Effect of different solvents and extraction conditions on fiber contents of local corns silk powders

Haslina\textsuperscript{1,2}, D Praseptiangga\textsuperscript{3}, V P Bintoro\textsuperscript{4}, and B Pujiasmanto\textsuperscript{5}

\textsuperscript{1} Doctoral Program of Agricultural Sciences, Graduate School of Sebelas Maret University (UNS), Jl. Ir. Sutami 36 A, Kentingan, Surakarta 57126, Central Java, Indonesia
\textsuperscript{2} Faculty of Agriculture Technology, Semarang University, Jl. Soekarno Hatta, Semarang 50196, Central Java, Indonesia
\textsuperscript{3} Department of Food Science and Technology, Faculty of Agriculture, Sebelas Maret University (UNS), Jl. Ir. Sutami 36 A Kentingan, Surakarta 57126, Central Java, Indonesia
\textsuperscript{4} Department of Agriculture, Faculty of Animal and Agricultural Sciences, Diponegoro University, Jl. Prof. Soedarto, SH, Tembalang, Semarang 50275, Central Java, Indonesia
\textsuperscript{5} Department of Agrotechnology, Faculty of Agriculture, Sebelas Maret University (UNS), Jl. Ir. Sutami 36 A Kentingan, Surakarta 57126, Central Java, Indonesia

E-mail: chana_panca@yahoo.com

Abstract. The aim of this study was to optimize the different solvents and extraction conditions on fiber contents of local cornsilk powders. Each sample of cornsilk powder extract was analyzed by enzymatic method for fiber content including soluble fiber, insoluble fiber and total fiber. Design using Factorial Randomized Block Design (RBD) with 4 factors are local varieties of cornsilk (Bisma, Arjuna and Srikanthi Putih), different solvents (water, methanol, ethanol and ethyl acetate), different concentration of solvents (methanol (60%, 70% and 80%), ethanol (60%, 70% and 80%), ethyl acetate (60%, 70% and 80%) and water), and ratio of material and solvent (1:5 (w/v), 1:10 (w/v), 1:15 (w/v) and 1:20 (w/v)). Response Surface Methodology (RSM) technique for optimum condition determination was used. Results showed that the difference in experimental conditions affects the content of soluble fiber, insoluble fiber, and total fiber produced by optimization analysis using the DX11.0\textsuperscript{®} Program with RSM-Central Composite Design (CCD). In conclusion, Bisma variety, solvent of methanol, concentration of 80% solvent and ratio of (1:20) was selected as the optimum condition of which resulted in soluble fiber content of 0.85%, insoluble fiber of 0.60% and total fiber of 1.45%.

1. Introduction
The average consumption of dietary fiber Indonesia’s population is 10.5 g/day. This number shows that people of Indonesia only meet the needs of fiber around a third of the ideal requirement of 30 g per day [1]. Fiber consumption is not related to where the population lives, but rather to the issue of economic status and knowledge. Another factor that influences is the availability of fibrous food and eating patterns and habits [2]. Important component in food ingredients is fiber which functions...
primarily in maintaining the health and balance of the function of the digestive system. Dietary fiber has an important health value in reducing the accumulation of cholesterol in the blood, improving glucose absorption for people with diabetes mellitus, preventing colon cancer, and helping to lose weight [3, 4]. In Indonesia, currently dietary fiber is still dominated by material from land plants because it is relatively cheap and easily obtained, while the utilization of materials derived from aquatic plants is still limited. Therefore, dietary fiber is not only derived from vegetable ingredients such as cellulose, hemi cellulose, pectin and lignin, but also includes polysaccharides of animal and microbes (aminopolysaccharides) such as chitin, chitosan, keratin sulfate, hyaluronic acid, chondroitin, xanthan gum, dextran and synthetic carbohydrates such as polydecstrose, methyl cellulose and hydroxypropylmethyl cellulose [5,6].

Corn silk is waste that is quite rich in dietary fiber with the characteristic form of set of smooth, soft stigma, it looks like thread or yellowish hair. Corn silk comes from female flowers from corn plants [7]. The chemical content of corn silk such as protein, carbohydrates, fiber of several vitamins such as vitamin B, vitamin C, vitamin K and essential oils. Mineral salts such as Na, Fe, Si, Zn, K, Ca, Mg and P. Phytochemical compounds such as sitosterol and stigmasterol, hasperidine derivatives and quercetin [8,9], contain phenols, terpenoids and glycosides [10]. Besides that, corn silk also contains maysin, β carotene, beta sitosterol, geraniol, hordenin, limonen, menthol and viteskin [11]. Corn silk is rich in phenolic compounds, especially flavonoids [12]. Local varieties of corn silk Bisma Indonesia have water content of 11.58%, fat content of 0.30%, ash content of 3.29%, protein content of 17.70% and carbohydrate content of 67.13%, and total phenol 8262.93 µgGAE/g, total flavonoids 236.03 µgGAEg, beta sitosterol 1343.93 ppm and 75% antioxidant activity. This result is different from previous researche [13].

The extraction process of corn silk powder is influenced by the extraction technique, extraction time, temperature, type of solvent, solvent concentration and the ratio of the solvent. Comparison of solvent materials, the number of extractants involved in displacement determines the degree of concentration difference that is very important in the diffusion process which will affect the content of the compound.

2. Experimental

The local corn silk varieties used in this study (Bisma, Arjuna and Srikandi Putih) each have different characteristics, obtained from Dukuh Lawangan, Lempuyang Village, Candidroto Districts, Temanggung District. The chemicals used were water, ethanol, methanol and ethyl acetate. Food fiber testing was conducted at the Laboratory of Food Chemistry and Biochemistry, Faculty of Agricultural Technology, Gadjah Mada University, Yogyakarta. Briefly, 3 g of fresh corn silk was washed with distilled water (aquadest) then it was dried in an oven at 60°C for 24 hours [14] until the water content was 10-11%. Further, it was pounded into powder using a vacuum grinder, packed and stored under -20°C until analysis. Corn silk was extracted using a modification method [15]. 3 g of corn silk powder were mixed with 30 ml of 80% methanol, then they were extracted at 70°C with a water bath shaker. The 1.5 hours later, they were filtered out the pulp using Whatman No. 1 paper. Solvent separation with rotary flash evaporator. Chemical analyses (soluble fiber, insoluble fiber and total fiber) were carried out using enzymatic methods [16]. The experimental design was factorial block randomized design with 4 factors and 2 replications. Local corn silk with different varieties (Bisma, Arjuna and Srikandi Putih) was extracted with different solvents (water, methanol, ethanol and ethyl acetate) with a solvent concentration of water, methanol (60%, 70% and 80%), ethanol (60 %, 70% and 80%) and ethyl acetate (60%, 70% and 80%) with a ratio (material:solvent) 1:5 (w/v), 1:10 (w/v), 1:15 (w/v) and 1:20 (w/v). The results were processed using Modde software 5 with Response Surface Methodology (RSM) program to determine the optimum concentration of corn silk powder extract.
3. Results and discussion

3.1. Soluble fiber
The content of soluble fiber in extracts of corn silk powder can be seen at the graph contour plot of Figure 1.

Table 1 shows that, the lowest mean soluble fiber was found in a combination of White Srikandi treatment, water solvent, 60% solvent concentration and with a ratio (1:5) is 0.06%, while the highest average soluble fiber was found in Bisma treatment combination, methanol solvent, 80% solvent concentration and with a ratio (1:20) is 0.85%. Design Expert 11.0 program optimization results at figure 1 in blue shows the lowest soluble fiber response value, which is 0.2%. The green color shows the highest soluble fiber response, which is 0.5%. This is presumably because the component of soluble fiber in corn silk is hydrolyzed, causing the release of pectin and oligosaccharides contained in the cell wall of corn silk [17].

3.2. Insoluble fiber
Insoluble fiber (insoluble in water) consists of carbohydrates that contain cellulose, hemicellulose and non carbohydrates that contain lignin. The results in table 1 shows that, the highest average insoluble fiber was found in Bisma treatment combination, methanol solvent, 80% solvent concentration with a ratio (1:20) is 0.60%. Figure 2 shows how the combination between components influences the response value of the insoluble fiber. The dark blue color shows the lowest insoluble fiber response
value, which is 0.1%. The light blue color shows the highest insoluble fiber response, which is 0.2%. Lines consisting of dots on the contour plot graph show combination of the four different components that produce the same insoluble fiber response. Corn plants are known to contain cellulose, the main hemicellulose in the first cell wall of vegetables are xyloglucan and gluconoxylan, whereas in the second cell wall is gluconoxylan [18]. The branch chain in xyloglucans is easier to hydrolyze than the main chain, while glucuronoxilane can be hydrolyzed in an acid medium in the glycosidic bonds between xylose. In addition, hemicellulose has a great ability to bind water molecules [19]. The insoluble fiber content is lower than soluble fiber, it is thought that because the cellulose cell wall in corn silk is degraded, the cellulose cell wall will be hydrolyzed to monosaccharide [20].

**Table 1. Description of the chemical content of the fiber**

| Treatment* | Soluble fiber (%) | Insoluble fiber (%) | Total fiber (%) |
|------------|-------------------|---------------------|---------------|
| A1B2C3D4  | 0.46              | 0.15                | 0.61          |
| A1B2C3D2  | 0.56              | 0.24                | 0.80          |
| A1B2C3D3  | 0.67              | 0.33                | 0.99          |
| A1B2C3D1  | 0.71              | 0.42                | 1.13          |
| A1B2C3D2  | 0.56              | 0.24                | 0.79          |
| A1B2C3D3  | 0.67              | 0.41                | 1.08          |
| A1B2C3D1  | 0.77              | 0.49                | 1.25          |
| A1B2C3D2  | 0.85              | 0.60                | 1.45          |
| A1B2C3D1  | 0.54              | 0.24                | 0.78          |
| A1B2C3D2  | 0.66              | 0.32                | 0.97          |
| A1B2C3D1  | 0.20              | 0.08                | 0.28          |
| A1B2C3D2  | 0.26              | 0.12                | 0.38          |
| A1B2C3D3  | 0.32              | 0.18                | 0.49          |
| A1B2C3D4  | 0.41              | 0.25                | 0.66          |
| A2B1C3D1  | 0.16              | 0.08                | 0.24          |
| A2B1C3D2  | 0.24              | 0.12                | 0.36          |
| A2B1C3D3  | 0.36              | 0.21                | 0.56          |
| A2B1C3D4  | 0.42              | 0.27                | 0.69          |
| A2B1C3D1  | 0.21              | 0.14                | 0.34          |
| A2B1C3D2  | 0.34              | 0.17                | 0.51          |
| A2B1C3D3  | 0.41              | 0.20                | 0.61          |
| A2B1C3D4  | 0.48              | 0.25                | 0.73          |
| A2B1C3D1  | 0.12              | 0.05                | 0.17          |
| A2B1C3D2  | 0.19              | 0.08                | 0.27          |
| A2B1C3D3  | 0.25              | 0.14                | 0.39          |
| A2B1C3D4  | 0.32              | 0.20                | 0.52          |
| A2B1C3D1  | 0.08              | 0.02                | 0.10          |
| A2B1C3D2  | 0.12              | 0.06                | 0.18          |
| A2B1C3D3  | 0.18              | 0.10                | 0.28          |
| A2B1C3D4  | 0.23              | 0.14                | 0.37          |
| A2B1C3D1  | 0.12              | 0.05                | 0.17          |
| A2B1C3D2  | 0.18              | 0.10                | 0.28          |
| A2B1C3D3  | 0.25              | 0.15                | 0.40          |
| A2B1C3D4  | 0.33              | 0.20                | 0.53          |
| A2B1C3D1  | 0.18              | 0.10                | 0.28          |
### Table 1: Treatment and Solvent Concentration on Total Fiber Response

| Treatment | Soluble fiber (%) | Insoluble fiber (%) | Total fiber (%) |
|-----------|-------------------|---------------------|-----------------|
| A_1B_2C_2D_2 | 0.25              | 0.14                | 0.39            |
| A_1B_2C_3D_1 | 0.35              | 0.18                | 0.52            |
| A_1B_2C_3D_2 | 0.41              | 0.22                | 0.63            |
| A_1B_2C_3D_3 | 0.10              | 0.03                | 0.13            |
| A_1B_2C_3D_4 | 0.13              | 0.08                | 0.21            |
| A_1B_2C_4D_1 | 0.20              | 0.12                | 0.32            |
| A_1B_2C_4D_2 | 0.26              | 0.16                | 0.42            |
| A_1B_2C_4D_3 | 0.06              | 0.01                | 0.07            |
| A_1B_2C_4D_4 | 0.10              | 0.07                | 0.17            |
| A_1B_3C_3D_3 | 0.16              | 0.10                | 0.26            |
| A_1B_3C_4D_4 | 0.21              | 0.12                | 0.33            |

* Description:

- Varieties: A_1: Bisma, A_2: Arjuna, A_3: Srikandi Putih
- Solvent: B_1: Ethanol, B_2: Methanol, B_3: Ethyl acetate, B_4: Water
- Solvent Concentration: C_1: 60%, C_2: 70%, C_3: 80%
- Ratio (material:solvent): D_1: (1:5), D_2: (1:10), D_3: (1:15), D_4: (1:20)

### 3.3. Total Fiber

The highest average total fiber in table 1 was found in Bisma treatment combination, methanol solvent, 80% solvent concentration with a ratio (1:20) is 1.45%. The dark blue color shows the lowest total fiber response value, which is 0.3%. The green color shows the highest total fiber response, which is 0.7% (Figure 3). This is presumably due to the total fiber content in the high corn silk extract powder. Soluble fiber content, insoluble fiber content and total fiber results of this study were lower than those carried out by [21] which reported levels of soluble food fiber 2.06% db, insoluble food fiber 15.83% db and total dietary fiber 17.89% db. Phenol components in cereal seeds (including rice) are strongly bound to wall-walled materials such as arabinoxilan and lignin which are components of dietary fiber. There are different locations and environments [22].

Regression coefficients in each response have no significant differences with \( p=0.05 \). The results of the quadratic equation show no significant difference for all factors with Lack of Fit values \( >0.05 \) (soluble fiber 0.30; insoluble fiber 0.83 and total fiber 0.05). The value of the coefficient of determination \( (R^2) \) for all factors in the equation model \( >0.05 \).

![Figure 5. Total fiber contour plot](image1)

![Figure 6. Total fiber 3d surface](image2)
Table 2. Components of optimized responses, targets, limits and interests in the formula optimization stage

| Response Name     | Goal          | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|-------------------|---------------|-------------|-------------|--------------|--------------|------------|
| A: Solvent conc.  | is in range   | 60          | 80          | 1            | 1            | 3          |
| B: Varieties      | is in range   | Bisma       | White Srikandi | 1            | 1            | 3          |
| C: Solvent        | is in range   | Etanol      | Air         | 1            | 1            | 3          |
| D: Ratio          | is in range   | 5           | 20          | 1            | 1            | 3          |
| Soluble fiber     | Maximize      | 0.06        | 0.85        | 1            | 1            | 5          |
| Insoluble fiber   | Maximize      | 0.01        | 0.6         | 1            | 1            | 5          |
| Total fiber       | Maximize      | 0.07        | 1.45        | 1            | 1            | 5          |

Table 2 shows the optimized components, targets, minimum and maximum limits, and the level of importance in the formula optimization stage. The response of soluble fiber, insoluble and total fiber is an optimized component with a maximum target with a level of importance of 5 (+++++). Soluble fiber, insoluble and total fiber is a response that greatly determines the efficiency of a business process, its relationship with economic value that will provide benefits. Whereas varieties, solvents, solvent concentrations and ratios were optimized with a range with a level of importance 3 (+++). Varieties, solvents, solvent concentrations and ratios will affect the quality of corn silk produced.

4. Conclusion
There was an interaction between varieties, solvents, solvent concentration, and the ratio to fiber content with a desirability value 0.717 which produced an optimal experimental formula on bisma varieties, methanol solvent, 80% concentration and ratio (1:20). In this condition, it produced chemical content (soluble fiber is 0.85%, insoluble fiber is 0.60% and total fiber is 1.45%).

References
[1] Astawan M and Wresdiyati T 2004 Diet Sehat dengan Makanan Berserat (Surakarta: Tiga Serangkai)
[2] Soerjodibroto 2004 Asupan Serat Makanan Remaja di Jakarta vol 54 No 10 Oktober 2004 (Jakarta: Majalah Kedokteran Indonesia) 397-401
[3] Jones P J, Raeini-Sarjaz M, Jenkins D J A, Kendall C W C, Vidgen E, Trautwein E A, Lapsley K G, Marchie A, Cunnane S C, and Connelly P W 2005 Lipids. 40(2) 169-174
[4] Wisten A and Messner T 2005 J. Carring Sci. 19 71-76
[5] Furda I 2001 Chitin and Chitosan, a Special Class of Dietary Fiber (CRC Handbook of Dietary Fiber in Human Nutrition 3rd ed ed Spiller GA CRC Press) 45-47
[6] Gallaher D D, Gallaher C M, Mahrt G J, Carr T P, Hollingshead C H, Hesslink Jr R H, and Wise J 2002 J. Am Coll Nutr. 21(5) 428-433
[7] Bhaigyabati T, Ramya J, and Usha K 2012 IRJP. 3(3)
[8] Ebrahimzadeh M A, Pourmoroad F, and Bekhradnia A R 2008 African Journal of Biotechnology. 7(18) 3188-3192
[9] Guo J, Liu T, Han L, and Liu Y 2009 Journal Nutrition and Metabolism Biomed Central. 6 47
[10] Sholihah M A, Nurhanan A R, and Rosli W I W 2012 International Food Research Journal. 19 (4) 1533-1538
[11] Rahmayani A 2007 Telaah Kandungan Kimia Rambut Jagung (Zea mays L) (Bogor: Institut Pertanian Bogor)
[12] Liu J, Wang C, Wang Z, Zhang C, Lu S, and Liu 2011 Food Chemistry. 126 261-269
[13] Haslina, Praspiptangga D, Bintoro V P and Pujiasmanto B 2017 International Journal of Advanced Science Engineering Information Technology. 7 1957-1963
[14] Hu Q L, Zhang L J, Li Y N, Ding Y J, and Li F L 2010 International Journal of Physical Sciences. 5 321-326
[15] Nurhanan A R, Rosli W I W, and Mohsin S S J 2012 Sain Malysiana. 40(2) 155-161
[16] Association of Official Analytical Chemist (AOAC) 2007 Official method 980.17 preservatives in ground beef spectrophotometric method (USA: AOAC International)
[17] Eveline, Antonius H C and Juanita R W 2014 ISBN 976-602-99334-3-7
[18] Yuanita L 2006. Indo J. Chem. 6(3) 332-333
[19] Sjostrom E 1993 Wood Chemistry Fundamentals and Applications Second Edition (New York: Academic Press)
[20] Yoon K Y, Cha M, Shin S R, and Kim K S 2005 Journal Food Chemistry. 92 151-157
[21] Damayanthy E and Listyorini D I 2006 Jurnal Gizi dan Pangan. 1(2) 34-44
[22] Guo W and Beta T 2013 Food Research International. 51 518-525