Effect of Modified Dietary Fiber Extracted from Wholegrain Wheat on the Physicochemical and Cake Properties

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Received August 28, 2015
Revised December 2, 2015
Accepted December 28, 2015
Published online April 30, 2016

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pISSN 1226-7708
eISSN 2092-6456

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Abstract Dietary fiber (DF) extracted from whole wheat was chemically modified by cross-linking (CL), carboxymethylation (CM), and hydroxypropylation (HP) and their physicochemical properties and baking performance were evaluated. CM contributed to the increase in soluble DF (SDF) contents and water solubility. While CL and HP contributed to the decrease in water solubility as insoluble DF (IDF) contents increased. In comparison to the chemically modified DF, CLDF deteriorated textural and physical properties of cakes. On the other hand, the cakes with CMDF and HPDF had improved cake qualities such as appearance, higher volume and lower hardness compared to cake with native DF. Consequently, this study suggested that the chemical modifications of wheat DF can effectively regulate the IDF/SDF ratio and total dietary fiber (TDF) contents, resulting in the change of physicochemical properties of them. When applied to cake, modified wheat DF diversified the physicochemical properties and quality by a functional group.

Keywords: wholegrain wheat, dietary fiber, chemical modification, cake property

Introduction

Dietary fiber (DF) has been confirmed as a key nutritional factor with a great physiological significance in the frame of a healthy diet (1). Cereal DFs have heterogeneous chemical structures and they are conventionally classified into insoluble and soluble DF fractions based on water solubility (2). Insoluble DF (IDF) mainly helps material smoothly move through the digestive system, thereby promoting laxation and it includes cellulose, hemicellulose, and lignin. Soluble DF (SDF) helps to reduce blood cholesterol and controls blood glucose levels and it includes oligosaccharides, pectins, β -glucans, and galactomanan gums (3). Also, DF plays some functional roles in food system such as enhancement or modification of hydration properties, oil holding capacity, gel formation, emulsifying properties and texture of food products (3). In recent years, since variations in IDF/SDF ratio influence the physicochemical as well as metabolic properties, the concept of DF with various IDF/SDF ratios have gained attention (4). Consequently, a lot of studies have attempted to modify the technological properties of DF using chemical, physical and/or enzymatic methods (5). Zhou *et al.* (6) reported that enzymatic treatment of tartary buckwheat (*Fagopyrum tatarium*) bran increased SDF/IDF, resulted in the improved water holding capacity and swelling capacity. Sugar beet fibers modified with acidic solutions increased the swelling capacity of IDF, which might be responsible for the damage of coherence of the cell walls (7).

High-quality bakery products have various advantages such as high volume, uniform crumb structure, tenderness, shelf-life and tolerance to staling (8). These characteristics depend on several factors such as characteristics and level of baking ingredients (9). Therefore, the functional ingredients such as the modified starches and hydrocolloids are able to improve product texture, moisture retention and the overall quality in bakery products (8). However, in some cases, DF addition in bread- or cake-type products does not meet the product quality as functional ingredients, causing decreased volume and mouth-feel. Traditionally, commercial cellulose derivatives (e.g. carboxymethylcellulose (CMC) and hydroxypropylmethylcellulose (HPMC)) have been used to improve baking quality such as mouth-feel, maintaining of moisture and anti-staling in bread and cakes (e.g. (10)). Chemical modification is widely achieved through etherification, esterification, and cross-linking. These modifications involve the introduction of functional groups into the biopolymer molecules, resulting in markedly altered physicochemical properties (8). Thus, chemically modified DFs have shown high potential to positively impact the quality of the bakery product by increasing nutritional value and improving physical properties. On the contrary, chemically modified DF is not commonly used in bakery products, which would be related to the concept of the consumers of such products to “unnatural” or “unsafe” constituents (11). However, the effects of chemical modification of DF could be a useful tool to understand their functional properties and widen the applications as food

technological agents. Therefore, the present study was undertaken to investigate the effect of chemical modification (e.g. cross-linking (CL), carboxymethylation (CM) and hydroxypropylation (HP)) on the physicochemical properties of DF extracted from wholegrain wheat and then, modified wheat DF with varying IDF/SDF ratio were added to wheat flour to research baking performance in real food.

Materials and Methods

Materials Wholegrain wheat (*Triticum aestivum*) used in this study was obtained from Nong-Hyup Market (Seoul, Korea). Wholegrain wheat was ground to pass through a 50 mesh sieve and whole wheat flours were stored in a plastic bag. Sodium trimetaphosphate (STMP), sodium tripolyphosphate (STPP) and propylene oxide were obtained from Sigma-Aldrich Chemical Co. (St Louis, MO, USA). Chloroacetic acid was obtained from Acros Organics (Morris Plains, NJ, USA). All other chemicals used in this study were analytical grade reagents. Commercial wheat flour was obtained from CJ Co., Ltd. (Seoul, Korea). Shortening, sugar, salt, non-fat dry milk, dried egg white, and baking powder were purchased from a local market. Wheat flour, Native-, CL-, CM- and HP-DF were passed through a 30- μ m mesh screen.

Extraction of DF from whole wheat flour Wheat DF was extracted from flour according to the procedure described by Park *et al.* (4). In brief, whole wheat flours (30 g) were suspended in 600 mL distilled water and stirred for 1 h. The mixture was centrifuged at $2,218\times g$ for 15 min. The residue was resuspended in 600 mL distilled water and autoclaved at 121°C for 15 min. After equilibrium at 70°C , α -amylase (Termamyl[®], 0.2% v/v) was added to the suspension, and the sample was then incubated for 2 h. The residue was precipitated by adding 95% ethanol and centrifuged at $2,218\times g$ to separate the fiber-enriched materials. After drying at 40°C for 24 h, the residue was ground and passed through a 100 mesh sieve.

Preparation of modified DF Cross-linked (CL) DF was prepared according to the method of Woo and Seib (12) with slight modifications. Wheat DFs (10 g) were suspended in 100 mL of distilled water containing 12% (flour basis) STMP:STPP (99:1). The pH was adjusted to 11.0 with 0.1 N NaOH and suspension was reacted at 45°C for 3 h in a shaking water bath. After 3 h, the suspension was neutralized with 1 N HCl, washed with distilled water, and dried at 40°C for 24 h in a dry oven. Carboxymethylation (CM) of DF was carried out according to the modified method of Bao *et al.* (13). Wheat DF (5 g) was suspended in 2-propanol of 150 mL and stirred for 30 min. The NaOH solution (13%) of 6.3 mL was slowly added over a period of 15 min and the suspension was vigorously stirred for 90 min. After 90 min, 15% chloroacetic acid (6.3 mL) was added and reacted at 50°C for 3 h in a shaking water bath. The suspension was neutralized with 1 N HCl, washed, and dried at 40°C for 24 h in an oven. Hydroxy-

propylation (HP) of DF was carried out as described by Waliszewski *et al.* with some modifications (14). A wheat DF (5 g) was suspended in distilled water (50 mL). Sodium sulfate (1 g) was added and the pH was adjusted to 11.0 with 1 M NaOH. Propylene oxide (2 mL) was added and the suspension was reacted at 40°C for 24 h in a shaking water bath. After 24 h reaction, the suspension was neutralized with 1 M HCl, washed, and dried at 40°C for 24 h in an oven.

Determination of TDF, SDF and IDF Soluble, insoluble, and total dietary fiber contents were determined according to the gravimetric enzymatic method as previously described by Prosky *et al.* (15).

Hydration property measurements The hydration property (water solubility, swelling power, and water absorption index) of native and chemically modified DFs were measured by the method of Lee and Inglett (16). Each sample (0.25 g) was dispersed in 10 mL of distilled water and mixed by vortexing. The mixture was placed at 25°C for 30 min and centrifuged at $3,000\times g$ for 20 min. The supernatant was dried at 105°C in an oven.

Water solubility, swelling power, water absorption index were calculated as follows:

Water solubility (WS, %)

$$=100\times\text{dry supernatant weight/dry sample weight}$$

Water absorption index

$$=\text{residual sample weight/dry sample weight}$$

Swelling power

$$=\text{wet sediment weight}/\{\text{dry sample weight}\times[1-(\text{WS}/100)]\}$$

Preparation of cake batter and baked cakes The batter was prepared according to the AACC Approved Method 10-09 (17) with slight modifications. The cake batter formulation contained the following ingredients: wheat flour (50 g), shortening (25 g), sugar (60 g), water (72.5 g), non-fat dry milk (6 g), dried egg white (4.5 g), baking powder (2 g), and salt (1.5 g). Cake batter containing DF of 1 and 3% contents was made by replacing wheat flour with DF (0.5 and 1.5 g). The cake batter without DF was used as a control. The shortening and sugar were placed in a mixer bowl of Kitchen Aid food processor (Kitchen Aid, St. Joseph, MI, USA) and mixed together using a paddle for 2 min. Water (30 g) was then added and mixed for 2 min. Other dry ingredients and remaining water (42.5 g) were added, and mixed at speed 1 for 0.5 min and scraped down. The cake batter was mixed at speed 6 for 2 min and scraped down again. The mixing was continued for a further 0.5 and 2 min. The cake batter was used for rheological measurement. The cake batter (200 g) was poured into a round cake pan (10 cm diameter) and baked at 150°C for 1 h. All experiments were done in duplicate, and cakes were cooled at room temperature for 1 h before analysis.

Cake physical properties Cakes were weighted and photographed

after 1 h after removal from the pan. All quality measurements were performed within 4 h after baking. Volume and specific volume of the cakes were automatically evaluated using a Volscan Profiler (Stable Micro System Ltd., Godalming, UK) at a set laser distance of 1 mm. The colors of cakes were measured using a Handheld Chroma Meter (CR-400; Minolta Co., Ltd., Osaka, Japan). The chromameter was calibrated using a Minolta calibration plate. The value of L^* (lightness/darkness), a^* (redness/greenness), and b^* (yellowness/blueness) were measured.

Cake texture measurements Textural properties of the cakes were measured using a texture analyzer (TA-XT2; Stable Micro Systems Ltd.). Cake slices (2 cm height×2 cm diameter) were placed on the platform of the texture analyzer and a 35-mm-diameter cylinder aluminum probe (P35) was used to compress the cake samples to 50% of the original height at a speed of 60 mm/min. For the deformation curve obtained, the hardness, springiness and cohesiveness were obtained.

Statistical analysis All analyses were carried out triplicate unless indicated. SPSS (SPSS Inc., Chicago, IL, USA) was used for statistical analysis. Analysis of variance (ANOVA) was applied to determine significant differences among samples, followed by Duncan's multiple range tests for mean comparison at a level of 5%.

Results and Discussion

Physicochemical characterization of modified DF Dietary fiber profiles of native (NADF) and chemically modified wheat dietary fiber (DF) are shown in Fig. 1. The total dietary fiber (TDF) content of cross-linked wheat dietary fiber (CLDF) was significantly increased, which was mainly composed of insoluble DF (IDF) ($p<0.05$). These results were consistent with those obtained by Yamazaki *et al.* (18) shown that Tossa jute IDF phosphorylated by sodium metaphosphate and sodium polyphosphate had greater TDF content than unmodified. Also, TDF content of hydroxypropylated wheat DF (HPDF) was increased by increasing the IDF content. The IDF content of CLDF and HPDF was increased from 47.1% (NADF) to 58.3 and 55.0%, respectively. On the other hand, CM treatment significantly reduced the IDF content (47.1→37.4%) while the SDF content significantly increased (4.5→11.0%) ($p<0.05$). It is likely that IDF turned into SDF after CM.

Fig. 1. Dietary fiber profiles of native and chemically modified wheat dietary fiber (DF). NADF, native wheat DF; CLDF, cross-linked wheat DF; CMDF, carboxymethylated wheat DF; HPDF, hydroxypropylated wheat DF

These results clearly showed that introduction of various functional groups significantly altered the DF content as well as IDF/SDF ratio. Hydration properties of native and chemically modified DF are shown in Table 1. CM treatment significantly increased water solubility compared to NADF ($p<0.05$). These properties may stem from the introduction of carboxymethyl groups, which strengthen the hydrophilic properties of DF and increase solubility (19). In contrast, CL and HP treatments decreased the solubility of wheat DF. The decreased solubility of CLDF was due to the formation of intermolecular bridges by phosphorous residues (20). Thus, the solubility of cross-linked DF decreased compared to that of native DF. In case of HP, many studies have shown that the introduction of hydroxypropyl groups increases the solubility of polysaccharide derivatives (21,22). This interpretation was supported by the finding published by Kittipongpatana and Kittipongpatana (22) who proposed that HP disrupted the molecular bonding force in the polysaccharides and weakened the structure. However, Choi *et al.* (23) reported the opposite results for hydroxypropylated rice starch. It is speculated that the decreased solubility of HPDF, when compared to native DF, was depending on differences in structural properties such as crystallinity and particle size. In this study, the water absorption index of wheat DF was increased by chemical modification; water

Table 1. Effect of chemical modifications on hydration properties of wheat dietary fiber (DF)

Hydration property	Wheat DF ¹⁾			
	NADF	CLDF	CMDF	HPDF
Water solubility (%)	28.32±0.94 ^{b2)}	16.01±0.13 ^d	39.07±1.24 ^a	19.49±0.24 ^c
Water absorption index	3.38±0.10 ^d	4.67±0.03 ^c	5.75±0.16 ^a	5.24±0.06 ^b
Swelling power (g/g)	4.72±0.13 ^d	5.56±0.04 ^c	9.44±0.23 ^a	6.50±0.06 ^b

¹⁾NADF, native DF; CLDF, cross-linked DF; CMDF, carboxymethylated DF; HPDF, hydroxypropylated DF

²⁾Mean values marked with different letters in the same rows are significantly different ($p<0.05$).

Fig. 2. Cake volume of control cake and cakes made with native or chemically modified wheat dietary fibers. Control, wheat flour; Native, native wheat DF; CL, cross-linked wheat DF; CM, carboxymethylated wheat DF; HP, hydroxypropylated wheat DF

absorption index and swelling power of chemically modified DF showed a similar tendency that CMDF was greater than those of CLDF and HPDF.

Physicochemical properties of cakes The appearance of cakes supplemented with different level of native (NADF) and chemically modified wheat DFs (CL, CM, and HP) is presented in Fig. 2. The NADF cake showed a poor quality like reduction of cake volume and very dense structure as compared to control made with wheat flour. These results were consistent with the study by Gómez *et al.* (24), who reported that adding dietary fibers from different source to bread decreased volumes due to their interaction with gluten, leading to a decrease of the gas retention capacity and restricted expansion of the gas cells. Thus, notable decrease volume in cake added with NADF was possibly attributed to a decreased entrapment

of gas bubbles, resulting in a dense structure (25). On the contrary, the cakes incorporating chemically modified DFs produced a well-aerated structure and high cake volume except for the cake added with 3% CLDF. This observation was confirmed in Table 2, which showed low volume and specific volume.

The effect of wheat flour replacement with native and modified wheat DF on cake properties (volume, crumb color, and crust color) is presented in Table 2. The substitution of NADF caused a significant decrease of cake volume as compared to control prepared with wheat flour ($p < 0.05$). On the other hand, the presence of modified DF, except when CLDF was added, caused an increase in cake volume. The volume of cake made with 1% CLDF was not changed whereas a significantly lower value was found at the level of 3% ($p < 0.05$). The volume of cakes added with CMDF remained relatively unchanged as CMDF level increased from 1 to 3% while the addition of HPDF

Fig. 3. Textural properties of cakes made with and without chemically modified wheat DF. Control, wheat flour; CL, cross-linked wheat DF; CM, carboxymethylated wheat DF; HP, hydroxypropylated wheat DF

significantly ($p < 0.05$) decreased the volume from 306.9 to 300.5 mL. It seemed that the more effective changing volume was exerted by the addition of CMDF, which was followed by HPDF. On the other

hand, when CLDF was applied to cake bakery, the volume was the smallest compared to other cakes made with chemically modified DF. It suggested that decreased volume by adding the cross-linked DF seemed to cause less soluble in water and less dispersed in the dough. As a result, CLDF were unevenly distributed and destabilized gas cells when made as cake (26). Generally, hydroxypropylmethyl cellulose (HPMC, E464) is widely utilized in bakery products (27) whereas carboxymethyl cellulose (CMC, E466) is quite limitedly used. However, in this study, the cake volume was the highest when CMDF was added, which would be related to the solubility in water. It is well known that HPMC with the hydroxypropyl group showed affinity to the non-polar or lipid phase of a multi-phase system such as bread dough (28). Therefore, the significant increase of volume in the HPMC cake was possibly attributed to a formation of an interfacial film at the boundaries of gas cells and stability for the cells during expansion.

Considering the crumb and crust color of cake (Table 2), L^* values of cakes incorporating the NADF and chemically modified DF were significantly lower than the control cake ($p < 0.05$). These values were decreased with the increasing DF levels. These results were not consistent with the study by Park *et al.* (29), who reported that adding wheat fiber to bread caused a pale crust color. A possible reason for this difference might be the source of wheat DFs which was produced from whole grain flours (30). The NADF or modified DF cakes exhibited significantly ($p < 0.05$) higher a^* values than the control cakes, which followed by CLDF > CMDF > HPDF > NADF. Also, a^* values of all samples were increased with the increasing DF levels, indicating a more red appearance. The cake containing 1% NADF and chemically modified DF exhibited similar b^* values and decreased significantly with DF level compared to control cake.

Regarding textural properties, cakes made with NADF and CLDF were not carried out (results not shown) because of unripe effect/ characteristics (except for those prepared with 1% CLDF) after baking. The CLDF cake showed the highest hardness and the lowest springiness and cohesive values (Fig. 3). On the contrary, cake made with CMDF and HPDF had significantly lower hardness than control on the day of baking as well as during storage of 5 days ($p < 0.05$). The hardness of CMDF and HPDF cakes decreased with increasing DF level. All cakes showed an increase in hardness during storage, but no significant differences were found (data not shown) in the cakes made with chemically modified DF and wheat flour. With respect to springiness, the cakes made with CMDF and HPDF showed higher springiness values than control cake after 5 days of storage. The cohesiveness values of the cakes made with CMDF and HPDF cakes were higher than that of the control cake, although no significant differences were found. The cakes made with CMDF and HPDF exhibited a decrease in cohesiveness as the addition of DF increased. The same trend was observed during each day of storage.

In the present study, the DF was extracted from the whole grain wheat, and chemically modified (cross-linking, carboxymethylation, and hydroxypropylation). Native and chemically modified wheat DF

Table 2. Volume and color of cakes made with and without chemically modified wheat dietary fibers

Sample ¹⁾	Volume		Crust			Crumb	
	(mL)	L*	a*	b*	L*	a*	b*
Control	297.2±0.3 ^{c2)}	60.4±0.8 ^a	-0.6±0.4 ^e	18.7±0.7 ^{bc}	62.50±0.5 ^a	-2.2±0.0 ^g	11.2±0.1 ^{cd}
NADF-1%	272.6±0.4 ^d	58.2±0.2 ^{bc}	0.3±0.2 ^d	18.9±7.3 ^{abc}	61.0±0.9 ^{ab}	-1.4±0.0 ^f	10.5±0.2 ^e
NADF-3%	261.9±0.6 ^e	56.7±1.0 ^d	1.1±0.4 ^{bc}	17.7±0.5 ^d	52.9±1.1 ^f	-0.4±0.0 ^c	9.0±0.2 ^g
CLDF-1%	295.4±0.3 ^c	57.1±0.9 ^c	1.7±0.9 ^{ab}	18.9±1.1 ^{abc}	61.4±0.4 ^a	-0.9±0.0 ^d	11.1±0.2 ^d
CLDF-3%	279.6±0.3 ^d	54.6±0.7 ^f	2.2±0.8 ^a	16.9±1.1 ^e	51.1±0.5 ^g	0.5±0.1 ^a	10.1±0.2 ^f
CMDF-1%	307.2±0.2 ^a	58.8±1.0 ^b	1.0±0.6 ^c	19.3±0.9 ^{ab}	59.6±0.9 ^c	-0.9±0.0 ^d	12.1±0.2 ^a
CMDF-3%	307.0±0.3 ^a	55.5±0.8 ^{ef}	2.3±0.6 ^a	18.3±0.6 ^{cd}	56.9±0.9 ^e	0.7±0.0 ^a	13.1±0.2 ^a
HPDF-1%	306.9±0.6 ^a	57.8±0.5 ^c	1.0±0.7 ^c	19.5±0.8 ^a	60.7±0.4 ^b	-1.3±0.1 ^e	11.4±0.1 ^c
HPDF-3%	300.5±0.2 ^b	55.1±1.4 ^e	2.2±1.1 ^a	18.6±1.0 ^{bc}	57.6±0.6 ^d	0.3±0.0 ^b	11.9±0.1 ^b

¹⁾Control, wheat flour; NADF, native DF; CLDF, cross-linked DF; carboxymethylated DF; HPDF, hydroxypropylated DF

²⁾Mean values marked with different letters in the same column are significantly different ($p < 0.05$).

showed different dietary fiber compositions and physicochemical properties. The characteristics of cakes made with chemically modified DF such as physicochemical and textural properties were significantly dependent on the types of chemical modification. These results indicated that variation of modified wheat DF could be the method to find their potential diverse application in fiber-rich food products.

Acknowledgments This research was supported by High Value-added Food Technology Development Program (Project title: Development and industrialization of import-substituting starch materials using domestic agricultural sources, Project No. 314041-3), Ministry of Agriculture, Food and Rural Affairs.

Disclosure The authors declare no conflict of interest.

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