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Characterization of Wheat Flour Treated by Supercritical Carbon Dioxide

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Abstract

This study was to investigate the production of reducing sugars in hydrolysates from raw and deoiled *Laminaria japonica* produced by subcritical water hydrolysis. Deoiled *Laminaria japonica* was collected by supercritical carbon dioxide (SCO_2) extraction process. Experiments were performed in a batch-type reactor with stirring. It investigated that the effects of reaction temperature and acetic acid as catalyst on content of reducing sugar production. The addition of acetic acid led to an increase in content of reducing sugar. But Removal of oil in *Laminaria japonica* by SCO_2 and increasing of temperature led to decrease in content of reducing sugar production. The highest content of reducing sugar was 814.10 mg/100 g raw dried sample at 200^{o}C, adding of 1% acetic acid as catalyst

Keywords: Subcritical carbon dioxide; Wheat flours; Acid value; Mycotoxin; Volatile organic compounds

1. Introduction

Wheat (*Triticum* spp.) [1] is a cereal grain, originally from the Levant region of the Near East, but now cultivated worldwide. Wheat is grown on more land area than any other commercial crop and is the most important staple food for humans. World trade in wheat is greater than for all other crops combined.[2] Wheat grain is a staple food used to make flour for leavened, flat and steamed breads, biscuits, cookies, cakes, breakfast cereal, pasta, noodles, couscous[3] and for fermentation to make beer,[4] other alcoholic

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beverages[5] . 100 grams of hard red winter wheat contain about 12.6 g of protein, 1.5 g of total fat, 71 g of carbohydrate (by difference), 12.2 g of dietary fiber, and 3.2 mg of iron (17% of the daily requirement); the same weight of hard red spring wheat contains about 15.4 g of protein, 1.9 g of total fat, 68 g of carbohydrate (by difference), 12.2 g of dietary fiber, and 3.6 mg of iron (20% of the daily requirement).[6] Wheat flour is a powder made from the grinding of wheat used for human consumption. More wheat flour is produced than any other flour. Wheat varieties are called "clean," "white," or "brown" if they have high gluten content, and they are called "soft" or "weak" flour if gluten content is low. Hard flour, or *bread flour*, is high in gluten, with 12% to 14% gluten content, and has elastic toughness that holds its shape well once baked. Soft flour is comparatively low in gluten and so results in a finer or crumbly texture. [7] Soft flour is usually divided into cake flour, which is the lowest in gluten, and pastry flour, which has slightly more gluten than cake flour. The aim of this present work was to assess the effect of supercritical carbon dioxide (SCO_2) treatment in different experiment conditions on quality inhibition effect of wheat flours. The conditions of extraction were selected by considering their feasibility for industrial application.

2. Materials and methods

2.1. Materials

The sample imported from North Dakota, USA was milled and used. The pure carbon dioxide (99.99%) was supplied by KOSEM (Yangsan, Republic of Korea). 70%-ethanol was purchased as additives.

2.2. Supercritical carbon dioxide extraction (SCO_2)

The Experimental conditions of supercritical carbon dioxide showed in Table 1. Batch reactor filled with 5 kg of flour, it was treated for 3 h by 60 L/h of CO_2 and 4.2 L/h of ethanol. Both were preheated at 40℃. After the treatment, flour was collected from the reactor and sieved (1,000 μm) by mesh. Treated samples were compared with the control group.

Table 1. Conditioning of supercritical carbon dioxide extraction

Flour From	condition
Control	untreated wheat flour
SFC1	wheat flour in treated pressure at 100 bar and temperature 40°C
SFC2	wheat flour in treated pressure at 200 bar and temperature 40°C
SFC3	wheat flour in treated pressure at 300 bar and temperature 40°C
SFC2E	wheat flour in treated pressure at 200 bar and temperature 40°C + 70%-ethanol
SFC3E	wheat flour in treated pressure at 300 bar and temperature 40°C + 70%-ethanol

2.3. Fat acidity

Samples were stored at room temperatures for 10 months and fat acidity was measured according to AACC method [8]. 10 g of samples were dissolved in 100 mL of distilled water and then, kept 1 h at room temperature. 17.6 mL of distilled water and 0.5 mL phenolphthalein solution was added in 17.6 mL of suspension of sample. Fat acidity was titrated with 0.1N-NaOH solution until pink color persists.

2.4. Acid value

The acid value was assessed according to the AOCS method [9]. 1 g of sample was dissolved in 100 ml of ether: ethanol (1:1) and shaking. Then phenolphthalein solution as an indicator was added drop wise. Acid

value of oil was analyzed by titration with 0.1N KOH-ethanol solution until the pink color persists for at least 30 s.

2.5. Microorganism

The detection of *E. coli* and aerobic was allowed microorganism assay of KFDA [10]. 1 g of sample was homogenized with 100 ml of sterile water and taken 1 ml. Then, samples for *E. coli* detection were stored for 24~48 h and samples for aerobic detection were stored for 24 h in incubator at 35±1˚C.

2.6. Mycotoxin

Mycotoxin was measured using the modified method of KFDA and Deok-hwa Jung (2007) [11]. 50 g of sample and 5 g NaCl was dissolved in 100 ml of 80% methanol and shaking violently. It was filtered using Whatman paper No. 4. 10 ml of filtrate was diluted in 40 ml of distilled water and again filtered by 1.6 μm Glass microfiber.

2.7. Volatile organic compounds

The analysis of volatile components was performed using a GC/mass spectroscopy (MS) system (HP 6890/QP 2010A, Shimadzu, Japan). Samples were volatilized at 50˚C in a drying oven during 30 min. Thereafter, the volatile compounds volatilized from each sample were absorbed on an absorption tubes (Tenax-TA, Supelco Inc., USA) for 5 min. The volatile compounds absorbed in tubes were desorber into the automatic thermal desorber (ATD; ATD-400, Perkin Elmer, UK) which is directly connected with GC/MS equipped with an AT-1 column (60 m x 0.32 mm i.d., 1.0 μm film thickness). The spectrum of each analyzed off-flavour compounds agreed with that presented in the mass spectrum library (NIST21, NIST107, and WILEY229). The percentage of identified off-flavour compounds was presented by peak area %.

3. Results and Discussions

3.1. Fat acidity

Acidity was used to indicate freshness of wheat flour. Fat acidity of wheat flour shows in Table 2. Untreated wheat flour and wheat flour treated by SCO_2 was respectively 0.37 and 0.12 mg on an average. Wheat flour treated by SCO2 and additives 70%-ethanol showed lower than SCO_2 treatment. Comparing to untreated wheat flour, wheat flour treated by SCO_2 maintained freshness well. It was regarded that fat content was decreased in wheat flour treated by SCO_2.

Table 2. Fat acidity and acid value of wheat flours

	Control (mg)	SFC1 (mg)	SFC2 (mg)	SFC3 (mg)	SFC2E (mg)	SFC3E (mg)
Fat acidity	0.37	0.18	0.10	0.08	0.09	0.09
Acid value	27.33	16.64	15.40	11.74	14.58	12.87

3.2. Acid value

Acid value of wheat flour shows in Table 2. Acid value was carried out to measure the quality of the oil and oxidation state of lipid. Wheat flour treated by SCO_2 showed low acid value than untreated wheat flour. It was also found that as the pressure increase, the amount of AV decrease.

3.3. Microorganism

Table 3 shows microorganism assay of wheat flour in different experimental conditions. *E.coli* was non-detected in all samples, and aerobic of wheat flour treated by SCO_2 was also non-detected. But 50 CFU of aerobic was detected in untreated wheat flour. SCO_2 treatment was regarded more efficient to sterilize microorganism like a precedent study (Korea university. 2007).

3.4. Mycotoxin

Mycotoxin activity of wheat flour shows in Table 3. Aflatoxin of untreated wheat flour was detected 0.59 ppb, but samples treated by SCO_2 were non-detected. In case of Ochratoxin A, all samples were non-detected. So, SCO_2 treatment was more efficient than untreated wheat flour.

Table 3. Microorganisms and Mycotoxin detection of wheat flours

	Control	SFC1	SFC2	SFC3	SFC2E	SFC3E
Aerobic	50CFU	ND	ND	ND	ND	ND
E. coli	[a]ND	ND	ND	ND	ND	ND
Aflatoxin	0.59 ppb	ND	ND	ND	ND	ND
Ochratoxin A	ND	ND	ND	ND	ND	ND

[a]ND : Not Detected

3.5. Volatile organic compounds

Table 4 shows volatile organic compounds of wheat flour in different experimental conditions. Total area of untreated wheat flour was 74.94; it was higher than that of SCO_2 treatment (38.13) on an average. And wheat flour treated by SCO_2 and additives 70%-ethanol was 25.56 on an average. Comparing to untreated wheat flour, wheat flour treated by SCO_2 and SCO_2 with additives 70% ethanol contained lower volatile compounds. So, it was regarded that Volatile compound was reduced in wheat flour by the pressure.

Table 4. VOCs in wheat flours using supercritical carbon dioxide at at 40℃, 100, 200 and 300 bar and additives 70%-ethanol

Compound name	R.T	Area%					
		Control	SFC1	SFC2	SFC3	SFC2E	SFC3E
Ammonium bicarbornate (pyrolysis)	2.93	27.30	[a]ND	ND	ND	ND	ND
Dimethylhydrazone	3.13	7.28	ND	ND	ND	ND	ND
Acetaldehyde	3.23	0.56	7.27	11.55	14.06	21.26	13.21
Methyl Alcohol	3.32	1.52	18.53	47.23	29.04	30.05	30.80
Butane, 1-isocyano-	3.97	0.59	ND	ND	ND	ND	ND
Acetone	4.15	0.20	ND	ND	ND	ND	ND
Nonanal	26.80	4.11	19.98	14.94	ND	11.54	21.93
Decanal	32.26	12.09	34.68	17.77	29.50	14.56	25.88
Octadecanoic acid, methyl ester	36.01	12.96	ND	ND	ND	ND	ND
Octadecanoic acid, methyl ester	36.20	8.14	ND	ND	ND	ND	ND
Cyclohexasiloxane, dodecamethyl-	36.74	2.44	ND	ND	ND	ND	ND
Butane, 1,1,3,2-tetrachloro-1,2,2,3,4,4,-hexafluoro-	38.44	6.56	19.06	4.98	25.85	9.12	5.41
3-Ethoxy-1,1,1,7,7,7,-hexamethyl-3,5,5-tris(trimethylsiloxy)tetrasiloxane	41.65	4.06	ND	ND	ND	8.90	ND
Silicate anion tetramer	46.16	0.80	ND	ND	ND	ND	ND
Hexadecanal	46.72	0.89	ND	ND	ND	ND	ND

Octadecanoic acid	48.00	0.87	ND	ND	ND	ND	ND
Heneicosane	48.39	0.79	ND	0.53	ND	2.03	ND
Isopropyl Myristate	48.84	0.59	0.48	0.50	ND	ND	0.49
N,N-Dimethylpalmitamide	49.20	0.33	ND	ND	ND	ND	ND
Eicosane	50.79	0.76	ND	ND	ND	ND	ND
[1,1':3',1"-Terphenyl]-2'-ol	50.91	2.60	ND	1.59	1.56	2.53	2.28
Octadecanoic acid, 12-oxo-, methyl ester	51.61	1.85	ND	ND	ND	ND	ND
1,2-Benzenedicarboxylic acid, dibutyl ester	52.45	1.03	ND	0.90	ND	ND	ND
Hexadecanoic acid, ethyl ester	52.74	1.68	ND	ND	ND	ND	ND
Total		100 (74950000)	100 (41290000)	100 (37750000)	100 (35360000)	100 (18200000)	100 (32920000)

[a]ND : Not Detected

4. Conclusion

In this study, quality inhibition effect of wheat flour was treated using SCO_2 at different conditions. Fat acidity and acid value was lower treated by SCO_2 than untreated wheat flour. Microorganism and mycotoxin activity was not found treated by SCO_2 and treated by SCO_2 with additives (70% ethanol). Volatile organic compound was reduced by the treatment of SCO_2 and SCO_2 with additives. SCO_2 extraction of wheat flour was more efficient than untreated wheat flour in terms of quality inhibition effect.

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