Apparatus

A 720-watt power home-use microwave oven (ME-66M, National Brand, Taiwan) was obtained from a local supplier, modified, and assembled as a microwave-assisted apparatus as shown in Fig. 1. An electronic balance (AND HR-200, Japan, precision: 0.1 mg) was used to measure the weight of wood dust.

Materials

Four 20–30-yr-old trees were obtained from plantations near Taichung. The trees included two hardwoods—Formosan sweet gum (*Liquidambar formosana* Hance) and mahogany (*Swietenia mahagoni* Jacq.) and two softwoods—Taiwania (*Taiwania cryptomerioides* Hayata) and china fir (*Cunninghamia lanceolata* (Lamb) Hook). Using a power-driven saw, a sample 1,000 mm long was cut from each log. All bark and knots, decayed portion, and other abnormalities were separated and discarded. The specimens were stored in a dry, cool room for wood analysis.

Logs were debarked and ground in a laboratory grinder. The finer material was separated by sifting on a 40-mesh screen. Accepts (size: 40–60 mesh) were used for hot water extraction.

Microwave extraction method

A 2-g sample was suspended in a 250-mL round bottom flask refluxed with 100 mL distilled water. The condenser was connected to the flask. The suspensions were irradiated for 5 to 20 min in a modified microwave oven (Fig. 1). The suspensions were heated to a boil. After irradiation at fixed times, the samples were transferred to a tared 1G3 glass filter for filtering. The sample was washed with 200 mL hot water and then dried to a constant weight at 105 ± 3°C.

Conventional extraction method

Two g of air-dry wood meal was transferred to a 250-mL round bottom flask attached to the reflux condenser, 100 mL hot distilled water was added, and the flask was placed in a boiling water bath with hot plate heating and digested for 3 h.

The water level of the bath was held above the stock level in the flask. The contents of the flask were transferred to a tared 1G3 glass filter, which had been previously dried to a constant weight at 105 ± 3°C in an air-circulating oven.

The hot water solubility was calculated as:

$$S(\%) = [(A - B)/A] \times 100 \qquad (1)$$

where

S% is the hot water solubility
A is the initial weight (oven-dry) of wood specimen
B is the weight (oven-dry) of wood specimen after extraction

The reported hot water solubility is the average of 5 determinations, to the nearest of 0.01%. The relative standard deviation (RSD) is also given.

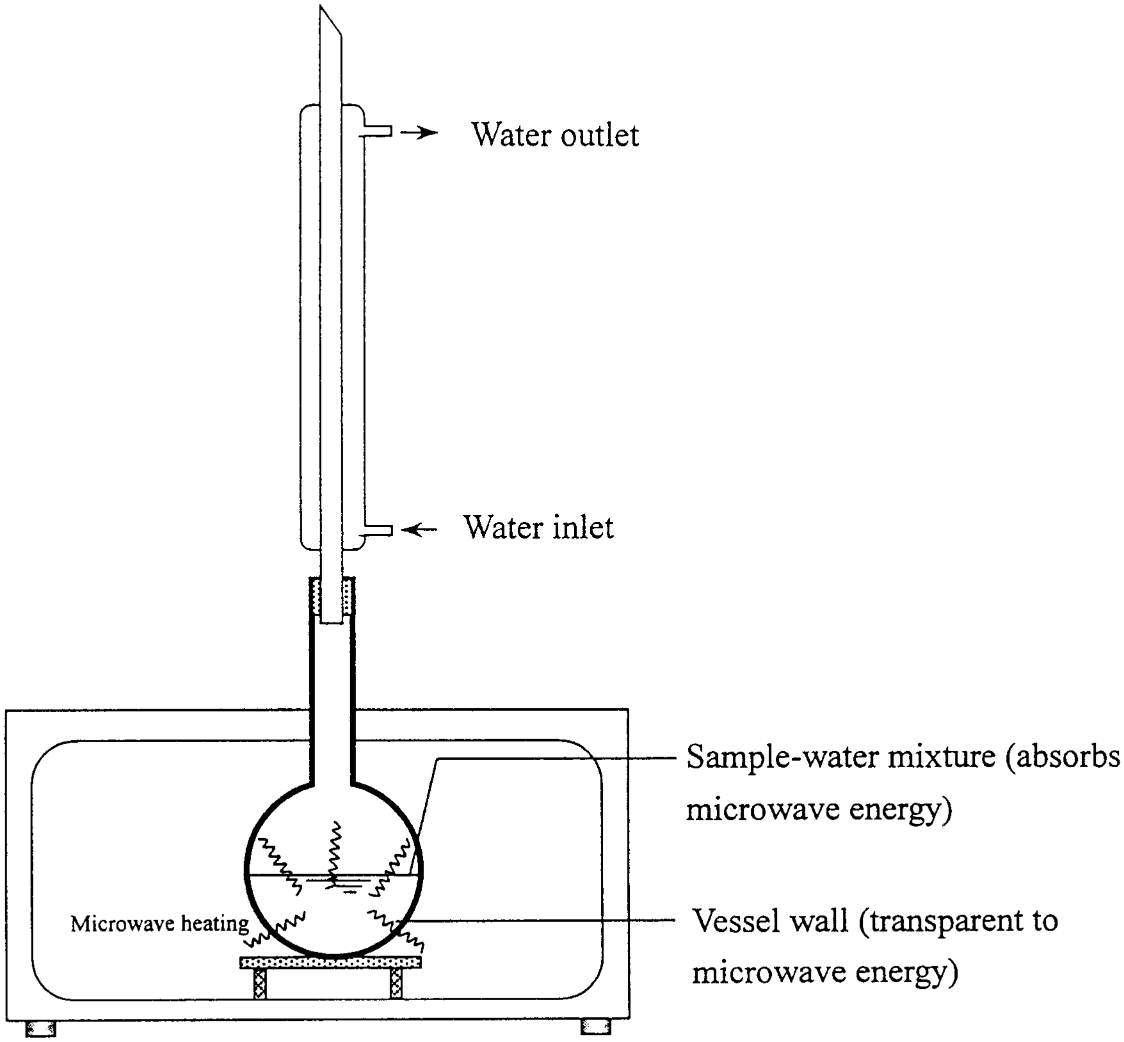

FIG. 1. A schematic diagram for illustrating the mechanism of microwave heating.

RESULTS AND DISCUSSION

Microwave heating

With conventional conductive heating, it usually takes 1 to 3 h or longer to complete a wet digestion for a wood sample in an open vessel. However, with microwave heating, it can be completed in 5–20 min. The difference is due to the sample heating method. Because vessels used in conductive heating are usually poor conductors of heat, it takes time to heat the vessel and transfer that heat by convection to the solution. On the other hand, it is easily understood that microwave energy is transferred directly to all of the molecules of water solution nearly simultaneously without heating the vessel. Therefore, when using microwave heating, the solution reaches its boiling point very quickly. Figure 2 demonstrates the temperature profile of the 100-mL distilled water solution by employing microwave heating at 720-watt power. The temperature rises rapidly, and reaches the boiling point (100°C) of water after only 75 seconds.

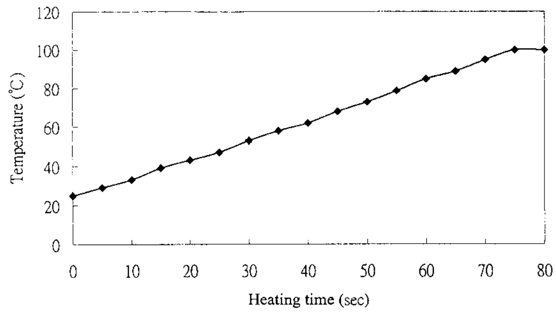

FIG. 2. The corresponding temperature of 100 mL water in microwave oven vs. heating time.

Determination of the hot water solubility by microwave-assisted method

In order to examine the applicability of microwave heating in the determination of hot water solubility, wood meal of four over 20-yr-old trees was extracted with the microwave-assisted method. Their hot water solubility was compared to those obtained by the conventional method. Table 1 lists the hot water solubility of the four wood specimens under varying microwave times.

As shown in Table 1, the quantity of detection increases with microwave irradiation time from 5 to 20 min, and approximates that obtained by the conventional method for Formosan sweet gum, mahogany and Taiwania wood species. This shows that the determinations of hot water solubility by conventional and microwave methods agree well for these wood species. Whereas the detection of quantity of Taiwania and China fir by microwave heating is approximate to that of the conventional method in 10- and 5-min irradiation, respectively, after the optimum heating time, Taiwania increases to 3.92% and China fir decreases to 8.10% at 20-min heating time. The difference of hot water solubility between the optimum time and 20-min microwave heating for test wood species depicts the difference in their intrinsic variations of the chemical compositions in the more dense hardwoods as compared with that of the softwoods. Additionally, by statistical analysis, the lower values of the RSD for each extraction also suggest that the method of microwave-assisted hot water solubility can likely be used as an alternative method to determine the hot water solubility of wood. Nonetheless, the standardization of the microwave-assisted method for determining the hot water solubility of wood needs further study.

CONCLUSIONS

From the results of the hot water solubility determinations, the microwave-assisted heating extraction method has proven to be effective in time-saving (15 min vs. 3 h). It indicates that the proposed microwave heating method has the potential to be an alternative to the conventional conductive heating method in the hot water solubility determination of

TABLE 1. *Comparison of microwave and conventional extraction methods for determining hot water solubility of four Taiwan wood species.*

Sample	Conventional[1]	Microwave heating time, min			
		5	10	15	20
	Hot water solubility (%)				
Liquidambar formosana Hance	6.07	5.63	5.89	6.04	5.78
	(1.45)[2]	*(1.16)*	*(1.50)*	*(1.15)*	*(1.49)*
Swietenia mahagoni Jacq.	2.30	1.99	2.19	2.30	2.20
	(3.75)	*(3.10)*	*(3.15)*	*(3.45)*	*(4.44)*
Taiwania cryptomerioides Hayata	3.70	3.59	3.70	3.71	3.92
	(2.25)	*(1.93)*	*(1.57)*	*(1.72)*	*(0.87)*
Cunninghamia lanceolata (Lamb) Hook	8.04	8.10	8.29	8.37	8.10
	(1.11)	*(1.23)*	*(0.79)*	*(0.82)*	*(1.05)*

[1] Conventional samples were heated in boiling water bath for 3 h.
[2] Numbers in ( ) are the relative standard deviations (%) for 5 extractions.

wood. The standardization of this new method needs to be further investigated.

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