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Evaluation, Characterization of Fiber Content, and Antioxidant Activity of Corncob (*Zea mays* L.) during Alkalization

Edi Suryanto[1]*, Mercy I.R. Taroreh[2]

[1]Department of Chemistry, Faculty of Mathematics and Natural Science, Sam Ratulangi, University, Manado, North Sulawesi
[2] Department of Food Science and Technology, Faculty of Agricultural, Sam Ratulangi University, Manado, North Sulawesi

*Corresponding author email: edi7suryanto@gmail.com

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ABSTRACT. Corncob fiber powder was prepared by alkaline process, and their effects were investigated on the composition, physicochemical, and antioxidant properties of dietary fiber (DF) products. DF content was determined using AOAC enzymatic-gravimetric analysis, while chemical composition in fiber was observed on cellulose, hemicellulose, and lignin. The results showed that cellulose content of significantly increased while hemicellulose and lignin was observed decreasing. Correspondingly, content of the insoluble, soluble, and total DF of corncob was increased by 14.72-20.77, 28.86-47.43, and 15.12-21.42%, respectively. Furthermore, the particle size of DF is distributed in a range from 58.50-83.90 μm. XRD analysis showed the characteristics of cellulose that exhibited increasing crystalline degree from 29.33% to 53.53% and 59.62%, respectively. FTIR shows that the DF matrix structure does not change and in the presence of special structures of cellulose compounds. The SEM results showed that the alkaline process could significantly decrease the particle size and influence on the physicochemical and functional properties of corncob insoluble DF. The results also showed that DF extract has lower DPPH radical scavenging activity. The results suggest that corncob DF have good potential candidate for dietary fiber containing antioxidant ingredient and for their further applications in food.

Keywords: Alkaline, antioxidant, corncob, dietary fiber, physicochemical

INTRODUCTION

Corncob is one of lignocellulosic biomass by product and a fibrous residue that results from corn shelled, which are frequently abandoned as wastes. It contains complex carbohydrate polymer, such as cellulose, hemicellulose lignin, and pectin which is very high, making it is possible to use them as a source of DF in functional foods (Zhu et al., 2014). DF consists mostly of plant cell walls which cannot be hydrolyzed by human digestive enzymes (Zhuang et al., 2012; Zhu et al., 2014). Therefore, dietary fiber has a major role in determining the health and disease conditions of various societies (Ham et al., 2017).

DF has been shown to play an important role in preventing the risk of carcinogenesis, atherosclerosis, as well as controlling the release of glucose over time to help in the proper management of diabetes mellitus and obesity (Tsai et al., 2011). Several studies have reported that the hydration properties of dietary fiber can be improved by milling or the presence of acid and alkaline treatment because of the increased ability to trap water in the fiber matrix (Zhang et al., 2014; Zhu et al., 2014). In addition, the micronization treatment of dietary fiber can increase solubility, water absorption capacity, swelling capacity, and oil absorption capacity (Zhu et al., 2010; Zhang et al., 2013). The solubility of dietary fiber also depends on the nature of the glycosidic components, and the structural characteristics of the food fiber (Zhu et al., 2014). Sangnark and Noomhorn (2003) stated that alkaline hydrogen peroxide treatment can affect physical and chemical properties, including hydration properties such as water holding capacity and oil binding capacity from bagasse dietary fiber. However, the weakness of lignocellulosic material is that it does not have good hydration properties, because it is not soft or does not blend when applied in food ingredients. Several researchers have reported improvements in the hydration properties of lignocellulosic materials using alkaline hydrogen peroxide treatment (Gould et al., 1989).

This alkaline process can also reduce the lignin content and also the stirring which is an integral part of the process, opening the fiber structure by mechanical cutting, making available free hydroxyl groups of cellulose to bind with water. Chemical components including cellulose, hemicellulose and lignin can be related to the physical properties of food fiber. Some physical properties of dietary fibers such as hydration, density and particle size distribution can increase their use for various food and industrial needs (Zhang et al., 2014; Sangnark and Noomhorn, 2003; Qi et al., 2015).

In addition to, corncob contain phenolic compounds, flavonoids, tannins and have antioxidant activity, singlet oxygen quenching and sunscreen (Suryanto et al., 2013; Suryanto & Momuat, 2017; Suryanto et al., 2018a). If look with its unique of corncob which has such as bioactive polysaccharide and rich phytochemicals, it is not impossible that this waste material can be a potential of fiber source for food ingredients. Recently, much attention has been paid to polysaccharides because they may possess a variety of biological activities such as inmunostimulating, antioxidant, antitumor, antihyperlipidemic and antidiabetic activities (Liu et al., 2015; Yu et al., 2015). In addition, polysaccharide are usually biodegradable, nontoxic, and with few adverse effect (Jia et al., 2013). Therefore, they may potentially be developed as natural functional products. In addition, corncob was obtained from plant material waste having the large amount of dietary fiber which can be used an attractive economic source in food and chemical industry.

Therefore, a series of studies in the fiber components of corncob are needed, and the physicochemical and functional properties using alkaline treatment to extract dietary fiber has not been reported, particularly content of dietary fiber and chemical composition. The purpose of this study was to evaluate the alkaline treatment effect on the fiber content and antioxidant activity and also to investigate characteristics of fiber-rich powder of corncob.

EXPERIMENTAL SECTION

The sample used was the corncobs variety Manado kuning obtained from Tompaso Minahasa, North Sulawesi, Indonesia. The chemicals used were ethanol, sulfuric acid, hydrochloric acid, sodium carbonate, Folin-Ciocalteou reagent from Merck (Darmstadt, Germany), 2-diphenyl-2-picrylhydrazyl (DPPH), 4,6, gallic acid, α-amylase (termamyl), protease, amiloglucosidase were obtained from Sigma Chemical Co. (St. Lois, MO). All the chemicals used in the experiment were analytical grade.

Preparation of Corncob DF

The corncob pulp is then heated with 1000 mL of distilled water for 1 h under constant stirring of 200 g, and filtered to separate insoluble fraction. Insoluble dietary fiber (IDF) fraction was dried in the oven at 55-60 °C for 48 hours, and ground by a laboratory mill using Formac type FCT-Z200 mill for 2 minutes, and passed through a 30-mesh and 200-mesh screen. All powders were hermetically packaged in plastic bags and stored at room temperature in a desiccator for the following step.

Alkaline Treatment

Corncob powder (25 g) in different particle sizes of 30 and 200-mesh were blended with 250 mL 2M NaOH for 5 minutes, and shaken at 250 rpm for 2 hours at room temperature and centrifuged at 5000 g for 15 minutes. The residue was neutralized with 6 M hydrochloric acid and distilled water to pH 7 and dried at 50-60 °C for 24 hours. The samples obtained were denoted as ADF30, and ADF200, respectively, while without alkaline samples were termed WADF. Furthermore, the sample was stored in a desiccator for further experiments.

Chemical Analysis of DF

Determination of total dietary fiber (TDF), insoluble dietary fiber (IDF), and soluble dietary fiber (SDF) contents in defatted corncob fiber sample were determined by an enzymatic gravimetric procedure according to method 991.43 (AOAC, 1995). In brief, dried powder samples were first gelatinized with heat stable α-amylase. After gelatinization, the samples were digested with protease and amyloglucosidase to remove protein and starch in the samples. Subsequently, IDF was filtered and washed with warm distilled water. The filtrate and washed water were combined and added with four volumes of 95% ethanol at 60 ˚C to precipitate the SDF. The mixture was filtered and residue (soluble fiber) was dried and weighed. The DF contents were corrected for residual protein, ash, and blank. After that, TDF was calculated values as the sum of IDF and SDF.

Analyses of Fibers Chemical Composition

Cellulose, hemicellulose and lignin content was determined with using Chesson method (Datta, 1981). In brief, dried powder (1 g) of lignocellulose refluxed for 2 h with 150 mL of H_20 at 100 °C for obtaining hot water soluble such as pectins and oligosaccharides. After that, dried residue refluxed for 2 h with 150 mL of 0.5M H_2S0_4 at 100 °C for obtaining hemicellulose. After cooling, dried residue treated with 10 mL of 72% (v/v) H_2S0_4 at room temperature for 4 h, then diluted to 0.5 M H_2S0_4, and refluxed at 100 °C for 2 h for obtaining cellulose. The residue is dried and then dusted to get lignin.

Particle Size Analysis

The particle size of dietary fiber from corn cobs with alkaline treatment and without treatment was measured by a laser particle size analyzer (Horiba, LA-960, Japan). The particle size distribution was characterized by the values D0.1, D0.5, and D0.9.

X-ray Diffraction (XRD) Analysis

Crystalline structure of dietary fiber from corn cobs with alkaline treatment and without treatment was evaluated using X-ray diffractometer (Brucker, Germany). The determination was executed using Cu Kα radiation using a wavelength of 0.1546 nm. A diffractogram was recorded at Bragg angle (2θ) in steps of 0.05 as well as a spinning rate of 2 = 5-50° used for this purpose. The degree of crystallinity is then taken as the ratio of the crystal peak weight to the entire area of the crystal peak plus background (Zhao et al., 2013). The crystallinity indices (CrI) of the samples were calculated using the following equation:

$$CrI = I_{0,02}-I_{am}/I_{0,02} \times 100$$

Where I0,02, is the maximum intensity of the (002) lattice diffraction peak which is located at a diffraction angle around

FTIR Analysis

A series of tests on the physio chemical properties of dietary fiber from corn cobs include Fourier Transform Infrared Spectroscopy (FTIR) spectra were collected using a FTIR spectrometer (Shimadzu FTIR 8201 PC) over the range 4000-400 cm^{-1} by the potassium bromide (KBr) and pressed into a 1 mm pellet method. The spectrum of pure KBr was used as background.

Scanning Electron Microscopy Analysis

The effect of alkaline treatment on physical structure of each dietary fiber was investigated by scanning electron micrograph. Dried ground native dietary fiber and alkaline-dietary fiber was sieved through a 74 μm screen, then deposited on a metal stub, coated with a thin layer of gold and examined by a scanning electron microscope (JSM-6510 LA, JEOL, Japan) with an accelerating voltage of 15 kV and magnifications of 1000.

Phytochemical Extraction

The phenolic compounds were extracted according to a procedure Suryanto et al. (2018b). Briefly, ten grams of corncob before, and after put into a 250 mL Erlenmeyer flask which was lined with aluminum foil to avoid light, then extracted by sonication for 30 minutes with 50 mL of ethanol-water (80:20, v/v). After that, filtered, and the filtrate is evaporated to obtain the extracts. Subsequently, the 80% ethanol was then added, so the volume of the supernatant was 50 mL in exact. The extract stored at 5 °C for the preparation of phytochemical analysis and testing of antioxidant activity.

Determination of Total Phenolic Content

The total phenolic content of the composite flour was determined using modified Folin-Ciocalteu colorimetric method from Li et al., (2012). Each sample solution (0.1 mL, 1 mg/mL) was added to Folin-Ciocalteu reagent (0.1 mL, 50%) in a test tube and then this mixture was vortexed for 3 minutes. After intervals of 3 minutes, 2 mL of Na_2CO_3 2% solution was added. After incubation at room temperature for 30 minutes, the mixture was kept in the dark for 30 minutes. The supernatant was measured using a spectrophotometer at 750 nm. The standard curve was prepared using different concentrations of gallic acid and the results were expressed as gallic acid equivalents in milligrams per milligram extract.

Determination of Free Radical Scavenging DPPH

Determination of free radical activity (scavenger) from CDF extracts measured by the method Suryanto et al. (2018) slightly modified. A total of 2 mL solution of 1.1-diphenyl-2-picrylhydrazyl (DPPH) 92 µM in ethanol was added 0.5 mL composite flour extract. The level of color reduction of the solution shows the efficiency of radical scavenger. The last five minutes of the 30 minutes, the absorbance was measured with a spectrophotometer at 517 nm. Free radical scavenger activity is calculated as a percentage reduction of DPPH color.

Statistical Analysis

Data was analyzed with computer software, SPSS version 18 using ANOVA analysis followed by Duncan's Multiple Range Test to determine the significant differences between the carrying a by 5%.

RESULTS AND DISCUSSION

Effect of Alkaline Treatment on Yield

The ADF30 and ADF200 yields from corncob treated with alkaline were 52.83% and 37.84%, respectively. ADF30 shows the highest yield compared to DF200. This might be due to a particles size of the ADF200 of corncob by continuous shaking, as well as chemical reactions during alkaline treatment. The reduction in particle size has a significant effect on chemical reactions of corncob fiber, which is related to it the surface area properties. It can expand the touch the surface of the sample, because the surface area can affect the solubility process and the more chemical components are dissolved during the extraction process with alkaline. According to Budtova & Navard (2016), extraction using NaOH greatly affected on the yield so that it can reduce cellulose content obtained in the extraction results.

Fibers Chemical Composition

The changes of chemical compositions of corncob fiber occurred during the alkaline treatment process because chemical reactions of alkaline. The changes include of the lignin binding with cellulose and hemicellulose, some degradation and solubilization of cellulose, hemicellulose and lignin, which result in the improvement on dietary fiber. Alkaline treatment can also release some cellulose and hemicellulose by hydrolyzing ester bonds between lignin and cellulose or hemicellulose so that can improve the biodegradability of lignocellulosic materials (Brodeur et al., 2011). Lignin, cellulose, and hemicellulose are the main compositions of corncob (92.37%) of the total dry mater of corncob. The result of analyses of main compositions during alkaline treatment is shown in **Table 1**.

After alkaline treatment, hemicellulose, and lignin content of ADF30 were reduced by 34.81 and 7.57%, respectively, and the corresponding contents were decreased from 38.84, and 11.96% to 25.32 and

2.94%, respectively, whereas ADF200 was reduced by 34.41 and 23.53%, respectively and those contents were decreased to 15.78 and 5.64%. Meanwhile, cellulose content of ADF30 and ADF200 significantly increased by 20.67 and 25% and the corresponding contents were increased from 41.57% to 62.24 and 66.57%, respectively. It exhibited that lignocelluloses of corncob were degraded after alkaline treatment.

The results indicate that destruction of hemicellulose was highest compared to lignin after treatment. This might be due to the chemical reaction of more hemicellulose as a consequence of alkaline treatment. In addition, alkaline effect also can be partly degradation of lignin and cellulose. Several studies reported that decreasing of cellulose, hemicellulose, and lignin content after alkaline treatment of lignocellulose materials (Shu et al., 2015; Budtova & Navard, 2016; Maharani et al., 2017). However, lignocellulose materials extraction was used NaOH of 0.3-0.7 M will be caused the increase of cellulose content (Maharani et al., 2017). The other studies reported that the effect of alkaline-hydroperoxide treatment (1%) can decrease the lignin content of bagasse and rice straw increase the content of cellulose and hemicellulose (Sangnark & Noomhorm, 2003; Shu et al., 2015). The changes of chemical compositions were generally assisted improve and contribute to the increase of DF content. The decreased content after treatment was also possible able to improving and contributing in soluble and insoluble DF from corncob.

DF content

Based on water solubility, dietary fiber can be divided into soluble dietary fiber (SDF) and insoluble dietary fiber (IDF). The results of characterization of corncob dietary fiber with alkaline and treatment are presented in **Table 2**.

The content of IDF in corncobs fiber increased from 59.32% to 67.88% and 72.77% after alkaline treatment in ADF30 and ADF200 while the SDF content also increased from 1.43% to 2.01% and 2.72%. This result was also followed by a significant TDF content ($p<0.05$). This is probably due to the alkaline treatment changing the fiber component from the TDF fraction to a water-soluble form. In addition, these results indicate that the alkaline treatment causes redistribution of the fiber components from the insoluble to the soluble fractions in the corncob fiber. Soluble fiber can help to lower blood cholesterol and regulate blood glucose levels (Zhu et al., 2014; Du et al., 2014). Several studies indicate that soluble fiber (SDF) is more important than insoluble fiber (IDF) in many health aspects (Du et al., 2014; Zhu et al., 2015). The increase in SDF and IDF content after alkaline treatment might be due to decreased lignin and hemicellulose and cellulose content (**Table 1**). The DF included cellulose, hemicellulose, pectins, gum, and mucilages, which are biosynthesized from pentose, hexose, and uronic acid, and lignin built from phenylpropane units such as cinnamyl and coumaryl alcohols (Han et al., 2017).

Particle Size Distribution

The particle size of ADF30 and ADF200 after alkaline treatment was distributed in a range from 58.50 µm to 83.90 µm with a mean particle size of 71.20 µm (**Table 3**). However, the mean particle size of corncob powder before alkaline treatment (WA) is 77.43 µm. The results reveal that ADF30 can effectively reduce the particles size by using alkaline treatment but no occur with ADF200. This is probably due to agglomeration in the smaller particle size in ADF200. D_{50} is considered as the average median diameter which is representative of the degree of powder cohesiveness (Zhang et al., 2012).

Table 1. Effect of alkaline treatment on the chemical composition of fiber from corncob.

Sample	Cellulose (%)	Hemicellulose (%)	Lignin (%)
WA	41.57±0,39^{a}	38.84±0.05^{a}	11.96±0.55^{a}
ADF30	62.24±4,14^{b}	25.32±0,86^{b}	2.94±0.64^{b}
ADF200	66.57±2.26^{c}	15.78±0.05^{c}	5.64±0.73^{c}

$^{a-c}$ Means with different letters are significantly different ($p < 0.05$). WA: without alkaline; ADF30: 30 mesh sieve with alkaline treatment, ADF200: 200 mesh sieve with alkaline treatment.

Table 2. Effect of alkaline treatment on the content of dietary fiber^{a}

Sample	IDF (%)	SDF (%)	TDF (%
WA	57.89±0,13^{a}	1.43±0.05^{a}	59.32±0.08^{a}
ADF30	67.88±0,05^{b}	2.01±0,01^{b}	69.89±0.06^{b}
ADF200	72.77±0.02^{c}	2.72±0.01^{c}	75.49±0.01^{c}

$^{a-c}$ Means with different letters are significantly different ($p < 0.05$). Abbreviations as in **Table 1**.

The results of the particle size showed that 10% of the alkaline treatment and un-treatment of ADF30, ADF200 and corncob were 144.27, 237.64, and 169.85 µm, respectively. Based on the span value of the three samples, it shows that the span value of WA and ADF30 are smaller than ADF200. The small span value indicates that BA and DF30 have a more uniform particle size compared to ADF200 and a narrower particle size distribution which was might be to due particles have agglomerated even more. Ma et al., 2016) revealed that IDF after the reduce particle size had a higher span value, indicating that the IDF in a larger particle size distribution. The results of this study suggest that functional properties of the insoluble DF is not only influenced by the particle size, and alkaline but also by the proximate composition, overall charge density, surface properties and hydrophobic state (Ye., 2015).

FTIR Spectrum

FTIR spectrum of IDF prepared by alkaline treatment and untreatment are presented in Figure 1. The results showed that ADF30, ADF200 and WA having similar characteristic absorption peaks at 3425-3433 cm^{-1} (O-H stretching vibration of hydroxyl group) and 2900 cm^{-1} (C-H stretching vibration of methyl and methylene), which exhibited the presence of the typical structure of cellulose component. The characteristic peak at 1720 cm^{-1} is related to the vibrational range of the carbonyl group of the ester (hemicellulose) group at ADF200 and untreated but not observed at ADF30, while the peak at 1635 cm^{-1} belongs to the aromatic benzene in lignin observed in all three samples (Liu et al., 2016). The result is supported in the absorption region between 1300-1200 cm^{-1} and 830 to 750 cm^{-1} which indicated a vibrations aromatic framework from lignin (Zhao et al., 2013; Liu et al., 2017). This is indicated by the peak of 1257 cm^{-1} (untreated) with a strong intensity, but not found at ADF200 and ADF30. In three samples observed the strong band at have 1056 cm^{-1} and 1026 cm^{-1} is assigned to C-O stretching in cellulose, hemicelluloses, and lignin or C-O-C stretching in cellulose and hemicelluloses. In addition, a small band at 894 cm^{-1} in the spectrum of ADF200, ADF30 and WA corresponded to the glycosidic C-H deformation with a ring vibrations contribution for and OH bending, which was indicative of the β-glycosidic linkages between glucose in cellulose (Liu et al., 2006). Compared with FTIR spectra of ADF200, the characteristic absorption peak of ADF200 shifted from 3425 cm^{-1} to 3433 cm^{-1}. The shift indicated that the alkaline treatment could break the intramolecular hydrogen bonds, which might be attributed to cellulose degradation.

X-ray diffraction

Figure shows the X-ray diffraction curves of the alkaline treatment of DF30, DF200 and without alkaline treatment (WA). As shown in **Figure** the diffractogram ADF30 shows prominent 2θ peaks at around 15.90° and 22.32° while ADF200 is around 15.74° and 22.41°. This data are believed to represent the characteristics of cellulose crystals (Chen et al., 2011). These results also did not show any transformation of the crystalline structure of cellulose with alkaline or untreated treatment. The cellulose crystalline product in WA, ADF30, and ADF200 contains 29.33, 53.53, and 59.62% (**Figure 3**). The crystallinity values of all alkaline treatments samples were higher than that of untreated (WA) corncob.

The cristalinity of ADF30 and ADF200 produced with alkaline treatment increased by 2 folders compared to without alkaline process. However, the result was lower than that of rice straw (1.5 M NaOH) which has crystallinity of about 64.33% (He et al., 2008). Lower crystallinity means higher amorphous regions, which are more accessible to chemicals and water. This is probably due to the degradation of partial amorphous regions of lignin and hemicellulose. Several studies also reported that a relatively higher crystallinity of the cellulosic preparations sugarcane bagasse fiber that treatments of the delignified with higher concentrations of alkali and made a contribution to a decrease in the crystallinity of the cellulose (Liu et al., 2006). Besides, the fact is that some components such as lignin and hemicellulose are still present in the amorphous regions of the corncob fibers which must be removed (Chen et al., 2011). According to Qi et al. (2015)

Table 3. Particles size distribution of IDF before and after the alkaline treatment of DF[a].

Particles size	Sample		
	WA	ADF30	ADF200
$D_{0.1}$ (µm)	9,23	22,36	35,99
$D_{0.5}$ (µm)	77,43	58,50	83,90
$D_{0.9}$ (µm)	169,85	144,27	237,64
Span[a] (µm)	2,07	2,08	2,40

[a]Span= $(D_{0.9} - D_{0,1})/D_{0,5}$. WA: without alkaline; Abbreviations as in **Table 1**

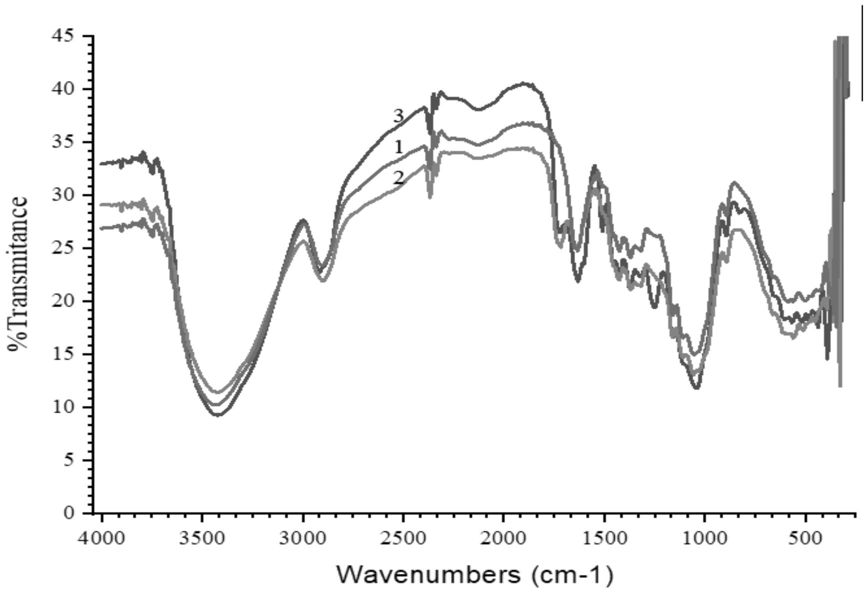

Figure 1. FT-IR Spectra of IDF before and after the alkaline treatment of dietary fiber. 1. WA, 2. ADF30: and 3. ADF200. Abbreviations as in **Table 1**.

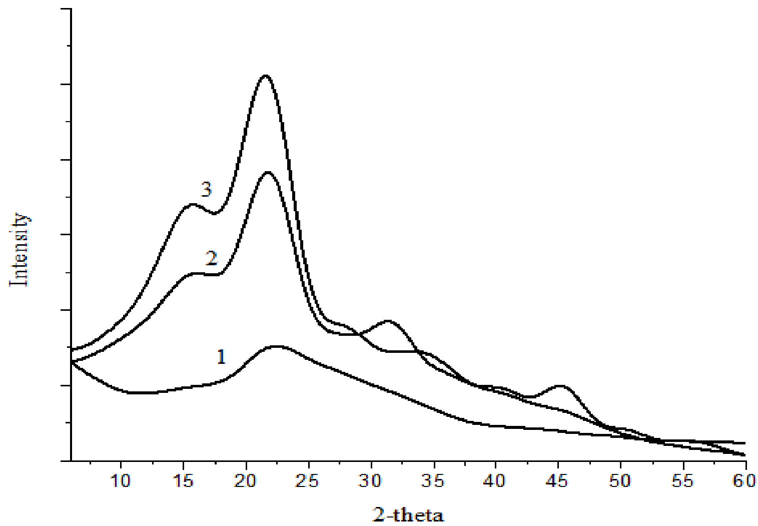

Figure 2. X-ray diffraction spectra of dietary fiber before and after the alkaline process. 1. WA, 2. ADF30: and 3. ADF200. Abbreviations as in **Table 1.**

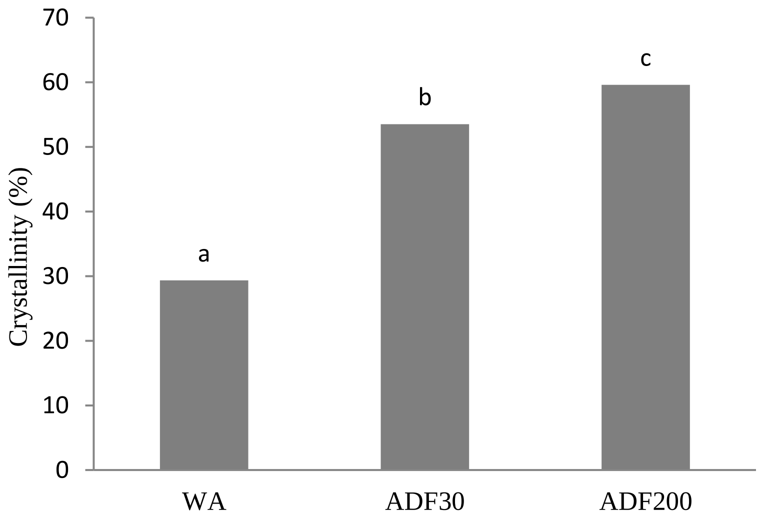

Figure 3. % crystallinity of dietary fiber before and after the alkaline process. Abbreviations as in **Table 1.**

that lower crystallinity might increase the water holding capacity (WHC) and swelling water capacity (SWC) of fiber, which could increase the transit time of the food stuff in the small intestine, and decrease the cholesterol availability in the small intestine. Hutomo et al. (2012) revealed that the high crystallinity can be causing to increase polarity, and low crystallinity causing to low polarity, so that the binding ability of oil or water is greatly influenced by the crystallinity properties of cellulose.

Scanning Electron Microscopy

The surface microstructure of before and after pretreatment were examined by scanning electron microscopy (SEM). **Figure 4,** shows the differences in the microstructure of corncob powder before and after treatment using alkaline. Before alkaline treatment, the surface of the corncob powder (WA) consisted of compact chunks form with small hole, indicating that it was not degraded lignin content. In addition, SEM images of WA observed the presence of porosity structure with a greater number of cracks at magnifications of 500x. The effect of alkaline treatment on ADF200 structure was similar to ADF30. The corncob powder with alkaline treatment was applied to remove the outer surface layer and more holes and larger size than before treatment. It might be due to the bonding lignin has degraded polysaccharides in corncob fiber, which in containing cellulose and hemicellulose. The larger surface the area of the chunk means the greater surface area and the surface chemistry also change during alkaline treatment so that has the best ability binding to compare with without alkaline treatment. The change of microstructures of ADF30 and ADF200 was related with the matrix structure of fiber, which might have an influence on the particle size, the physicochemical and functional properties of IDF from corncob.

Total phenolic content

Total phenolic content of composite flour was determined by using Folin-Ciocalteu reagent. The mechanism is based on the reduction of phosphomolibdate-phosphotungstate complex in Folin-Ciocalteu reagent by phenolic compound contained in sample. The complex is then turned into molybdenum, which can be identified qualitatively by the formation of blue color and thus detectable with a spectrophotometer at 750 nm. According to Li et al. (2012), all phenolic substances including simple phenols can react with Folin-Ciocalteu reagent. The phenolic total content in a corncob dietary fiber before and after alkaline are shown in **Figure 5**.

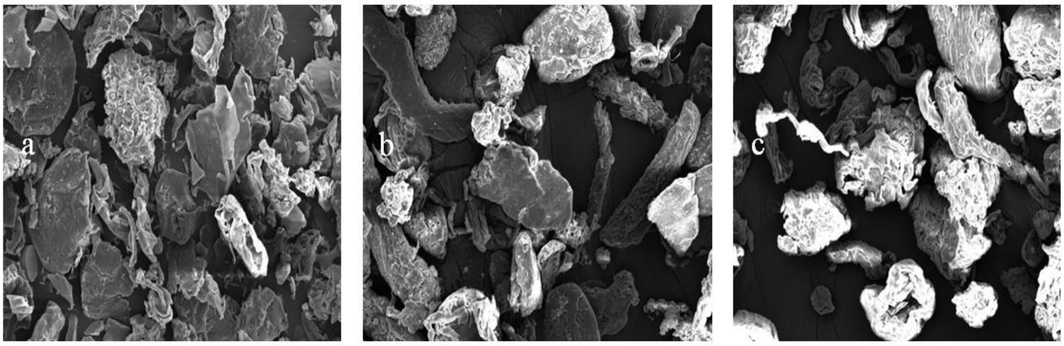

Figure 4. SEM images of dietary fiber before and after the alkaline process at 500x magnification. a. WA, b. ADF30 and c. ADF200. Abbreviations as in **Table 1**.

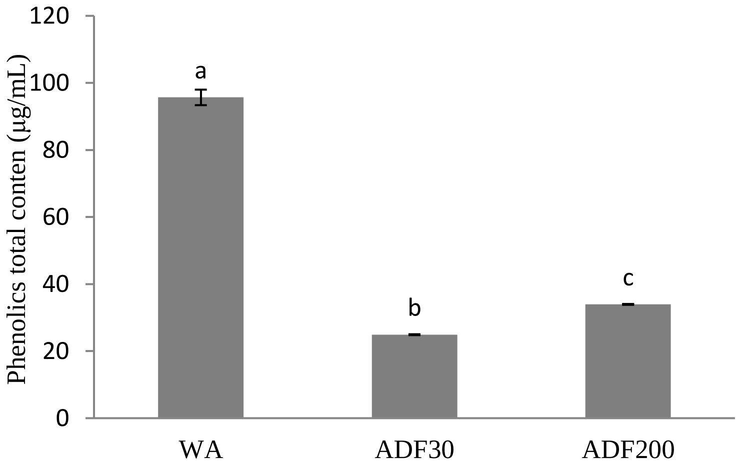

Figure 5. Phenolic total contents in corncob dietary fiber extracts before and after alkaline. Abbreviations as in **Table 1**.

The result showed that total phenolic of samples ADF30 and ADF200 after alkaline were significantly decreased 3.42 and 2.82 times, respectively, when compared with raw material before alkaline (WA) (p<0,05). These decreased are related to the content of lignin and hemicellulose (**Table 1**). This might be due to degradation of the two components in corncob as a consequence of alkaline treatment.

These results are supported that several studies reported that lignocellulose material that is extracted with alkaline containing phenolic compounds such as p-hydroxybenzoic acid, phydroxybenzaldehyde acid, vanillic acud, syringic acid, vanillin, syringaldehide, p-coumaric acid and ferulic acid (Xu et al., 2006). Therefore, the decreased hemicellulose content can also cause low phenolic content. This may be due to the phenolic components bound to the hemicellulose framework being extracted by the alkaline solution. The phenollic compounds may contribute to overall antioxidant activity.

DPPH Radical Scavenging Activity

DPPH radical scavenging activity in a corncob dietary fiber before and after alkaline are presented in **Figure 5**. Corncob in after alkaline at particle size of ADF200 was exhibited stronger anti radical activity than particle size of ADF30. However, free radical scavenging activity of ADF30 and ADF200 was lower than corncob extract before alkaline (WA). As shown in Figure 5, the free radical scavenging of ADF30, ADF200 and WA extract were 49.61, 59.72, and 83.41%, respectively. All extracts in corncob after alkaline showed antioxidant activity bigger than a high level of 50%. The DPPH radical scavenging activity was decreased after alkaline extraction. One possible explanation is that due to its lower phenolic compounds in ADF30 and ADF200, both phenolic extracts accumulated in the alkaline solution of the corncob extract. This data was an agreement with lignin and hemicellulose content that a tendency in the descending order and followed in lower total phenolic in both corncobs extract after alkaline of ADF200 and ADF30. A probable explanation is that may related with lignin and phenolic content from ADF30 and ADF200 that attributed to the degradation of lignin and causing some phenolic compounds linked in matrix to be released as a consequence of alkaline treatment.

The phenolic compounds may contribute to overall antioxidant activities. Therefore, phenolic present in a corncob after alkaline are good electron donors and could react with free radicals by converting free radical to more stable products. Besides, the result shows there was positive relationship between the amounts to total phenolic compounds in corncob after alkaline with DPPH radical scavenging activity. Therefore, the present of phenolic compounds is in dietary fiber corncob extracts can increase antioxidant activity furthermore terminate in free radical chain reaction. In conclusion, the antioxidant activities were related to the total phenolic content. During the process of alkaline treatment, the DPPH radical scavenging activity showed in descending, when compared with raw material (WA). These decreased are related to degradation of lignin and hemicellose and released phenolic compounds in the matrix of polysaccharide as a consequence of alkaline treatment.

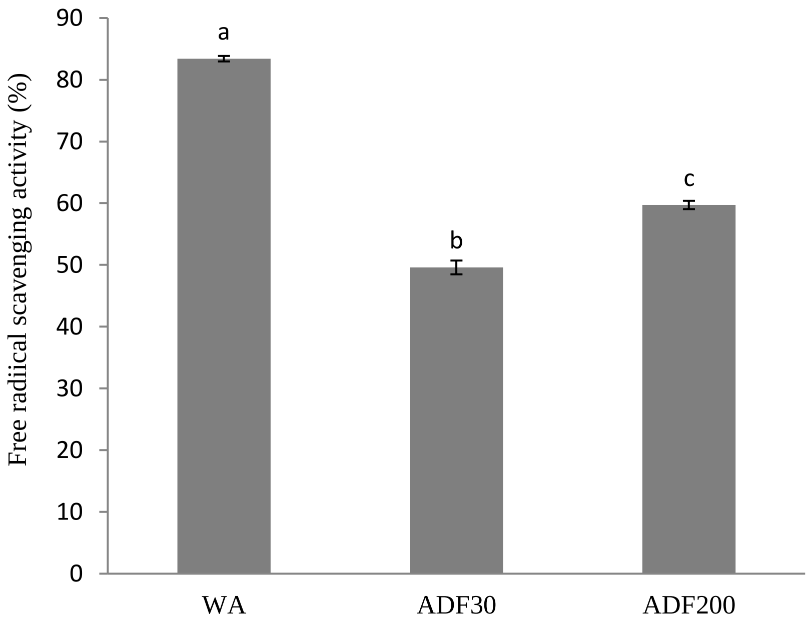

Figure 6. DPPH free radical scavenging in corncob dietary fiber extracts before and after alkaline. Abbreviations as in **Table 1**.

Similar result is shown that catechin, epicatechin, or procyanidin dimers were degraded by 85% at pH 7.4 after 24 h and by 100% at pH 9.0 after 4 h (Zhu et al., 2002). Accordingly, the result agrees with the found that alkaline pretreatment disrupts the lignin structure and degrades the associated between lignin and the other hemicellulose fractions in lignocellulosic biomass, thus making the hemicellulose contained minor quantities of lignin and its phenolics composition such as ferulic and *p*-coumaric acid (Xu et al., 2006). In addition to the use of an alkali cause the degradation of ester and glycosidic side chains resulting in structural alteration of lignin and partial solvation of hemicellulose and remove to 93.76% of hemicellulose and 86.14% of lignin, whereas cellulose remains largely undissolved. (Brodeur et al., 2011; Qu et al., 2017). Despite these result reported that the process may be destroyed or modified phenolic compounds at alkaline pH. However, few data exist on the detailed physicochemical and functional properties, and dietary fiber content of corncob powders as candidate for dietary fiber containing phytochemical antioxidant.

CONCLUSIONS

The results of the research showed that the alkaline process could effectively break the corncob IDF of leading to increases in content of a soluble and insoluble dietary fiber. FTIR analysis showed that no change observed in the functional groups of macromolecular, but the positions of some peaks with different size particles were different. The changes in chemical compositions, chemical structure, and physicochemical characteristics attributed of cellulose, hemicellulose and lignin during alkaline treatment were responsible for the improved and enhanced insoluble and soluble dietary fiber production from corncob. Moreover, corncob before alkaline has high antioxidant activity while alkaline process could reduce the total phenolic content, and DPPH radical scavenging activity in corncob dietary fiber. There is a potential for exploitation of corncob dietary fiber containing antioxidant a candidate for developed as a functional ingredient in food health.

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