Alkaline Extraction of Xylan from Wood Using Microwave and Conventional Heating

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ABSTRACT: This study aims to investigate the effect of microwave heating versus conventional heating for the alkaline hydrolysis of xylan from birch wood to understand the effect of the heating process on the dissolution of wood, the yield of xylan, and the degree of polymerization of the isolated xylan. The results indicate that the rate of wood dissolution is significantly higher (0.020/s) during microwave extraction than the conventional extraction (0.001/s). Wood solubilization, after an initial rapid removal of damaged fibers, is linear with time for both conventional and microwave extraction, with microwave showing a rate 20 times faster. The yield of xylan reaches a limit of about 60% for both processes but then declines slowly as thermal degradation become significant. Microwave heating provides 60% yield in 1/10th the time of the conventional process. This is found to be associated with the rapid temperature rise, and also with local "hot spots" generated during microwave treatment. The results indicated that xylan degradation was significant above 95°C. The nature of the isolated xylan was different for the two heating methods: the xylan isolated using microwave extraction for 20 min exhibits higher molecular weight (i.e., a greater degree of polymerization, about 150) than the xylan isolated using conventional extraction for the same duration (degree of polymerization, about 124) demonstrating the effectiveness of microwave heating for extraction of xylan from wood. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 41330.

INTRODUCTION

Hemicelluloses are the second versatile carbohydrate polymers after cellulose. Glucurono xylan is one of the major hemicelluloses present in woody biomass and is associated with cellulose and lignin through hydrogen bonding and ester and ether linkages, respectively.[1] An increased research interest in xylan isolation to develop an environmentally sustainable platform for fuel, chemicals, and materials has been noticed.[2–5] The challenge involved in the extraction of hemicelluloses is the recalcitrance of biomass because of the close association of the hemicelluloses with the other components of cell walls such as cellulose and lignin through physical and chemical bonds. Currently, the main methods used for extracting xylan from woody or non-woody lignocellulosic feed stocks include acid/alkali, or auto hydrolysis, or explosion techniques under conditions of high temperatures and/or high pressure.[3,6–9] These studies revealed that long reaction time and/or use of high pressure lead to a high energy consumption and unwanted degradation reactions producing undesirable degraded products.

Alkaline hydrolysis was reported to be advantageous over acid or auto hydrolysis or steam explosion to extract long chain hemicelluloses.[10,11] Alkaline hydrolysis at low temperature prefers to get high molecular weight hemicelluloses; however, low temperature requires extensively longer time for considerable amount of extraction. Sun et al.[12] reported that about 76% of arabinoxylan was extracted from wheat straw at room temperature by means of 1.5% NaOH solution for 144 h. On the other hand, high temperature alkaline hydrolysis leads to molecular breakdown on the polymer.[13] Conventional method of alkaline extraction was usually achieved by conductive heating from an external heat source, where the heat transfer is mainly through conduction, convection, and radiation. The slow heat transfer during conventional heating requires more time for the extraction of these polymers and the longer exposure at high temperature leads to the degradation of these polymers.

Microwave heating is known to be an efficient and environmental friendly alternative to conventional heating. The heating occurs through the direct interaction between the

Table I. Experimental Conditions

Method of extraction	Microwave (110 W)	Conventional (90°C)
Time, (min)	0.5, 1, 2, 4, 6, 8, 10, 12, 15, 18	5, 10, 30, 90, 120, 150, 180, 240

electromagnetic energy and the material of interest.[14] Advantages of using microwave energy as an alternative method to conventional heating include acceleration of reaction rates, because of the efficient internal heating produced by the direct coupling of microwave energy with the molecules present in the reaction mixture. Another advantage is the preferential absorption of energy by target compounds with high dielectric loss and high polarity in the reaction mixtures and acceleration of specific and selective reactions.[14,15] Over the past three decades, this technology has been widely used in analytical chemistry,[16] synthetic organic and polymer chemistry,[17–19] composite processing,[20] environmental engineering,[21,22] and wood liquefaction[23] applications.

Microwave heating for the pretreatment of lignocellulosic materials to improve the digestibility of these materials for the production of cellulosic ethanol has been undertaken by Ooshima et al.,[24] and Azuma et al.[25] Several more recent studies have been reported for the pretreatment of lignocellulosics to improve further saccharification of the biomass.[26–29] Hu and Wen[30] compared the microwave heating with the conventional heating at 190°C and found that sugar yield obtained after enzymatic hydrolysis of the residue after microwave heating was higher as compared to conventional heating. A similar observation was reported recently using a study for the comparison of the microwave heating with sand bath heating for the pretreatment of lignocelluosics at two different temperatures (140 and 180°C).[31] This technology has also been used for the isolation of hemicelluloses from agro-residues and forest residues using acid, alkali, or autohydrolysis.[32–35] Most of the reported methods were conducted at temperatures above 100°C and pressures above atmospheric pressure. These reaction conditions lead to degradation of the hemicelluloses and to the production of low molecular weight oligomers. The effects of these two heating processes on the extraction of hemicelluloses under mild conditions as well as on the molecular degradation of the isolated hemicelluloses have not been reported yet. In this study, we investigate the effect of microwave heating on the extraction of xylan from birch wood using alkali under mild extraction conditions and compare with conventional extraction. The research focused on two objectives: (i) comparison of the effect of microwave heating and conventional heating on the alkaline degradation of wood and the yield of xylan during the extraction of xylan; and (ii) study the effect of microwave heating on physico-chemical properties of xylan.

Two different approaches were used to accomplish the research objectives. In the first approach, characteristics of the low power input microwave assisted extraction (110 W) were compared with a conventional isothermal extraction at 90°C. The temperature selected for the conventional extraction was based on our previous studies.[23] Analysis of wood dissolution and yield of xylan obtained were compared to establish the effect of microwave heating over conventional heating for the alkaline extraction of xylan from wood. In the second approach, microwave extraction was performed for different durations and the final temperature of the slurry after each extraction was measured. Then conventional extractions were performed isothermally at each of these final temperatures obtained after microwave extraction and for the same duration as that of microwave extraction to differentiate the effect of temperature from the microwave effects. Comparison of wood dissolution and yield of xylan was again performed to see if there is any difference in the extraction process. Characterization of xylan obtained was performed to examine the effect of heating processes on the physico-chemical properties of xylan. The results indicated microwave extraction can efficiently isolate the xylan from birch wood without much molecular break down; whereas conventional extractions leads to molecular degradation of the isolated xylan.

MATERIALS AND METHODS

Materials
Wood fibers (Birch wood) prepared from two different sources (St. Mary's paper Company, ON, Canada, and the wood logs obtained from premises near to the University of Toronto) were used in this study. The preparation of the wood fibers used for extraction was reported elsewhere.[36] Four percentage reagent grade sodium hydroxide solution was used for extraction. $0.5M$ cupriethylenediamine from Fisher Scientific, Canada was used for the viscosity determination of xylan.

Methods
Extraction of Xylan from Birch Wood. In the first part of the study, 3 g of wood fiber (oven dry (OD) basis) were extracted using 30 mL of 4 wt % NaOH solution (solid to liquid ratio of 1:10 g:mL) using microwave and conventional methods of heating. Experimental conditions used are given in Table I. Microwave extraction was performed using a power input of 110 W for different durations, while conventional extraction was performed isothermally at 90°C. The details of the experimental procedures used for the extraction and separation of xylan have been reported elsewhere.[36] In brief, the wood slurry was subjected to microwave irradiation or conventional extraction for different durations. The treated slurry was filtered to separate the wood residue from the liquid phase. Xylan was precipitated from the liquid phase by neutralizing with acetic acid to a pH of 4.6 and the precipitated xylan was separated from the supernatant by centrifugation. The separated xylan was washed with water, ethanol and then freeze dried.

Time and temperature combinations of the microwave assisted and conventional extraction used in the second part of the study are given in Table II. In this study, 5 g of wood fiber (OD basis) was extracted using 50 mL of 4 wt % NaOH solution. A higher amount of material (volume of solution) was used in this study to prevent evaporation of the solution because of the longer duration of extraction. The temperatures selected for each conventional extraction performed was the final

Table II. Time-Temperature Combinations Used in the Microwave and Conventional Extractions

Time (min)/Temperature (°C)	
Microwave[a]	Conventional (isothermal)
1/37	1/37
5/60	5/60
10/80	10/80
20/95	20/95
30/98	30/98
40/100	40/100

[a] Final temperature of the slurry after extraction.

Figure 1. Percentage of wood solubilized and yield of xylan after microwave and conventional extraction; (M) microwave, (C) conventional heating at 90°C (wood sample size: 3 g). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

temperature of the slurry obtained after the same duration of microwave assisted extraction. Since conventional heating has a long thermal lag time, the NaOH solution and the wood fibers (previously weighed) were preheated to the required temperature before being mixed. The protocol used for the extraction, separation of wood fibers and precipitation of xylan was same as in the first part of the study.

Sugar Composition and Lignin Content. The constituent sugars and lignin content of the xylan extracted were determined using a two-step hydrolysis with 72% and then 4% sulfuric acid.[36,37]

Scanning Electron Microscopy. The wood fibers before and after alkaline extraction, was subjected to SEM (Hitachi S-2500, Tokyo, Japan) to study the changes in the microstructure during extraction. The air-dried fibers after extraction were mounted on metal stubs and were sputter coated with a thin layer of gold to avoid electrostatic charge during examination. Microscopic images were taken using an accelerating voltage of 15 kV and a working distance of 10 mm.

Viscosity Measurement. The extracted xylan was purified by redissolving in sodium hydroxide solution followed by reprecipitation by adding three volumes of ethanol. The precipitated xylan was then washed with ethanol and freeze-dried. Dried xylan was dissolved in 0.5M cupriehtylenediamine to prepare solutions of various concentrations (0.1–0.5 g/dL). Relative viscosity of the xylan solutions prepared was determined using a Cannon–Fenske viscometer at 25°C. Intrinsic viscosity ($\eta = (\eta_{sp}/C)_{c=0}$) was obtained by extrapolating the reduced vis-

cosity calculated using the eq. (1) to zero concentration by the least squares method.

$$\text{Reduced viscosity } (\eta_{sp}/C) = (t - t_0)/t_0 C \qquad (1)$$

where C is the concentration of the xylan solution (g/dL), t and t_0 are the times required for the solvent and solution to flow down the capillary tube of the viscometer, respectively.

RESULTS AND DISCUSSION

Comparison of Microwave and Conventional Alkaline Extraction: Wood Solubilization

During alkaline extraction of wood, alkali hydrolyses the intermolecular ester linkages between plant polysaccharides and lignin and leads to the dissolution of the hydrolyzed components (hemicelluloses and associated lignin) to the solution.[38–43] The major hemicellulose present in birch wood is xylan. The birch wood used in this study contained about 22% of xylan, 42% glucan, and 1.8% of mannan.[36] Since the major hemicellulose present in birch wood is xylan, the results are expressed in terms of xylan. The dissolved xylan was separated from the solubilized part of wood, by precipitation using acetic acid. The percentage of solubilized wood and the yield of precipitated xylan obtained after different duration of extraction under two different processes were calculated using the following equations:

$$\text{Solubilization of wood } (\%) = \frac{(wt. \text{ of OD wood used for extraction} - wt. \text{ of OD wood after xylan extraction})}{wt. \text{ of OD wood used for extraction}} \times 100 \qquad (2)$$

$$\text{Yield of xylan } (\%) = \frac{\text{Amount of xylan precipitated}}{\text{Amount of xylan in the wood used for extraction}} \times 100 \qquad (3)$$

Figure 1 shows the amount of wood solubilized and the yield of xylan calculated using the eqs. (2) and (3) for two different methods of extraction under the experimental conditions given

in Table I. The figure shows clearly that the time required to solubilize wood is much faster with microwave heating than conventional heating. This difference is attributed to the difference in the heating mechanism involved in the two processes. Microwave heating is volumetric and rapid as the microwave heats the target compound through the direct interaction of the objects with the applied electromagnetic field, whereas, in conventional heating, heat transfers from the heating device to the
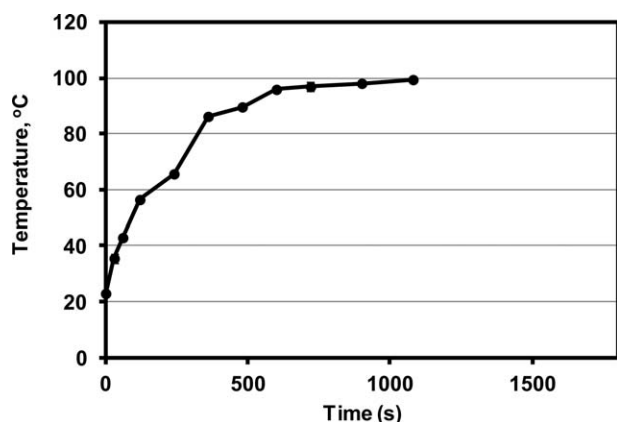
Figure 2. Time-temperature profile during microwave extraction.

sample in a rather slow and homogeneous manner through conduction and/or convention, which is based on inter- and intra-molecular heat transfer. The rapid temperature rise (Figure 2) in the system can accelerate the hydrolysis reaction and hence the faster dissolution during microwave assisted extraction.

For a detailed analysis of the effect of heating on the solubilization of wood during each extraction process, the graphs of wood solubilization were re-plotted as in Figure 3. From the figure, it is clear that solubilization of wood can be considered as two stages under the experimental conditions studied; where in
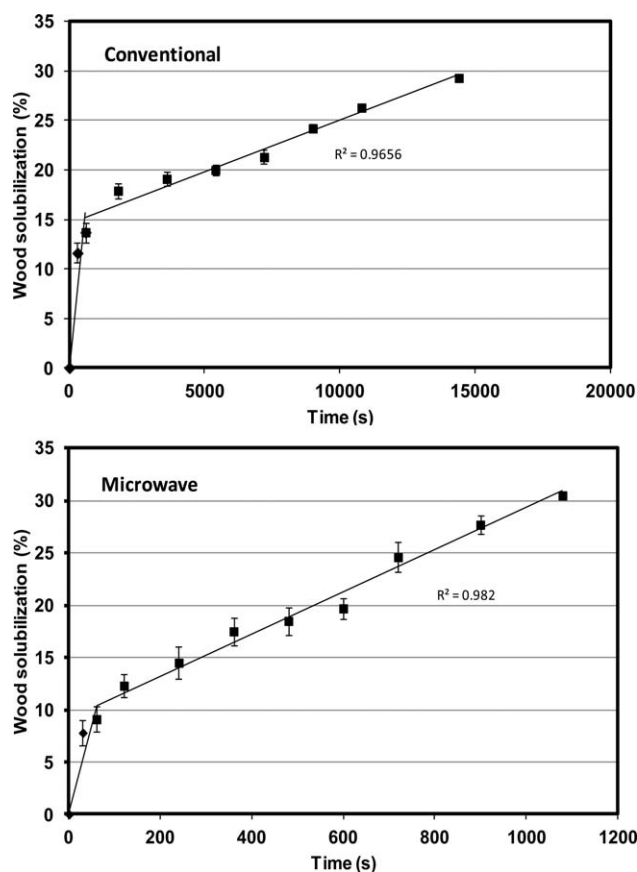

Figure 3. Comparison of the amount of wood solubilized during microwave and 90°C conventional extraction of 3 g of wood.

the first stage a fast dissolution occurs and is followed by a steady increase in the dissolution. The faster initial stage dissolution is believed to be due to the dissolution of the surface debris present on the fibers during the fiber preparation. The scanning electron microphotographs (Figure 4) demonstrated that the debris present on the fiber surface before extraction was removed after extraction and the fiber surface becomes smooth after extraction.

The slower second stage dissolution is believed to be the result of the various mass transfer processes during alkaline hydrolysis of the wood fibers. The major mass transfer processes during alkaline extraction include hydrolysis of hemicellulose-lignin linkages and their dissolution, diffusion of NaOH to the fibers to replace the alkali consumed by the hydrolysis, and diffusion of the hydrolyzed components (hemicelluloses and lignin) to the fiber surface and dissolution to the mass solution. The results pointed out that wood solubilization during both stages of dissolution using microwave assisted extraction are faster than the conventional extraction. The maximum amount of wood dissolved during the extractions is about 29–31%. The time required to obtain the maximum dissolution indicates that wood solubilization using microwave heating is 13 times faster than conventional heating. The slope of the linear portion of the graph after the initial dissolution provides the rate of dissolution of wood during hydrolysis. Rate of the dissolution, $(\Delta Y/\Delta t)_{\text{Microwave}} = 0.020 \gg (\Delta Y/\Delta t)_{\text{Conventional}} = 0.001$, where Y is the percentage of wood solubilized and t is the time in seconds. This indicate that rate of the wood solubilization during microwave assisted extraction is 20 times faster than the conventional extraction.

The slurry temperature increased from 56 to 100°C after the initial stage of wood dissolution during the microwave extraction (Figure 2). The conventional extraction performed is an isothermal extraction at a temperature of 90°C, which is closer to the temperature generated after 8–18 min of microwave extraction. Considerably higher rate of dissolution observed using microwave extraction can be attributed to the rapid increase in temperature of the fiber slurry, which increases the hydrolysis and the dissolution of the wood components. It is well known that increasing temperature will increase reaction rates; however, a 10° rise would not be expected to account for the significantly higher rate of wood dissolution (20 times) than conventional extraction. Earlier studies of chemical reactions using microwave energy reported that exceptional accelerations were observed in reaction rates and were thought to be due to the efficient internal heating produced by the direct coupling of microwave energy with the molecules present in the reaction mixture.[14] Lewis et al.[44] observed enhanced reaction kinetics during microwave assisted chemical reactions and the authors attributed this phenomenon to the nonuniform energy distribution at the molecular level during the chemical reactions and were reported to be the major microwave effects produced. An assumption is that microwave heating can induce "hot spots" in the biomass due to the selective interaction of the microwaves with the inhomogeneous lignocellulosic materials, and hence disrupt the structure of the materials. For example, Hu and Wen[30] found that microwave treatment enhances surface disruption and breaking of lignin structures in alkaline treated switch
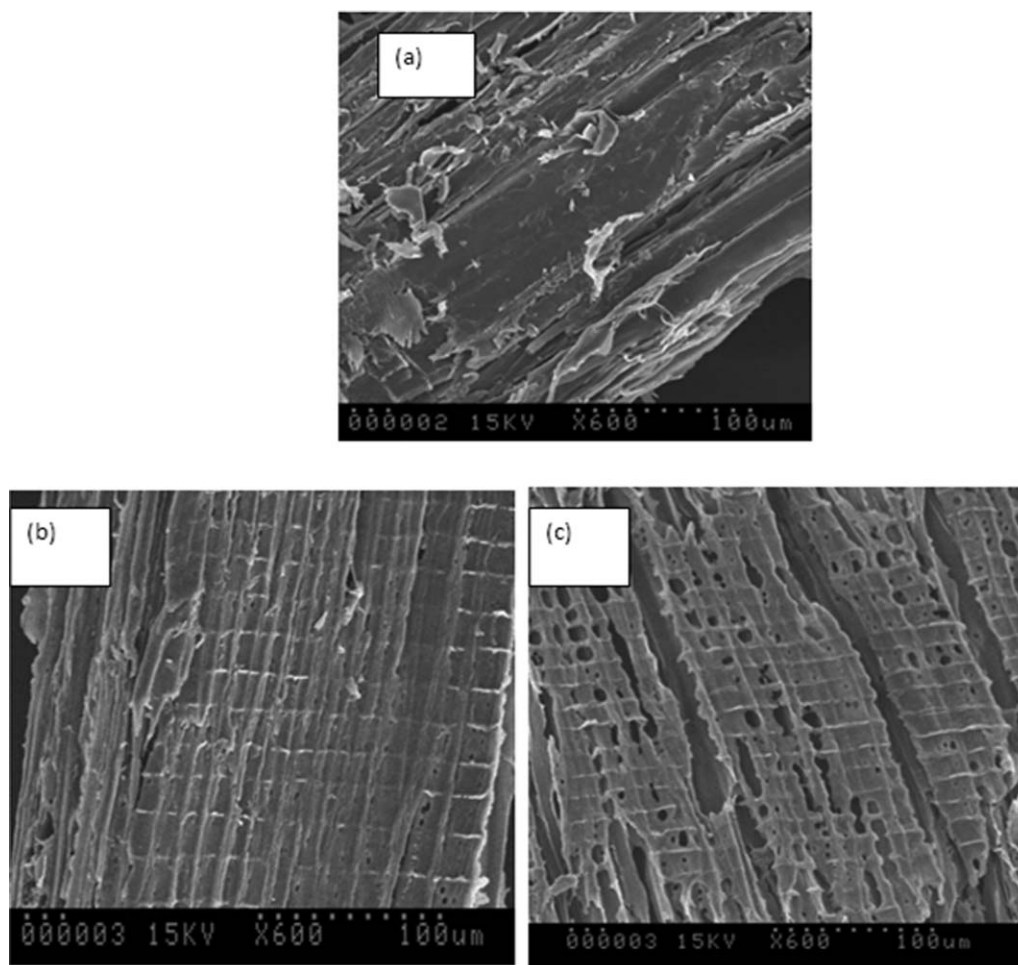
Figure 4. SEM photomicrographs of wood fiber before and after extraction (a) before extraction (b) after conventional extraction (90 min at 90°C), (c) after microwave extraction (10 min at a power level of 110 W).

grass, whereas in conventional heating, the fibers remain intact. These studies lead to the speculation that the significant enhancement observed in the rate of wood dissolution or reduction in the time of extraction could be the result from the combined effect of thermal and the "microwave effects" caused during the microwave extraction. It is hypothesized that the selective heating feature of microwaves leads to direct interaction of the microwaves with the more lossy component in the reaction system (alkali present in the fibers), raises the temperature inside the fibers and the inhomogeneous temperature distribution produces an "explosion" effect in the wood fibers, leading to the rupture of the fiber structure and facilitates the hydrolysis and diffusion of the hydrolyzed components to the solution. A comparison of SEM photomicrographs of the wood fibers before and after both extraction processes supports this hypothesis (Figure 4). After conventional extraction, the fiber surfaces are smooth and no significant change is noticed in the overall fiber structure [Figure 4(b)], whereas the fibers after microwave extraction showed a ruptured fiber structure [Figure 4(c)]. The more ruptured structure should lead to faster diffusion and dissolution of the hydrolyzed components and hence the difference in the rate of wood dissolution.

However, in order to accurately differentiate the two processes and to confirm the microwave synergic effect during extraction, it is essential to have almost identical experimental conditions for both processes. Therefore, another set of experiments was performed where conventional extraction was performed at different constant temperatures for the same duration as that of microwave extraction. In this part of the study microwave extractions were performed for different extraction duration (time of extraction was varied from 1 to 40 min) as given in Table II. The temperature of the slurry was noted after each set of experiments. Conventional experiments then were performed isothermally at each of these final temperatures of the slurry and for the same duration as that of microwave extraction. Further, to decrease the temperature gradient that occurs during the conventional heating of the wood slurry, and for faster attainment of the temperature, the NaOH solution was preheated to the required temperature, and the wood fibers were kept at 40°C.

The calculated wood solubilization using the eq. (2) during microwave and conventional alkaline extraction of wood fibers at different durations of extraction is shown in Figure 5.

The results indicate that the rate of wood dissolution is similar in both cases unlike the results of Figure 3. The microwave
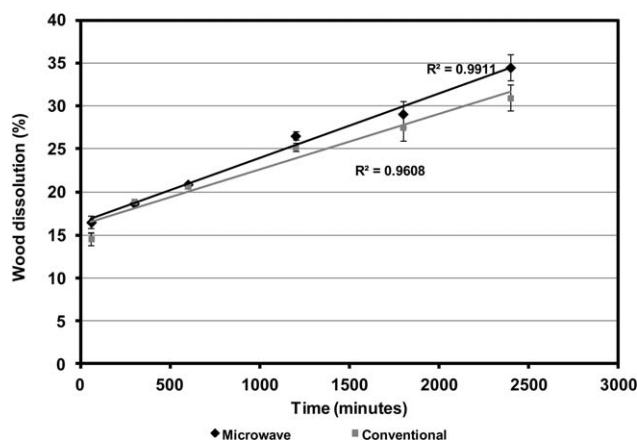
Figure 5. Effect of microwave and conventional extraction of xylan on the solubilization of 5 g of wood.

extraction is slightly slower than found in Figure 3 and is proportional to the sample size. The conventional extraction is much faster than found in Figure 3 and reflects the rapid attainment of the isothermal temperature. These results show the importance of temperature in the rate of extraction. However, the bulk temperature of the extraction is not the only important variable. By comparing the extraction rates for microwave and conventional, it is clear that the microwave extraction $[(\Delta Y/\Delta t)_{Microwave} = 75 \times 10^{-3}/s]$ is statistically faster than the conventional extraction $[(\Delta Y/\Delta t)_{Conventional} = 65 \times 10^{-3}/s]$. Under the conditions of Figure 5, the microwave extraction would have a lower average temperature than the isothermal temperature of the conventional extraction. Thus if temperature were the only factor in extraction, then the isothermal extraction should have been faster but was not found experimentally. This confirms the hypothesis that the synergic effect observed during microwave extraction is not only due to the temperature effect on the hydrolysis and wood solubilization, but also due to the physical changes caused by the "explosion" effect induced by the selective interaction of microwaves with the alkali present in the fibers. The physical changes of the wood after the extraction processes can be seen in SEM photomicrographs (Figure 6). The residual fiber morphology is quite different under the two processes. After conventional extraction, for all the time and temperature combinations, the fiber structure is smooth but with time becomes striated and thinner. After microwave extraction at all durations of extraction, the fiber structure is broken and more porous. This fiber damage and porosity likely explains the higher dissolution of the wood components. For short extraction times in Figure 5 there is no significant difference in the wood solubilization. These results support the contention that early stage wood dissolution is mainly due to the dissolution of the surface debris on the fiber.

Comparison of Microwave and Conventional Heating on the Yield of Xylan

The yield of precipitated xylan calculated for different duration of extraction under two different experimental conditions (Table I) are shown in Figure 1. It is clear from the figure that under these experimental conditions, time

required to extract a definite amount of xylan during microwave assisted extraction is significantly lower as compared to the conventional extraction. To understand how conventional and microwave extraction affects the yield of xylan, the graphs were replotted as a function of time and is shown in Figure 7. The figure shows that the yield obtained during both extraction types followed a similar path; there is a very fast initial (Stage 1), then a slow and linear increase (Stage 2) yielding a maximum of 60% xylan extraction, followed by a slow decrease in yield (Stage 3). It is clear from the Figure 7 that a portion of xylan (about 32%) can be extracted easily within a short duration of extraction and may correspond to the xylan dissolved from the initial dissolution of the debris present on the wood fibers. After this period of extraction, yield of xylan obtained increased with the duration of extraction to a maximum of 60% of xylan. The time required to obtain the maximum yield during microwave extraction was about 1/10th of the time required during the conventional extraction. The temperature of the wood slurry increased from 56°C to about 96°C during the extraction period. Thus the increased rate of xylan extraction in the microwave assisted process can be explained in terms of enhanced hydrolysis and diffusion of hydrolyzed components to the solution due to the faster heating of the microwave energy and the resulting damaged fiber structures.

Alkaline extraction, in addition to the hydrolysis and dissolution of the xylan from the plant cell wall, is known to cause degradation of xylan into low molecular weight oligomers, sugars, and other products. A decrease in the xylan yield was noted at about 720 s and 7200 s, respectively during microwave and conventional extraction (Figure 7) indicating that prolonged exposure to high temperature has an adverse effect on the yield of xylan. Longer duration of extraction at higher temperature may leads to the degradation of the carbohydrates into low molecular weight sugars and other products such as saccharinic acid. Figure 7 shows that the rate of degradation (determined from the slope of the third stage) was higher in the microwave extraction $((\Delta Y/\Delta t)_{Microwave, stage 3} = 0.005/s)$ as compared to conventional extraction $(\Delta Y/\Delta t)_{Conventional, stage 2} = 0.002/s)$. This difference is likely due to the higher temperatures (above 95°C) generated during microwave extraction as xylan degradation would be expected to be faster at higher temperatures. Degradation of xylan at high temperature was further confirmed by determining the degree of polymerization of the xylan obtained in the second part of this study.

The yield of xylan for microwave and isothermal conventional extraction (Table II conditions) were compared. The yield of xylan calculated for the microwave and conventional extraction is shown in Figure 8. Whereas the solubilization of wood was linear with time for both conventional and microwave treatments, the xylan yields for both processes followed a 1st order process for the first 20 min. For longer times the conventional isothermal extraction showed a more rapidly decreasing yield of xylan. In these experiments, the 98 and 100°C isothermal temperatures for 30 and 40 min extractions for the conventional extraction would have been at temperature longer than in the respective microwave extractions. Xylan degradation at high
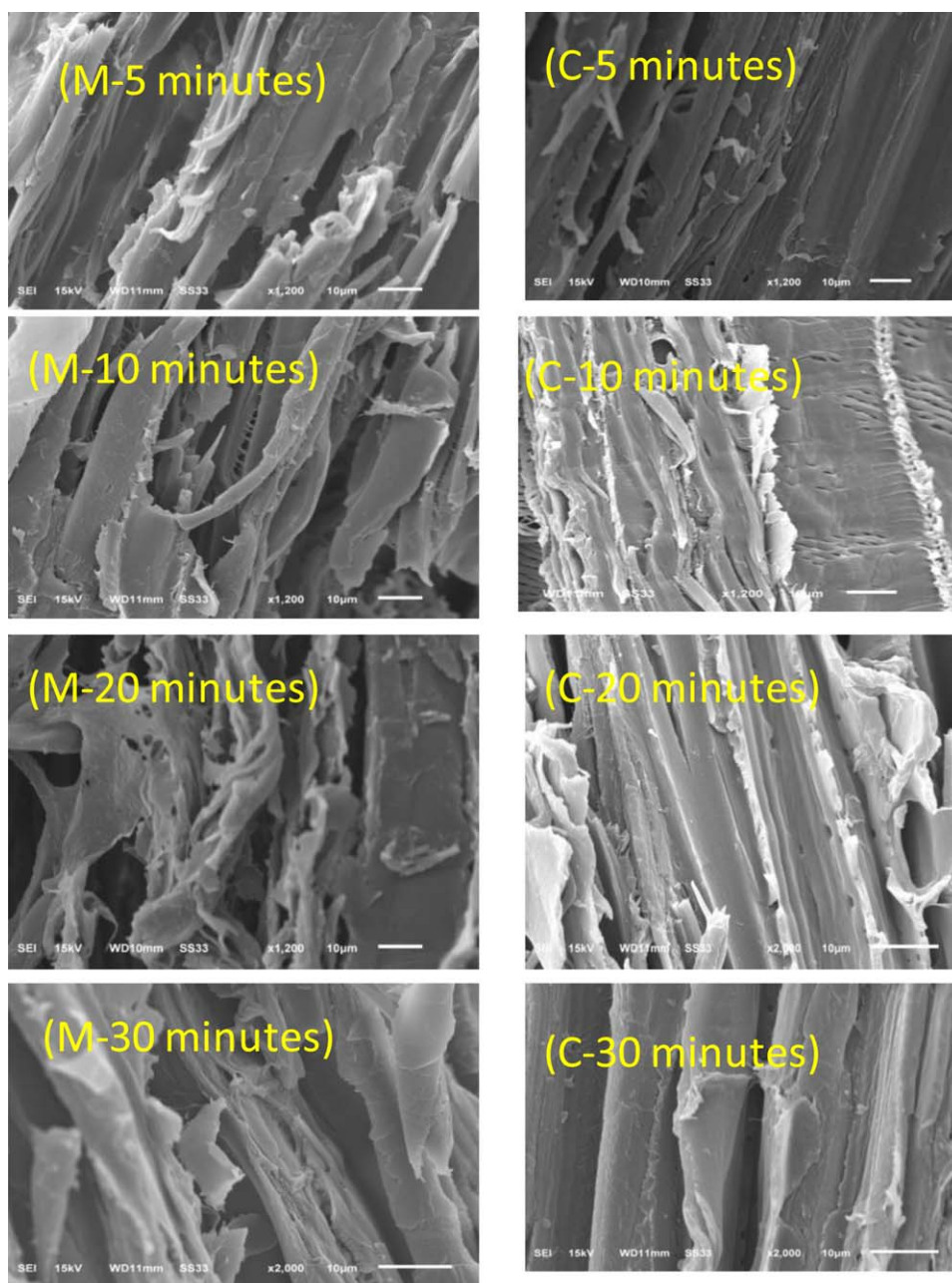
Figure 6. SEM photomicrographs of birch wood fibers after microwave and conventional extraction; M-microwave extraction and C-conventional extraction. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

temperature extractions will be an important factor to consider in optimizing yields.

Xylan Characterization

Chemical Composition of Xylan. The chemical composition of xylan isolated using microwave and conventional extraction under the conditions of Table II are given in Table III. Xylan obtained contained about 78–93% xylose, with minor amount of glucose (not shown), and 2–3.8% lignin, indicating xylan was a polymer with a xylopyranose backbone.

Viscosity and Degree of Polymerization. Viscosity measurements of xylan dissolved in $0.5M$ cupriethylenediamine (CED)

solution were used to determine the intrinsic viscosity of the solutions. The plots of viscosity data to determine the intrinsic viscosity of the xylan obtained using microwave and conventional extraction described in Table II are shown in Figure 9.

The intrinsic viscosity, $[\eta]$, is related to the molecuar weight of a polymer by the Mark–Houwink equation $[\eta] = K\, M_v^a$, where M_v is the viscosity average molecular weight and 'K' and 'a' are constants. The values of 'K' and 'a' depend on the polymer-solution interaction and the respective values for xylan in CED are 8.5×10^{-6} and 1.15.[45] Since number average degree of polymerization is more informative than the viscosity average molecular weight, conversion of intrinsic viscosity value into
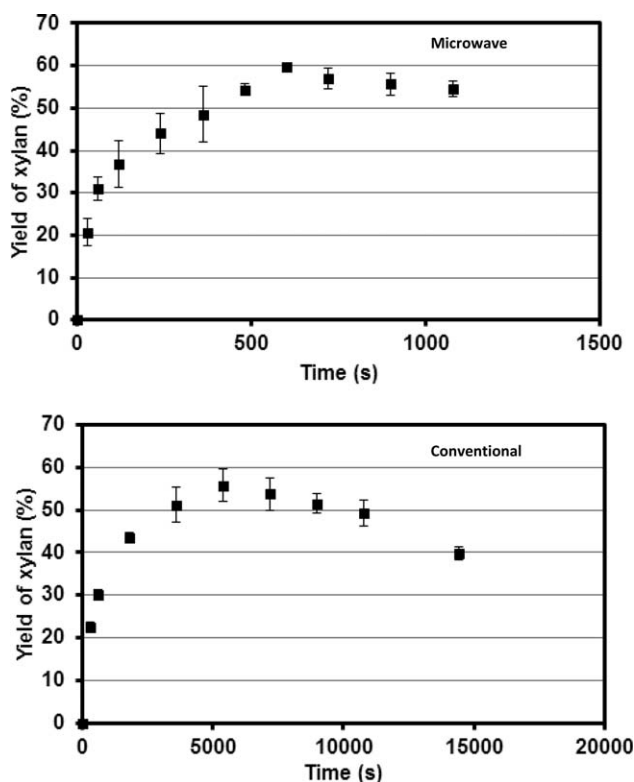
Figure 7. Comparison of the yield of xylan (based on the total xylan) during microwave and conventional extraction at 90°C (wood sample size: 3 g).

number average degree of polymerization was carried out according to the following equation $[\eta]_{CED} = DP_n \times 4.7 \times 10^{-3}$, where DP_n is the number average degree of polymerization.[45] The intrinsic viscosity of the xylan obtained for different extraction times using microwave and conventional extraction processes determined using the procedure described in the viscosity measurement section are shown in Figure 10, and the calculated viscosity average molecular weight, and number average degree of polymerization of xylan obatined are given in Table IV.
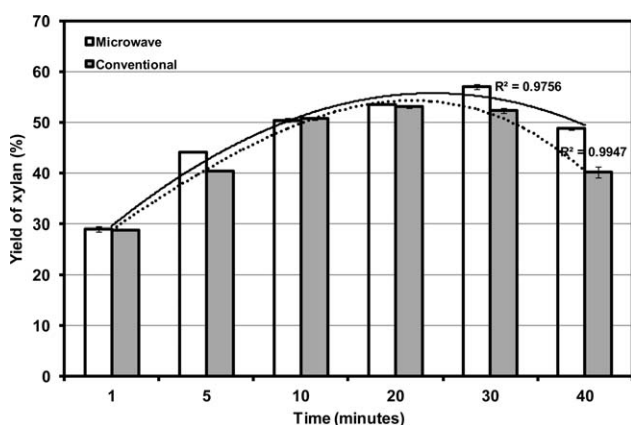

Figure 8. Effect of microwave and isothermal conventional extraction of xylan on the yield of xylan (wood sample size: 5 g).

Table III. Chemical Composition of Xylan Isolated Under Different Extraction Conditions

| Sample ID	Xylose	Lignin
XM[a]−5 min	78.9 ± 2.7	2.2 ± 0.3
XM-10 min	81.2 ± 1.9	2.1 ± 0.1
XM-20 min	92.1 ± 3.4	2.7 ± 0.5
XM-30 min	93.4 ± 1.2	3.6 ± 0.3
XC[a]−5min	74.5 ± 1.4	2.1 ± 0.6
XC-10 min	75.5 ± 1.5	2.7 ± 0.8
XC-20 min	87.6 ± 3.7	3.8 ± 0.5
XC-30 min	87.2 ± 1.7	3.7 ± 0.3

[a] XM-xylan obtained after microwave extraction; XC- xylan obtained after conventional extraction; Conditions given in Table II.

It is clear from Figure 10 that intrinsic viscosity of the xylan obtained during conventional extraction decreases with time whereas it remains more or less constant upto 20 min of microwave extraction and decreased afterwards. A similar trend was
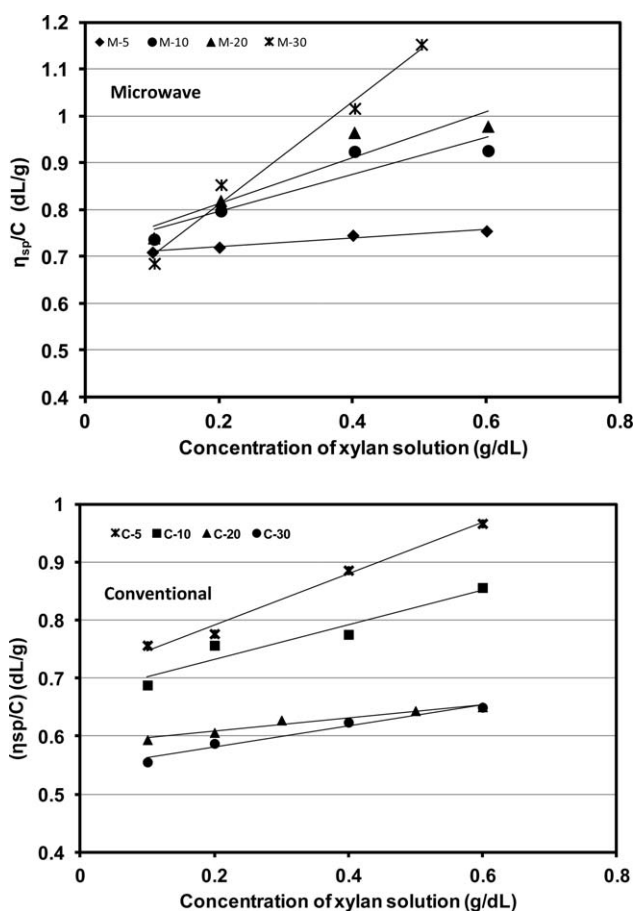
Figure 9. Plots for intrinsic viscosity determination of xylan (ηsp/C vs. Concentration of xylan) obtained using Table II conditions. (M and C represents xylan obtained using microwave and conventional extraction, respectively; 5, 10, 20, and 30 represents the duration of time in each process).
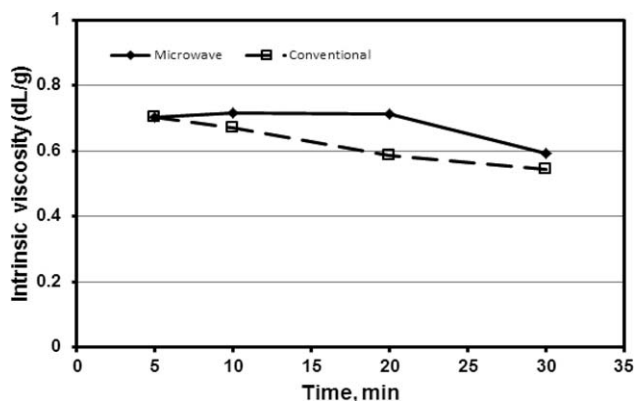
Figure 10. Intrinsic viscosity of xylan solutions in CED (xylan obtained using Table II conditions).

observed in the molecular weight and degree of polymerization of the xylan (Table IV).

Viscosity average molecular weight of the xylan obtained is in the range of 18000–19000, which corresponds to a degree of polymerization of about 150. Such a degree of polymerization of birch xylan was reported earlier by Lebel et al.[46] The degree of polymerization of xylan obtained using conventional extraction decreased with time from 149 to 115, whereas it remained almost constant during 20 min of microwave extraction before decreasing to 126. It is important to note that the decrease in the intrisic viscosity as well as degree of polymerization was significant when the temperature of the system is at (as in conventional) or above 95°C (as in microwave extraction). These results support the contention that tempertures above 95°C enhance the degrdation of xylan in alkali. The results also show that xylan underoges significant degradation during conventional extraction as it takes longer for an equivalent extraction. On the otherhand, microwave assisted extraction can provide xylan with high degree of polymerization since the extraction needs considerably less time, which reduces degradation of the extracted xylan.

Table IV. Intrinsic Viscosity, Viscosity Average Molecular Weight, and Number Average Degree of Polymerization of Xylan Obtained by Microwave Conventional Extraction

Sample ID	Temperature (°C)	Viscosity average molecular weight	Number average degree of polymerization
XM[a]−5 min	60	18,859	149
XM- 10 min	80	19,198	152
XM- 20 min	95	19,160	152
XM- 30 min	98	16,271	126
XC[a]−5min	60	18,899	149
XC-10 min	80	18,156	142
XC-20 min	95	16,116	124
XC-30 min	98	15,125	115

[a] XM-xylan obtained after microwave extraction; XC-xylan obtained after conventional extraction; conditions as in Table II.

Factors to be Considered for Future Development. The results from this study indicate that a low power input microwave heating can provide a faster dissolution and high xylan yield as compared to the conventional heating because of the rapid heating process. Efficiency of the extraction process is highly dependent on the process conditions selected. Power input and temperature are interrelated as higher power input can bring up the temperature very fast and considerably reduces the heating time required for extraction. Hence, it is expected that a higher input microwave extraction leads to a higher wood dissolution and xylan yield. However, our earlier study using different power input microwave extraction indicated that higher power input has a negative impact on the yield of xylan and the maximum yield was obtained at the lower power input. Further, microwave power is directly related to the quantity of sample and the extraction time required. Hence, effect of factors such as quantity of the sample, sample size (surface area), and solid to liquid ratio on the extraction have to be investigated for providing a better understanding of the processing conditions to be selected for future development. From the degree of polymerization of xylan obtained it was clear that the xylan polymer undergoes degradation above 95°C. So, a consecutive extractions using lower power input can be a preferable alternative for a better yield of xylan than using higher input microwave extraction.

CONCLUSIONS

Two different studies were performed to compare the effect of microwave heating and conventional heating on the alkaline extraction of xylan from birch wood and to understand the mechanism involved in the processes. The effect of two different heating processes on the degree of polymerization of the isolated xylan was also studied. The following conclusions were drawn from the study.

a. Wood dissolution was significantly faster during microwave extraction than conventional extraction.
b. The time required to get the maximum yield of xylan was significantly reduced using microwave extraction.
c. The results on the dissolution of wood during microwave and conventional extraction confirmed the enhanced dissolution caused by the microwave effect.
d. Degree of polymerization of xylan obtained using microwave extraction was higher as compared to conventional extraction.
e. Degradation of xylan polymer was found to be significant at temperatures at or above 95°C.

The overall conclusion was that microwave heating can be used as an alternative to conventional heating for the extraction of xylan as the xylan can be isolated using 1/10th of the time required by conventional extraction without degrading the xylan. However, more studies are needed for the future development and industrial use of the microwaves for the extraction of hemicelluloses from the biomass.

ACKNOWLEDGMENTS

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