The Chemical and Physical Properties of Steam-Exploded Wood at Different Temperatures and Times at the Same Severity as a Dietary Fiber Source

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The chemical and physical properties of steam-exploded pine chips were analyzed by varying the temperature (190 °C to 230 °C) and time (1.5 min to 20.0 min) of steam explosion at the same severity factor (R_o), *i.e.*, 4.0. Then, their potential as a dietary fiber source was evaluated. Overall, the chemical properties, *e.g.*, solid recovery, cellulose, hemicellulose, and total dietary fiber content, showed a tendency to decrease as the temperature increased as well as an increasing lignin content. The physical properties, *e.g.*, water-holding capacity, oil-holding capacity, and swelling capacity, showed decreased values in pine chips subjected to steam explosion at a temperature of 210 °C or greater. The chemical and physical properties of the steam-exploded pine chips, which were subjected to steam explosion at different temperatures and times at the selected R_o) considerably changed, starting at a temperature of 200 °C for 11.5 min. Even with the same severity factor, the steam-exploded pine chips at a temperature of 200 °C for 11.5 min showed the greatest improvement in the physical properties, *i.e.*, water holding capacity (8.3 g/g), oil holding capacity (6.8 g/g), and swelling capacity (4.9 mL/g).

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Keywords: Pinus densiflora; Severity factor; Steam-explosion; Insoluble fiber; Physicochemical properties

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INTRODUCTION

Dietary fiber has historically been regarded as an anti-nutritional factor because of its negative impacts on nutrient utilization (Annison 1993; Jha *et al.* 2010). However, dietary fiber has recently gained special attention because of its functional value in improving the gut health of monogastric animals (Jha and Berrocoso 2015). Maintaining or improving gut health is essential to enhance feed efficiency, promote growth performance, and maintain the overall health of monogastric animals. Dietary fiber is subdivided into insoluble and soluble dietary fibers, depending on their solubility in water. Insoluble dietary fiber has poor hydrolysis performance, and it usually manifests with high resistance to chemicals and enzymes. Compared with insoluble dietary fiber, soluble dietary fiber generally has better functional properties, *e.g.*, fermentability, solubility, and water/oil holding capacity (Chen *et al.* 2019; Moczkowska *et al.* 2019).

Recently, feeding experiments have been primarily carried out with insoluble fiber sources that arise as by-products during industrial production, *e.g.*, oat hulls, sunflower hulls, soybean hulls, wheat bran, or wood shavings. In this context, the effect of lignocellulose as an insoluble dietary fiber source is increasingly being investigated. Water-insoluble wood is a potential cheap source of dietary fiber. One of the cheapest means of dietary fiber administration is the addition of processed fibers to regular foods (Abdulrahaman *et al.* 2013). Lignocellulose is a component of plant cell walls and consists primarily of insoluble carbohydrate polymers cellulose and hemicellulose, as well as phenolic polymer lignins. Lignocellulose is chemically and physicochemically different from other insoluble fiber sources and thus may have different effects on poultry compared to traditional fiber sources.

Softwood, *e.g.*, pine wood, contains large amounts of cellulose, hemicellulose, and lignins. In addition, pine wood is one of the major plantation tree species in the Republic of Korea (Jung *et al.* 2019a; Min *et al.* 2019; Lee *et al.* 2020). In general, softwood has a higher cellulose content, higher lignin content, and lower pentosan content compared to hardwoods (Sjöström and Alén 1999). Major dietary fiber fractions, *e.g.*, cellulose, hemicelluloses, and lignins, are insoluble and have beneficial effects on the human intestine (Mudgil and Barak 2013). All the components (cellulose, lignin, hemicellulose, pectins, gums, and mucilages) of dietary fiber are the major constituents of plant cell walls (Selvendran 1984; Selvendran and MacDougall 1995). However, the nutritional value and functional characteristics of dietary fiber were determined by their chemical composition and structure. Research has shown that dietary fiber composition, source, and preparation method could affect the effectiveness of its adsorption characteristics, *e.g.*, water holding capacity, swelling power, and binding capacities for oil and cholesterol, to a large extent *in vitro* (Galisteo *et al.* 2008).

Steam explosion can degrade cellulose and hemicellulose to increase the content of soluble polysaccharides by breaking the glycosidic and hydrogen bonds in the fiber (Wang *et al.* 2015; Wang *et al.* 2019; Overend *et al.* 1987; Muzamal *et al.* 2015). Steam explosion can affect the composition and structure of dietary fibers, leading to changes in the functional and enzymatic hydrolysis properties (Gan *et al.* 2021). While softwood was formerly considered unsuitable for steam explosion, studies have been conducted to separate the components of softwood *via* steam explosion (Saddler *et al.* 1993).

The severity factor is a measure of the pretreatment intensity in hydrothermal processes, and it includes the combined effect of temperature and reaction time. It can be calculated using the expression proposed by Overend *et al.* (1987). The log value of the reaction ordinate gives the severity factor used to map the effects of steam explosion pretreatment on biomass. The severity may be the same, but the temperature and time may be different. Softwood is composed of very complex celluloses, hemicelluloses, and lignins, with different dissolution temperatures. Therefore, even with the same severity, the chemical and physical properties of softwood may vary depending on the temperature difference. As such, the authors did not find any research that studied the differences in physicochemical properties with temperature and time at the same severity in previous studies.

The previous studies of the authors have reported that steam explosion (severity factor (R_o) = 3.94) increases the physical properties, *i.e.*, water-holding capacity, oil-holding capacity, and swelling capacity, of hardwood chips to different extents (Jung *et al.* 2019b). In addition, a diet containing 1.0% steam-exploded hardwood chip promoted broiler growth performance (body weight: 858.9 g), improved blood characteristics (130.0

mg/dL), intestinal morphology (V to C ratio: 7.50), and organ weights (length of intestine: 17.6 cm/100 g body weight). In this study, the effects of the same severity factor values, but different temperatures and times, on the chemical composition and physical properties of pine chips were investigated.

EXPERIMENTAL

Materials

Pine (*Pinus densiflora*) was collected from the experimental forest of Gyeongsang National University, Jinju, South Korea. The pine was chipped to a particle size of approximately 2 cm × 2 cm × 0.5 cm for steam explosion and stored at a temperature of 20 °C at a moisture level below 10% to 15%.

Steam Explosion

The pine chips were then steam-exploded in the reactor of the steam explosion apparatus with a maximum operating pressure of 30 kg/cm^2 (as shown in Fig. 1).

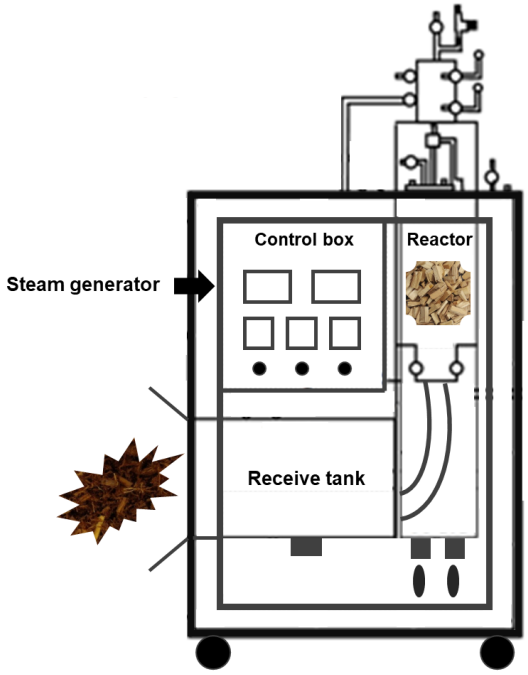

Fig. 1. Steam explosion apparatus of lab scale.

Steam temperatures of 190 to 230 °C and treatment times of 1.5 to 20.0 min were used to achieve the following value for the severity factor (R_o), *i.e.*, 4.0. Each experimental condition is represented by a value of R_o, which unifies time and temperature in a single factor and thus allows the comparison of different treatment conditions (Overend *et al.* 1987). The R_o value was calculated using Eq. 1,

$$R_o = \log[t \times \exp(T - 100/14.75)] \quad (1)$$

where t is the residence time (min) and T is the reaction temperature (°C). The pine chips were treated according to the conditions listed in Table 1. After the explosion, the material

was recovered in a cyclone. The wet material was cooled to a temperature of approximately 40 °C and filtered to recover the solids. The solid fraction was analyzed using the procedures described below.

Table 1. Steam-explosion Conditions

Severity Factor (R_o)	Temperature, T (°C)	Time, t (min)
4.0	190	20.0
4.0	200	11.5
4.0	210	6.0
4.0	220	3.0
4.0	230	1.5

Determination of the Chemical Properties of the Steam Exploded Pine Chips

Solid recovery

The solid recovery was calculated using Eq. 2,

$$\text{Solid recovery (g/g)} = W_1/W \times 100 \quad (2)$$

where W_1 is the weight after steam explosion and drying (g), and W is the weight of sample (g).

Cellulose, hemicellulose and lignin content

The carbohydrate content was determined based on the total monomer content measured after a two-step acid hydrolysis procedure to fractionate the fiber. The first step involved exposure to 72% H_2SO_4 at 30 °C for 60 min. In the second step, the reaction mixture was diluted to a final H_2SO_4 concentration of 4% and subsequently autoclaved at 121 °C for 1 h. The solid residue remaining after acid hydrolysis was considered to be the total lignin content. After hydrolysis with sulfuric acid and conversion into alditol acetates (ASTM method E1821-96), the monomeric sugar (arabinose, xylose, mannose, galactose, and glucose) content of this hydrolyzed liquid was analyzed by gas chromatography (GC) in a YL6100 device (Young Lin Ins. Co., Ltd., Korea).

Dietary fiber content

Dietary fiber content was determined using an established method (AOAC 2000). Briefly, the samples were treated with thermo-stable α-amylase and subsequently digested with a protease, followed by incubation with amyloglucosidase to remove starch and protein components. Following enzymatic digestion of starch and protein, the insoluble dietary fiber was separated by centrifugation at 1,000 × g for 15 min, and soluble dietary fiber was precipitated with 95% ethanol. Dietary fiber content was calculated as the sum of the insoluble and soluble dietary fibers.

Determination of Physical Properties of Steam Exploded Pine Chips

Water holding capacity

The water-holding capacity (WHC) was determined according to the method described by Chau and Huang (2003) with minor modifications. First, 1 g of the sample was mixed with 10 mL of distilled water at room temperature (25 °C) for 24 h. After centrifugation at 4000 rpm for 30 min, the supernatant was removed, and the sediment was weighed using a centrifuge tube. The WHC was calculated according to Eq. 3,

$$\text{Water holding capacity (g/g)} = W_1/W \quad (3)$$

where W_1 is the weight of the sediment minus the sample (g), and W is the weight of the sample (g).

Oil-holding capacity

The oil holding capacity (OHC) was determined according to the method described by Chau and Huang (2003) with minor modifications. First, 1.0 g of the sample was mixed with 5 mL of soybean oil at a temperature of 25 °C for 30 min and shaken once every 30 min. Then, the mixture was centrifuged at 4000 rpm for 30 min. The upper free oil was removed, and the residue was weighed using a centrifuge tube. The OHC was calculated according to Eq. 4,

$$\text{Oil holding capacity (g/g)} = W_1/W \quad (4)$$

where W_1 is the weight of the residue minus the sample (g), and W is the weight of the sample (g).

Swelling capacity

Swelling capacity is defined as the volume of a sample after immersion and soaking in water (Ralet *et al*. 1993). First, 1.0 g of the sample was placed in a test tube, 10 mL of water was added, and it was hydrated for 18 h at a temperature of 25 °C. The volume of each sample was then recorded. The swelling capacity was calculated according to Eq. 5,

$$\text{Swelling capacity (mL/g)} = V/W \quad (5)$$

where V is the final volume occupied by the sample (mL), and W is the weight of the sample (g).

Statistical Analysis

All experiments were performed in triplicates. Data were analyzed using SAS statistical software, and Duncan's multiple range test was used to compare treatment means when p-values were significant ($p < 0.05$).

RESULTS AND DISCUSSION

Chemical Properties of the Steam Exploded Pine Chip by Varying the Temperature and Time at the Same Severity

Solids recovery

The previous studies by the authors reported that steam-explosion treatment (at a severity factor (R_o) = 3.94) could effectively improve the physicochemical properties and digestive functionality of hardwood. Based on the previous results, the severity factor was

set to 4.0, the temperature was 190 to 230 °C, and the time was 1.5 min to 20.0 min. Figure 2 shows the images of the pine chips after different steam explosion conditions at the same severity factor (R_o) of 4.0. While it was at the same severity factor value, it is clear that the sizes of pine chips tended to be smaller at higher steam temperatures. In addition, more fibers were found when the samples were treated at a higher temperature.

Fig. 2. Images of the pine chips obtained from different steam explosion conditions at the same severity factor (*R*o) of 4.0: (a) 190 °C, 20.0 min; (b) 200 °C, 11.5 min; (c) 210 °C, 6.0 min; and (d) 220 °C, 3.0 min; (e) 230 ℃, 1.5 min

The solids recovery of the solid fraction after steam explosion at different temperatures and times is shown in Fig. 3. The change in yield of the solids was not significant when the temperature and time were varied. The solid recovery yields ranged from 92.4% to 96.4% depending on the steam explosion conditions at a severity factor (R_o) of 4.0. The average fiber recovery at R_o equal to 4.0 was 94.8%. Solids losses occur during steam explosion because of the deposition of the fibers on the walls of the container, as well as in the connecting piping between the reactor vessel and the container. Losses also occurred through the escape of volatiles with the steam and through the degradation of sugars into furfural and 5-hydroxymethyl furfural, both of which are volatile compounds (Huynh and Phan 2011).

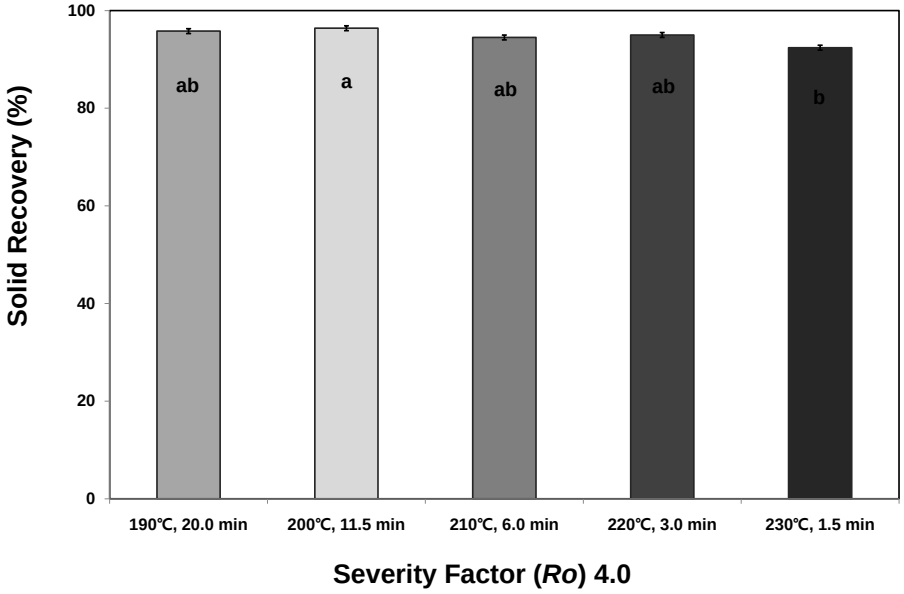

Fig. 3. Solid recovery of the steam-exploded pine chips obtained from different steam explosion conditions at a R_o = 4.0. The data are expressed as the mean ± SD (n = 3). The statistical significance of the results was assessed by Duncan's t-test (p ≤0.05).

Cellulose and hemicellulose content

Cellulose is straight chains are bound closely together by multiple intermolecular hydrogen bonds, producing a water-insoluble fibrous substance that is relatively inert (Cummings 1984). Hemicellulose also describes a heterogeneous group of chemical structures, and hemicelluloses may exhibit a water-soluble and insoluble nature (Mudgil 2017).

The cellulose and hemicellulose content of the steam-exploded pine chips obtained from different steam explosion conditions at an R_o value of 4.0 are shown in Fig. 4.

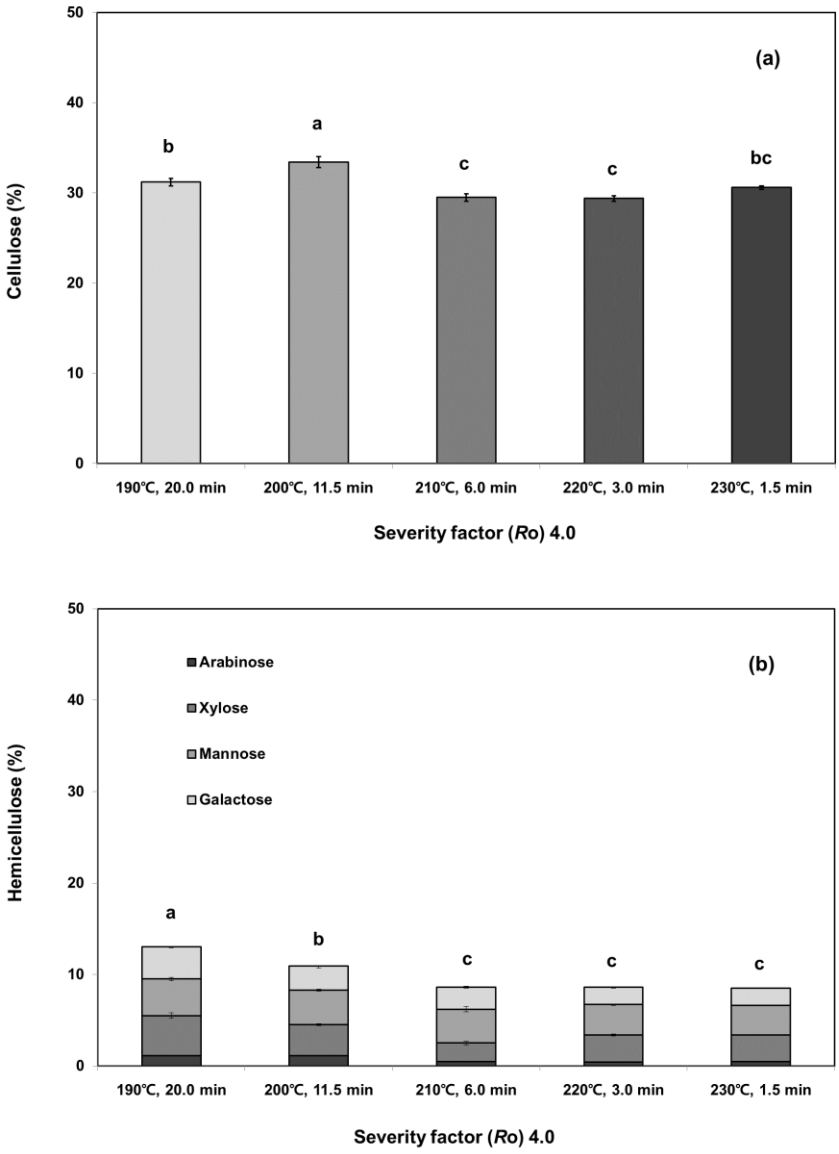

Fig. 4. The cellulose and hemicellulose content of the steam-exploded pine chips obtained from different steam explosion conditions at a severity factor (R_o) of 4.0: (a) cellulose content; and (b) hemicellulose content. The data are expressed as the mean ± SD (n = 3). The statistical significance of the results was assessed by Duncan's t-test ($p \leq 0.05$).

The cellulose and hemicellulose content showed slight differences according to temperature and time, even with the same severity factor. Depending on the steam

explosion conditions, the cellulose content ranged from 29.4% to 33.4% (as shown in Fig. 3a), and there was no significant change in the cellulose content according to the steam explosion conditions. Cellulose degradation occurs between temperatures of 240 and 350 °C, and the crystalline structure resists thermal depolymerization better than unstructured hemicelluloses (Mohan *et al.* 2006). In this study, since the pine chips were subjected to steam explosion at a temperature of 190 to 230 °C, it was found that there was no significant effect on the change in the cellulose content.

The hemicellulose content was expressed as the sum of arabinose, xylose, mannose, and galactose, and Fig. 4b shows the hemicellulose content according to the steam explosion conditions. The hemicellulose content ranged between 8.5% and 13.0%. The hemicellulose content showed a tendency to decrease in the steam exploded pine chips at a temperature of 200 °C or greater, and the decrease in arabinose was the largest among the components of hemicellulose, *i.e.*, arabinose, xylose, mannose, and galactose. Tumuluru *et al.* (2011) reported that thermal degradation of hemicellulose initiates at 150 °C, with the majority of weight loss occurring above 200 °C, depending on the chemical nature of the hemicellulose and the relationship with lignin within the cell.

Lignin content

Lignin is relatively hydrophobic, aromatic, and considered an insoluble fiber. In addition, lignins may reduce fat absorption, reduce bile salt pool size, increase cholesterol turnover, and reduce the formation of carcinogenic metabolites from bile salts (Pomare and Heaton 1973; Eastwood 1975; Drasar and Jenkins 1976; Eastwood and Mowbray 1976).

The lignin content of the steam-exploded pine chips obtained from different steam explosion conditions at an R_o value of 4.0 are shown in Fig. 5.

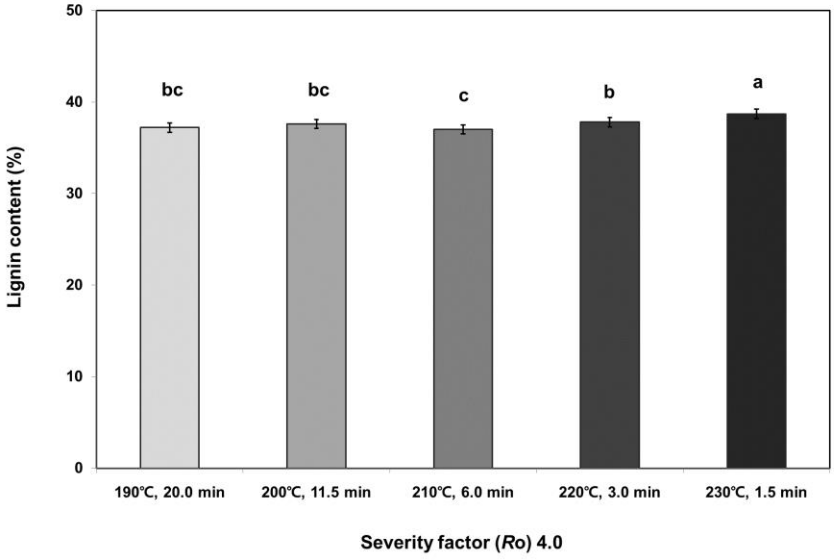

Fig. 5. The lignin content of the steam-exploded pine chips obtained from different steam explosion conditions at a severity factor (R_o) of 4.0. The data are expressed as the mean ± SD (n = 3). The statistical significance of the results was assessed by Duncan's t-test ($p \leq 0.05$).

The lignin content ranged from 37.0% to 38.7%, and as the steam explosion temperature increased and the time decreased, the lignin content showed a tendency to slightly increase. Lignins are known to decompose when heated to a temperature of 280 to

500 °C. It is removed only to a limited extent during the steam explosion but is redistributed on the fiber surfaces as a result of melting and depolymerization-repolymerization reactions (Li *et al.* 2019). In this study, steam explosion was carried out at a temperature of 190 to 230 °C, and the increase in the lignin content was considered to be relative to the decrease in carbohydrates.

Total dietary fiber

The total dietary fiber, soluble dietary fiber, and insoluble dietary fiber content of steam-exploded pine chips obtained from different steam explosion conditions at an R_o value of 4.0 are shown in Fig. 6. The total dietary fiber content ranged from 81.2% to 84.7%, and the highest total dietary fiber content was shown in the pine chips subjected to to steam explosion at a temperature of 200 °C for 11.5 min. The insoluble dietary fiber content of the steam exploded (at an R_o value of 4.0) pine chip was 80.6% to 83.9%, most of which was insoluble dietary fiber, with only 0.6% to 0.8% being soluble dietary fiber.

Insoluble fiber, *i.e.*, cellulose, hemicellulose, and lignins, is thought to be inert in the sense that it does not interfere with nutrient absorption, but it may accumulate in the gizzard, increasing the retention time of smaller particles and the digestibility of starches, fats, and crude protein (Mateos *et al.* 2012).

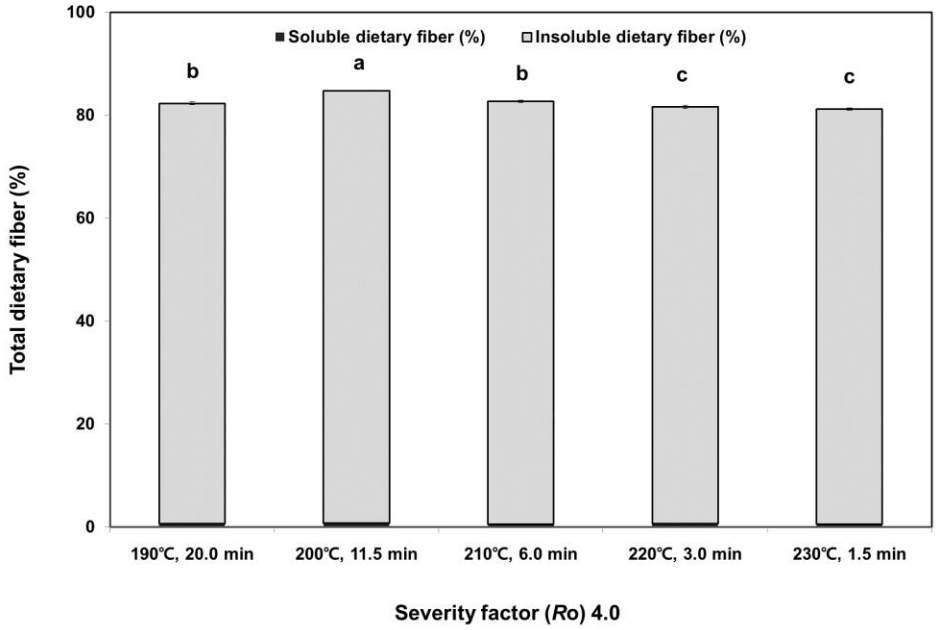

Fig. 6. The total dietary fiber of the steam-exploded pine chips obtained from different steam explosion conditions at a severity factor (R_o) of 4.0. The data are expressed as the mean ± SD (n = 3). The statistical significance of the results was assessed by Duncan's t-test ($p \leq 0.05$).

Physical Properties of the Steam Exploded Pine Chip by Varying the Temperature and Time at the Same Severity

The physical properties of insoluble fiber investigated in this study included the water holding capacity, oil holding capacity, and swelling capacity. Figure 7 shows water holding capacity (a), oil holding capacity (b), and swelling capacity (c) of the pine chips after different steam explosion conditions at the same severity factor of 4.0.

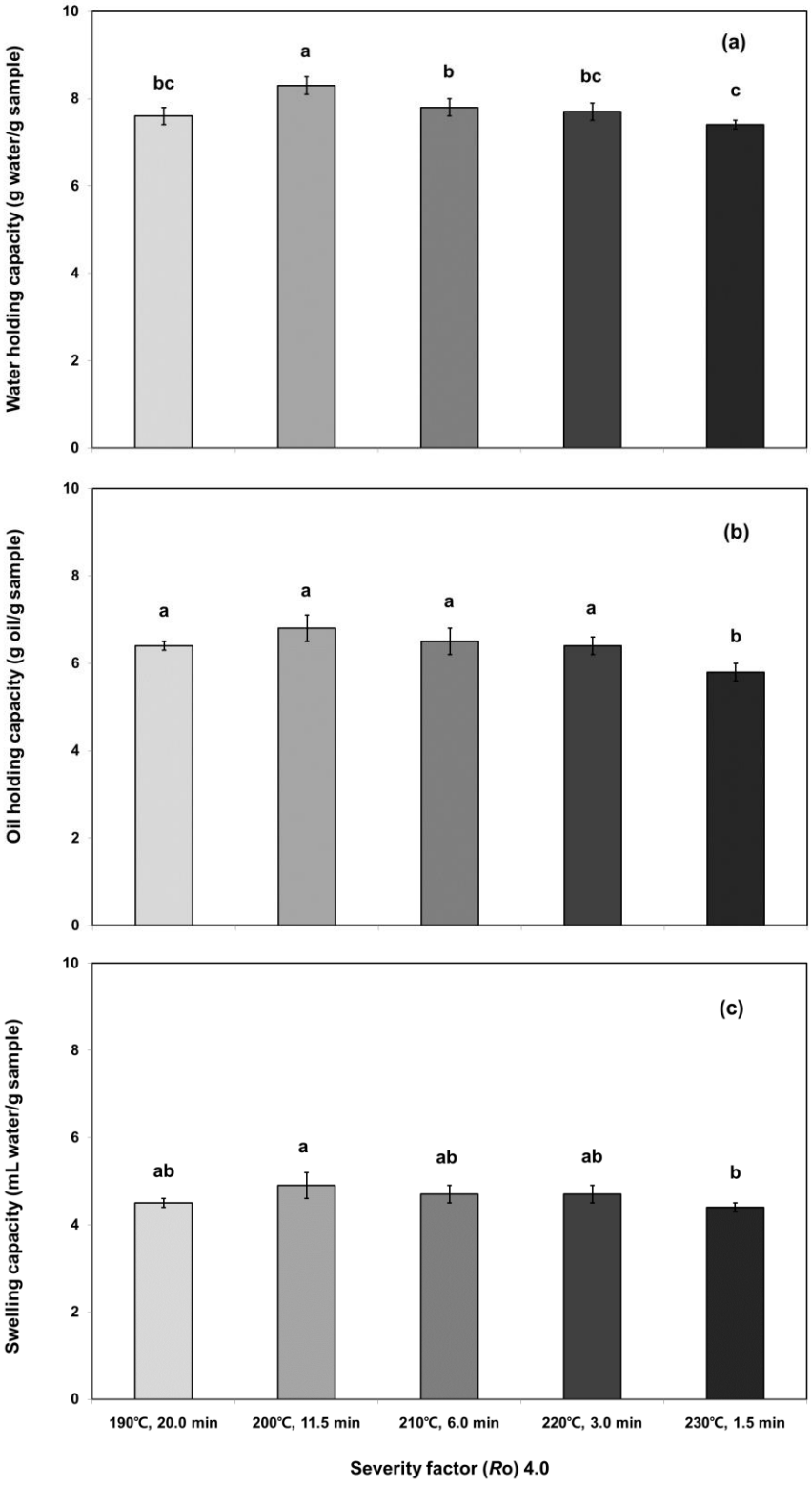

Fig. 7. Water holding capacity, oil holding capacity and swelling capacity of the steam-exploded pine chips obtained from different steam explosion conditions at a severity factor (R_o) of 4.0. The data are expressed as the mean ± SD (n = 3). The statistical significance of the results was assessed by Duncan's t-test ($p \leq 0.05$).

The water holding capacity ranged from 7.6 to 8.3 g/g, the oil holding capacity ranged from 5.8 to 6.8 g/g, and the swelling capacity ranged from 4.4 to 4.9 mL/g. When steam explosion was performed at different temperatures and times at the same severity, there was a significant difference in the hydration properties, *i.e.*, the water holding capacity and swelling capacity, but no significant difference in the oil holding capacity. However, in general, the physical properties, *i.e.*, the water holding capacity, swelling capacity and oil holding capacity, showed changes at a temperature of 200 °C and 11.5 min. This is considered to be related to the cellulose content (Fig. 4a).

The increased water holding capacity of steam-exploded insoluble fibers may be due to the increased amount of water that can be trapped by the structure due to the increased surface area after the water contained within the lignocellulosic biomass is released during the steam explosion treatment (Elleuch *et al.* 2011). In addition, the swelling capacity depends on the physical structure (porosity and crystallinity) of the fiber matrix; therefore, an increase in swelling capacity might be attributed to a rise in the amount of short chains, and to the increased surface area of dietary fiber *via* steam-explosion treatment (Wang *et al.* 2015).

It was found that the physical properties decreased when the temperature was 230 °C. The hygroscopicity of the biomass was high due to the presence of hydroxyl (OH) groups in the hemicellulose and cellulose. Those OH groups provide active bonding sites for water molecules. These results are considered as a sharp decrease in the number of available hydroxyl groups according to the severity of the steam explosion process.

CONCLUSIONS

1. The chemical (cellulose, hemicellulose, lignin, and total dietary fiber content) and physical properties (water-holding capacity, oil-holding capacity, and swelling capacity) of steam exploded pine chips showed significantly different results according to the temperature and time at the same severity (R_o), *i.e.*, 4.0.
2. When the steam explosion temperature (190 to 230 °C) and time (20.0 to 1.5 min) were changed, the chemical properties were altered. The solid recovery ranged from 92.4% to 96.4%, the cellulose content ranged from 29.4% to 33.4%, the hemicellulose content ranged from 8.5% to 13.0%, the lignin content ranged from 37.0% to 38.7%, and the total dietary fiber content ranged from 81.2% to 84.7%, which indicated a significantly changed result.
3. The physical properties, *i.e.*, the water-holding capacity, oil-holding capacity, and swelling capacity, ranged from 7.6 g/g to 8.3 g/g, 5.8 g/g to 6.8 g/g, and 4.4 mL/g to 4.9 mL/g, respectively, and were the highest in the steam exploded pine chips at a temperature of 200 °C for 11 min. Steam explosion treatment has been effective in enhancing the physical properties of lignocellulosic biomass and is applicable to industrial processes because of its low capital investment, moderate energy requirements and low environmental impact. Also, it is necessary to consider various temperatures and conditions during the steam explosion treatment.

ACKNOWLEDGMENTS

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