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PROCESS OPTIMIZATION FOR DIETARY FIBER PRODUCTION FROM CASSAVA PULP USING ACID TREATMENT

– Research paper –

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Abstract: Cassava pulp, the side product of tapioca industry consists of starch and fiber as the major component. Acid treatment was employed in the conversion process of cassava pulp into dietary fiber to remove the starch component, to increase fiber content, and to modify the structure of fiber. This study purposed to obtain optimum process conditions (acid concentration, temperature, and reaction time) in the production of dietary fiber from cassava pulp. Process optimization was conducted using Response Surface Methodology (RSM) for maximizing Total Dietary Fiber (TDF), Water Holding Capacity (WHC) and Oil Holding Capacity (OHC) as the responses. The optimum process was gained at 6% H_2SO_4 concentration, 127°C, and 45 mins. Prediction values of TDF, WHC, and OHC were 100%, 10.47 g/g, and 3.60 g/g, respectively. Validation was carried out and resulted in TDF 96.95%, WHC 10.47 g/g, and OHC 3.55 g/g. Physicochemical properties of the resulting dietary fiber were significantly improved. The fiber structure has modified which characterized by the changes in morphology and crystallinity.

Keywords: cassava pulp, dietary fiber, optimization, acid treatment, physicochemical properties

INTRODUCTION

Cassava pulp is a typical solid residue of tapioca production which is abundant in Indonesia but it has not been processed and utilized optimally. In 2013, the amount of cassava processed to tapioca was about 7.8 million tons in Indonesia (CDMI, 2014). It generated 780,000-1,170,000 tons dry cassava pulp and increased every year. Due to its large amount, cassava pulp will become a pollution if it is not handled or processed further. Recently, the utilization of cassava pulp in Indonesia is still limited as fermentation substrate, animal feed component, mosquito coil raw material as well as sauce raw material and included as byproducts with low economic value.

Cassava pulp contains 55.5% starch and 35.2% total dietary fiber (Chaikaew et al., 2012). With its ample fiber content, cassava pulp is very potential to be used as a fount of dietary fiber. In addition, another advantage is that cassava pulp has very low-fat content; it is around 0.12% (Sriroth et al., 2000) to 1% (Chaikaew et al., 2012), so it can avoid the problem of rancidity during storage. Dietary fiber is the polysaccharide that can not be digested in the intestine but it can be metabolized by bacteria that are located and through the digestive tract (AACC, 2001). Dietary fiber has good health implications for preventing chronic illnesses such as colon cancer, diabetes mellitus, and heart illness (Lv et al., 2017). The competitiveness of fiber-fortified food product markets makes the exploration of alternative sources of dietary fiber, especially from byproducts of food processing industries become important (Elleuch et al., 2010). Recently, Lv et al. (2017) have studied dietary fiber from the core of maize straw.

Cassava pulp should be treated in a certain way in order to have functional properties as dietary fiber. Preparation of dietary fiber from cassava pulp can be carried out with enzymatic or acid treatment. The treatment is performed to reduce starch component from about 60% to 15% (maximum), and to increase TDF from about 30% to 70% (minimum) (Lacourse et al., 1994). The weakness of the enzymatic method is that the slurry concentration of cassava pulp is very low, only 5-10% w/v (Lacourse et al., 1994) so the process is inefficient. At high substrate

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concentrations, the slurry of cassava pulp becomes very thick at its gelatinizing temperature and this is not proper to the use/action of the α-amylase enzyme (Srikanta et al., 1987). The use of cassava pulp substrate at high concentration (20-50%) in the hydrolysis process can be performed using an acid catalyst (Ahmed et al., 1983; Srikanta et al., 1987). The acid treatment is expected to modify the fiber structure to improve its functional properties. In addition, acid catalyst requires a relatively cheaper cost.

In this study, the effects of acid (H_2SO_4) concentration, temperature and time of reaction on the Total Dietary Fiber (TDF), Water Holding Capacity (WHC), and Oil Holding Capacity (OHC) were investigated. Process optimization was conducted in order to maximize TDF, WHC, and OHC. In addition, chemical composition, morphological structure, crystallinity and physicochemical properties of cassava pulp and obtained dietary fiber were also analyzed. Process optimization was carried out using Response Surface Methodology (RSM). RSM is an efficacious statistical method to obtain optimal process variables that improve the responses (Fan et al., 2008). The experiment was arranged using the central composite design (CCD). The advantage of CCD is that the number of runs of experiments is fewer but accurate data can be generated for optimization so the cost and time are more efficient than other designs (Anderson and Whitcomb, 2005).

MATERIALS AND METHODS

Material: Cassava pulp was obtained from CV Semangat Jaya, medium-scale tapioca industry in Lampung Province, Indonesia. Sulfuric acid (H_2SO_4), NaOH and other chemicals for analysis were purchased from Merck.

Raw material preparation: Cassava pulp output from tapioca industry extractor (± 85% moisture content) was squeezed using screw press until ± 50% moisture content and dried with sun drying in order to have ≤13% moisture content. The dried cassava pulp was milled and sieved with 40 mesh size.

Dietary fiber preparation: Preparation of acidified water was performed by adding H_2SO_4 (ρ: 1.835 g/mL) at a certain volume (according to the experimental concentration) into the water (666 mL) in 2 L tank. Furthermore, the slurry was prepared by adding dry cassava pulp (particle size 40 mesh) of 200 g into the acidified water. The reaction was carried out in the autoclave at the temperature and reaction time according to the experimental design. After the reaction completed, the slurry was filtered using a filter cloth and separated from the hydrolyzate (sugar syrup). pH of cake (fiber) obtained was increased to pH 6 by adding NaOH 5%w/w, then washed and re-filtered. The fiber obtained was dried at temperature 60°C until moisture content being relatively constant. The dried dietary fiber was milled and sieved at 100 mesh size.

Optimization of dietary fiber production process: The experimental design was arranged using CCD with 3 factors as independent variables: sulfuric acid concentration, temperature and time of reaction. The fixed variable chosen was cassava pulp slurry concentration (30%w/v). The levels of the independent variable are reaction temperature (lower level 107°C; upper level 127°C), reaction time (lower level 15 minutes; upper level 45 minutes), and concentration of H_2SO_4 to the weight of cassava pulp substrate (lower level 2%v/w; upper level 6%v/w). The function of this optimization process was to maximize TDF, WHC, and OHC.

Statistical analysis of the data: Experimental design arrangement and data processing were done using Design-Expert® software 7.0.0. The response surface and contour plot were used to show the effect of the factors on the responses obtained. The coefficients on the empirical model were estimated using multi-regression analysis. The significance of the empirical model obtained was tested using Analysis of Variance. Optimization was performed using numerical optimization.

Validation: It was carried out using the value of independent variables obtained from the results of numerical optimization. Furthermore, the actual response value was compared to the prediction response value.

Chemical composition analysis: Soluble Dietary Fiber (SDF) and Insoluble Dietary Fiber (IDF) were determined by the enzyme-gravimetric method described by AOAC Official Methods 993.19 and 991.42. TDF was calculated

by adding SDF and IDF. Moisture, protein, fat, and ash content were determined using the method previously explained by AOAC (1995). Starch content was measured by the enzymatic method, through determination of glucose obtained from the saccharification using enzyme of α-amylase and amyloglucosidase (AMG). Glucose was measured using the Spectroquant® UV-Vis pharo300 spectrophotometer using the dinitrosalicylic acid (DNS) method.

Morphology: The morphology of cassava pulp and dietary fiber obtained were analyzed by scanning electron microscopy (EVO MA 10, Carl Zeiss, Germany) at 1500x magnification. The sample surface was coated with a thin gold layer at 60 second sputter time and 20 mA sputter current. Samples were fed into the SEM tool and the surface image was taken using the Secondary Electron (SE) detector, working distance (WD) 8-9 mm and EHT 12.0 kV.

Crystallinity: The X-Ray Diffractogram and crystallinity of the material were analyzed using X-Ray Diffraction. Measurements were performed at 2Ɵ: 5-45° intervals, at 40 kV, 30 mA, and speed of 2° per min.

Physicochemical properties analysis: Water Holding Capacity (WHC) was measured using the procedure previously described by Suzuki et al. (1996) with minor modification. A total of 200 mg of sample was placed into a centrifuge tube, then 20 ml of distilled water was added. The tube was incubated for 24 h in the water bath shaker, then centrifugation at 3,000 x g for 60 min was performed. The supernatant was removed and the water content of the pre-weighed pellet was measured by drying using an oven at 120°C for 2 h. WHC of the sample was stated as the weight of water held by one gram of sample.

Oil Holding Capacity (OHC) measurement was conducted using the method previously described by Caprez et al. (1986). Approximately 2 g of sample was placed in a 50 ml centrifuge tube, then 20 ml of corn oil was added. The tube is left for 30 minutes at room temperature with agitation. Then, centrifugation (2,500 x g, at room temperature for 30 mins) was performed and the oil supernatant was poured and weighed. The OHC of the sample was stated as the gram of oil retained by one gram of sample.

Cation-Exchange Capacity (CEC) was analyzed according to Moorman et al. (1983). Emulsifying Activity (EA) was determined according to Yasutmasu et al. (1972). Viscosity was determined using viscometer. Swelling Capacity (SC) was performed according to Gómez-Ordóñez et al. (2010). Solubility measurement was performed using the method previously described by Paton and Spratt, (1984).

RESULTS AND DISCUSSIONS

Chemical composition of cassava pulp

In this study, the chemical composition (dry basis) of cassava pulp consisted of 69.76% of starch, 27.40% of TDF (25.25% of IDF and 2.15% of SDF), 1.37% of ash, 2.03% of protein and 0.07% of fat. Similar observations were carried out by (Sriroth et al., 2000; Rattanachomsri et al., 2009; Chaikaew et al., 2012; Virunanon et al., 2013; Zhang et al., 2016), which reported that cassava pulp consisted of major components of starch and fiber. There was a small amount of protein, ash, and fat. Cassava pulp generated in high quantities as a side-product of tapioca industry is a potential source of biomass (Vaithanomsat et al., 2016).

The starch component of cassava pulp has a larger portion than the fiber component, while dietary fibers are components of non-starch carbohydrates. It needs an appropriate conversion to remove/decrease the starch component and increase the TDF. Acid treatment could be employed to hydrolyze starch into sugar and to modify the structure of the fiber from the crystalline structure to amorphous structure.

Optimization process by RSM

RSM is a statistical technique that used to obtain the best empirical model and problem analysis on responses that are influenced by several variables and aims to obtain optimum responses (Montgomery, 2008). From each of the predetermined levels of the factors, an experimental design constructed with CCD was arranged using Design-Expert® 7.0.0 software and shown in Table 1. TDF was selected as a chemical quantitative response, whereas WHC and OHC are selected as the responses that represent the quality (physicochemical properties) of dietary fiber products.

Table 1. Experimental design and the values of observed responses

Std order	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
	A: Concentration of H_2SO_4 (%v/b)	B: Temperature (°C)	C: Time of Reaction (mins)	TDF %	WHC (g/g)	OHC (g/g)
1	2.00	107.00	15.00	72.07	6.74	2.11
2	6.00	107.00	15.00	73.15	7.33	2.12
3	2.00	127.00	15.00	85.09	10.43	2.89
4	6.00	127.00	15.00	94.20	10,.0	3.58
5	2.00	107.00	45.00	72.45	8.08	2.25
6	6.00	107.00	45.00	73.70	8.06	2.45
7	2.00	127.00	45.00	96.68	10.30	3.22
8	6.00	127.00	45.00	97.19	10.35	3.69
9	0.64	117.00	30.00	57.99	6.11	1.83
10	7.36	117.00	30.00	85.15	8.46	2.99
11	4.00	100.18	30.00	73.01	7.32	2.56
12	4.00	133.82	30.00	94.25	11.18	3.11
13	4.00	117.00	4.77	74.08	7.75	2.37
14	4.00	117.00	55.23	97.73	8.38	3.53
15	4.00	117.00	30.00	76.42	8.56	2.69
16	4.00	117.00	30.00	74.64	7.85	2.63
17	4.00	117.00	30.00	78.28	8.93	2.64
18	4.00	117.00	30.00	76.21	7.97	2.94
19	4.00	117.00	30.00	76.38	9.26	2.94
20	4.00	117.00	30.00	75.94	8.67	2.61

ANOVA of TDF response model indicates that TDF is a function of H_2SO_4 concentration (A), temperature (B) and reaction time (C). By applying multiple regression analysis, it was obtained the regression equation for the quadratic model as follows:

Total Dietary Fiber = $76.23 + 4.22A + 8.60B + 4.05C + 0.91AB – 1.05AC + 1.71BC – 1.16A^2 + 3.10B^2 + 3.91C^2$ (1)

The quadratic model p-value of 0.0033 indicates a significant model. There is only a possible 0.33% error in drawing conclusions. p-value less than 0.0500 indicates components of model are significant. In this case, A, B, C, and C^2 are significant model components to the TDF response. The values of R^2 (0.857) and R^2adj (0.7283) indicate that the ability of the independent variable to explain the variance of the dependent variable is good (85.7% and 72.8% respectively). “Adequate precision” is a measure to compare predicted design point values to the predicted mean error values. The value of “adequate precision” must be greater than 4.0, meaning that the model has high precision (Kumari et al., 2008). “Adequate precision” value of 8.725 indicates an adequate signal that this model has a high precision and it is eligible for use.

The ANOVA of WHC response model shows that the WHC is a function of the concentration of H_2SO_4 (A), temperature (B) and reaction time (C). The obtained model is a regression equation for a linear model as follow:

Water Holding Capacity = $8.61 + 0.34A + 1.31B + 0.21C$ (2)

The linear model p-value of 0.0001 indicates a significant model. There is only a possible 0.01% error in drawing conclusions. In this case, B is a significant component, which means that the temperature is a variable that significantly affects the WHC response. From the "Lack of Fit test" of the model, it was obtained p-value 0.1589> 0.05 indicates that the “Lack of Fit” is not significant relative to the pure error. Non-significant “lack of fit” implies that the model is good. The values of R^2 (0.7227) and R^2adj (0.6707) indicate that the ability of the independent variable to explain the variance of the dependent variable is good enough (72.3% and 67.1% respectively). An “adequate precision” value of 12.571 (greater than 4) indicates a model with high precision is eligible for use.

The ANOVA of OHC response model shows that the OHC is a function of the concentration of H_2SO_4 (A), temperature (B) and reaction time (C). The obtained model is a regression equation for a linear model as follow:

Oil Holding Capacity = $2.76 + 0.24A + 0.39B + 0.21C$ (3)

The linear model p-value of 0.0001 indicates a significant model. There is only a possible 0.01% error in drawing conclusions. In this case, A, B, and C are the significant model components to the OHC response. From the "Lack of Fit test" of the model, it was obtained p-value 0.0505> 0.05 indicates that the “Lack of Fit” is not significant relative to the pure error. Non-significant “lack of fit” implies that the model is good. The values of R^2 (0.7203) and R^2adj (0.667) indicate that the ability of the independent variable to explain the variance of the dependent variable is good enough (72.03% and 66.7% respectively). An “adequate precision” value of 12.938 (greater than 4) indicates that this model has a high precision and eligible for use. Response surface of the TDF, WHC, and OHC and the effect of interaction between variables can be seen in Figures 1(a)-(i).

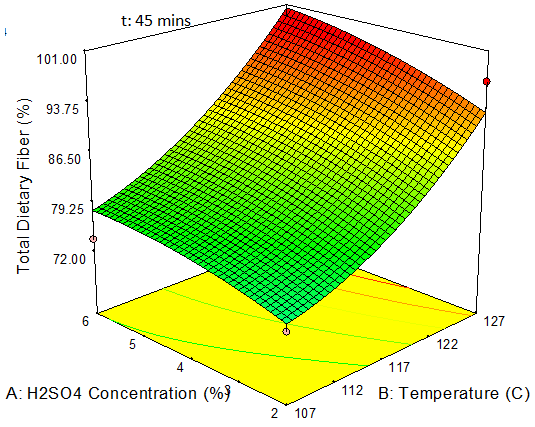

Figure 1(a). Response surface of TDF on the effect of H_2SO_4 concentration and temperature

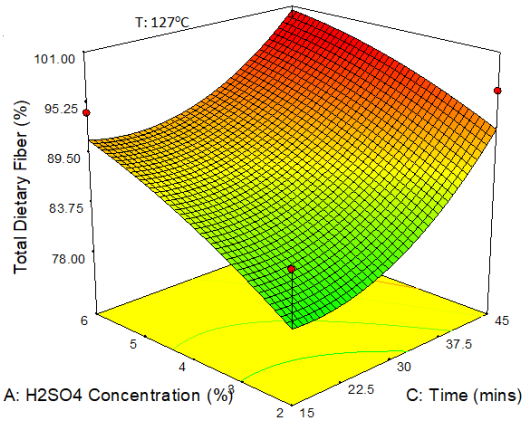

Figure 1(b). Response surface of TDF on the effect of H_2SO_4 concentration and time of reaction

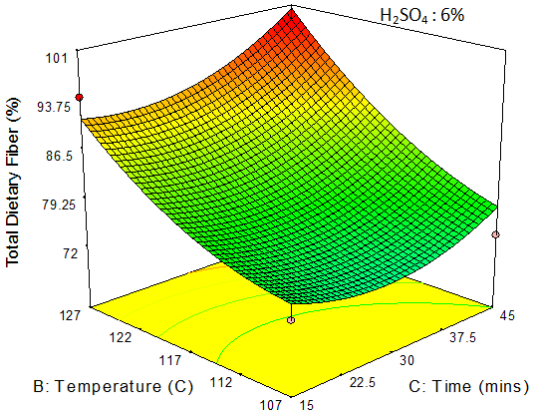

Figure 1(c). Response surface of TDF on the effect of temperature and time of reaction

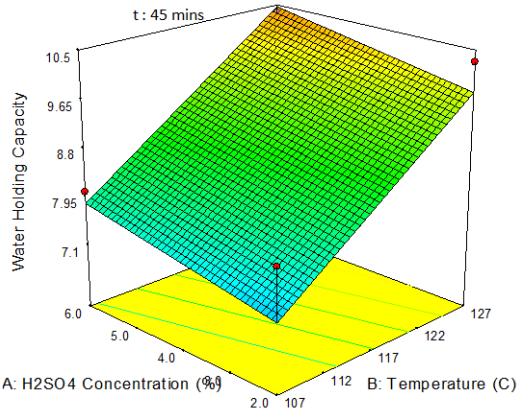

Figure 1(d). Response surface of WHC on the effect of H_2SO_4 concentration and temperature

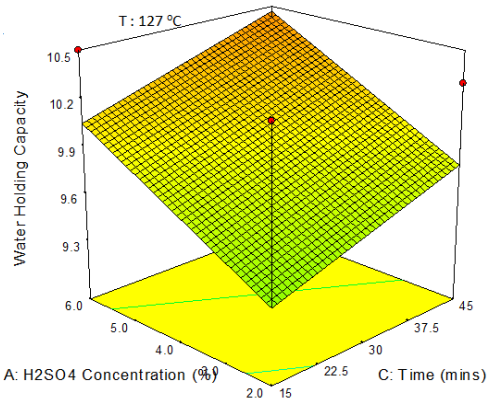

Figure 1(e). Response surface of WHC on the effect of H_2SO_4 concentration and time of reaction

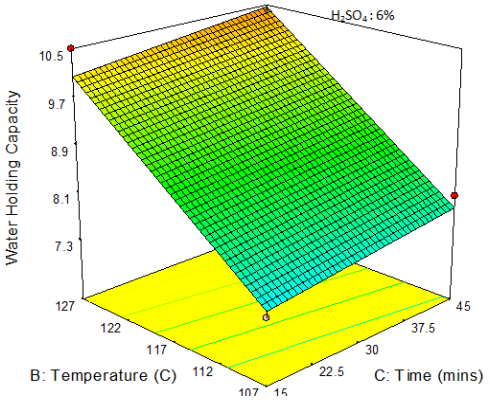

Figure 1(f). Response surface of WHC on the effect of temperature and time of reaction

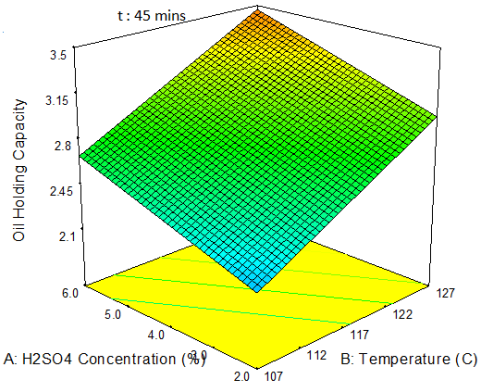

Figure 1(g). Response surface of OHC on the effect of H_2SO_4 concentration and temperature

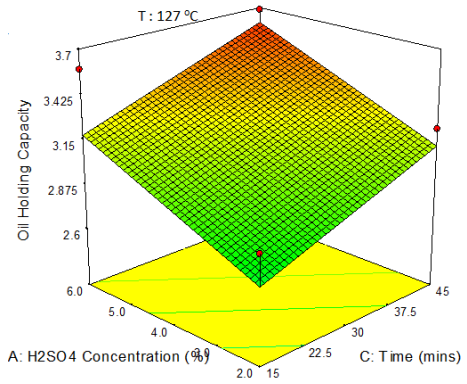

Figure 1(h). Response surface of OHC on the effect of H_2SO_4 concentration and time of reaction

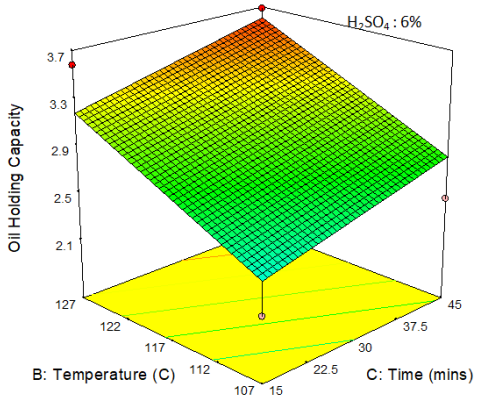

Figure 1(i). Response surface of OHC on the effect of temperature and time of reaction

Optimization was performed by setting the condition of independent variables within the experimental range and setting the TDF, WHC, and OHC at maximum conditions. Several optimization solutions were generated. From the solution provided by the program, the optimal solution has the maximum desirability value, in this case, 0.937 was selected as the best solution. The optimum process conditions were obtained at 6% H_2SO_4 concentration, 127°C temperature and reaction time 45 mins, with prediction response value of TDF 100%, WHC 10.47 g/g and OHC 3.6 g/g according to Figure 1.

Validation

Validation was performed at optimum process condition. The actual responses value were TDF 96.95%, WHC 10.47 g/g and OHC 3.55 g/g. The differences that occur between the predicted values and the actual values at optimum conditions were 3.1% for TDF, 0% for WHC and 1.4% for OHC. The optimization result was acceptable because the actual values of TDF, WHC, and OHC were in accordance with the model prediction. This was indicated by three responses value meet the 95% confidence intervals presented by the program.

Effects of acid concentration, temperature and reaction time on TDF, WHC, and OHC

Figure 1(a),(b),(c) show the interaction effects of two factors on the TDF. The concentration of H_2SO_4, temperature and reaction time had a significant effect on TDF response, where each variable gave positive effect to increase TDF in experimental range. TDF is a fiber component consisting of IDF (for instance cellulose, hemicellulose, and lignin) and SDF (pectin). The higher the temperature, the greater the TDF content of the product. This occurred because the rate of starch hydrolysis reaction in the cassava pulp also increased so that at the same time starch would be more hydrolyzed into sugar (Wahyudi et al., 2011). The hydrolysis reaction is an endothermic reaction that requires heat to react, corresponding to the Arrhenius equation:

$$k = Ae^{\frac{-EA}{RT}} \quad (4)$$

k = rate constant ; A = pre-exponential factor; EA= activation energy ($J.mol^{-1}$); R = the gas constant ($J.mol^{-1}.K^{-1}$); T = temperature (K).

In addition, the longer reaction time the higher TDF content of the product. Starch more hydrolysable into sugar during reaction time so the starch content in dietary fiber products became less. The similar trend also occurred in the variable of H_2SO_4 concentration. The higher

the concentration of H_2SO_4, the activation energy became smaller, and the reaction rate constant became larger (Artanti and Andik, 2006). While the reaction rate increased, the starch hydrolyzed into sugars became more. Then, TDF content increased when hydrolyzate (sugar syrup) separated from the fiber.

Figure 1(d),(e),(f) describe the interaction effects of the factors on the WHC. In this case, the temperature was a variable that significantly affected the WHC, whereas the H_2SO_4 concentration and reaction time had no significant effect on the WHC. The higher the temperature the WHC value increased. WHC optimum achieved at temperature 127^{o}C.

Figure 1(g),(h),(i) describe the interaction effects of the factors on the OHC. The concentration of H_2SO_4, temperature and time were the variables that significantly affected the OHC. There was a linear relationship between concentration of H_2SO_4, temperature and reaction time with OHC, where the increase of each variable would increase the OHC value. In the previous discussion, it was known that the increased concentration of H_2SO_4, temperature and reaction time would increase the TDF. The higher TDF content indicates that the fiber component which can absorb/retain oil will also increase. The more oil retained the higher the OHC value.

Chemical composition of dietary fiber products

The chemical composition of dietary fiber obtained from the optimization result was analyzed. Dietary fiber consisted of 2.7% starch, 96.95% of TDF (94.36% of IDF and 2.59% of SDF), and 1.3% of ash. The TDF content in dietary fiber product increased significantly from originally 27.4% in cassava pulp (raw material), while the starch content in dietary fiber decreased significantly from originally 69.76% in cassava pulp. This occurred because the starch component of cassava pulp was hydrolyzed by acid as a catalyst into hydrolyzate in the form of sugar syrup which was then separated as a byproduct.

Starch granules on the cassava pulp were trapped within the matrix by the fiber structure. To remove the starch granules, the acid through the hydrolysis process will cut the polysaccharide bonds, especially the starch with the α-1,4 glycosidic bonds into smaller molecules sequentially. The starch molecule initially breaks into shorter chains of glucose called a dextrin. Dextrin has broken down again into maltose and maltose and can then break down into glucose (Gaman and Sherrington, 1981). The process of acid hydrolysis will produce hydrolyzate (by-products) which can be separated from the fiber component in the cassava pulp. The hydrolyzate products need to be detoxified to eliminate toxic compounds such as furfural and HMF before used in applications in the industry. At high temperatures and pressures, the sugar from hydrolysis of starch and hemicellulose such as glucose, galactose, and mannose is degraded into HMF and xylose into furfural. HMF compounds can also be derived from browning (Maillard) reactions between amino groups derived from proteins in cassava pulp and glucose formed (Palmqvist and Hagerdal, 2000). Detoxification method using activated carbon is a good method with relatively lower costs (Mussatto and Roberto, 2001). The use of 5% activated carbon concentration could reduce HMF concentrations by 85% (Hodge et al., 2009).

Physicochemical functional properties

Physiological effects are correlated with the physicochemical properties of dietary fiber and its fractions (Marsono, 2004). Dietary fiber specific properties associated with their physiological effects include fermentability, water-binding capacity, organic molecular absorption, viscosity and ion exchange properties. Some physicochemical properties for this investigation are summarised in Table 2. Table 2 shows that there are significant improvements of cassava pulp physicochemical properties after conversion to dietary fiber through acid treatment. The dietary fiber obtained from optimization results has the highest WHC, followed by barley bran, wheat bran, and cassava pulp. Fiber especially IDF has the ability to hold the water (Marsono, 2004). WHC and swelling capacity values follow the trend of fiber content in the ingredients. It was found that dietary fiber had the highest TDF content (96.95%), followed by barley bran and wheat bran, 54.83% and 31.73% (Matin et al., 2013), respectively. WHC is a measure of the quantity of water held on to the fibers after undergoing pressures as centrifugation (Nelson, 2001). WHC properties need to be measured before the dietary fiber is used as a functional food. OHC is a physicochemical property of fiber material that can be applied in food formulations. The porosity of fiber structures is a major factor affecting the ability of fibers to retain oils (Nelson, 2001). In addition,

hydrophobicity, the number of lipophilic sites and capillary appeal are also complex factors affecting OHC (Kinsella, 1976). Materials that have high OHC and emulsifying activity are able to act as emulsifiers in formulated foods. The dietary fiber obtained from optimization results showed the highest OHC and emulsifying activity values compared to barley bran and wheat bran. The other important property of dietary fiber is ability as an ion exchanger. This property can be a negative characteristic of dietary fiber because it can reduce mineral availability and absorption of electrolytes (Marsono, 2004). According to Carvalho et al. (2009), sulfate content, uronic acid, and load density affect cation exchange capacity. The dietary fiber obtained has a lower cation exchange capacity compared to barley bran and wheat bran. On the other hand, viscosity of the fiber suspension related to the solubility, molecular weight, and structural chemical bonds of the material (Choct, 2002). The dietary fiber obtained from the optimization result showed lower viscosity than barley bran and wheat bran, due to the lower solubility and SDF content (2.59%). Gum and pectin are included in the SDF group. Many studies have shown that gum and pectin increase the viscosity of intestinal contents, whereas the IDF does not have that property, but this fiber accelerates intestinal emptying and transits throughout the intestine (Marsono, 2004). IDF raises the number of feces because it is resistant to bacterial degradation while the SDF is easily fermented by bacteria resulting in an increase of bacterial mass. The obtained dietary fibers have very high TDF content with good physicochemical properties so the product is suitable for use in food applications for low-calorie diets, processed meats, fat replacer, emulsifying agents and for the bakery industry. Dietary fiber is appropriate for reducing fat content in meat-based products in order to obtain healthier food. In addition, the use of dietary fiber in meat products can improve the texture profile and characteristics associated with the cooking process (Tufeanu and Tita, 2016).

Table 2. Physicochemical properties of cassava pulp, the dietary fiber obtained, wheat bran and barley bran

Properties	Cassava Pulp (Raw material)	Dietary Fiber (Product Obtained)	Wheat bran (Matin et al. 2013)	Barley bran (Matin et al. 2013)
Water Holding Capacity (g/g)	2.97	10.47	3.41	4.57
Oil Holding Capacity (g/g)	1.36	3.55	1.21	1.79
Cation-Exchange Capacity (meq/kg)	3.7	12.7	16.9	29.9
Emulsifying Activity (%)	57	77.89	32.84	52
Viscosity (cP)	0.3	0.35	1.74	2
Swelling Capacity (ml/g)	6.35	11.47	5.75	8
Solubility (%)	0.45	0.53	-	-

Morphology

The results of the morphological analysis of cassava pulp and dietary fiber products are shown in Figure 2. The results showed that the starch granules in cassava pulp were bound in a trapped position on the biomass fiber matrix (Figure 2a). Therefore, the grinding process can be carried out to minimize particle size and increase its surface area and to partially cut the fiber matrix wrapping the starch granules. It may increase access to hydrolyze starch granules (Hermiati, 2012). Cassava pulp underwent gradual degradation during acid and heat treatment as shown in SEM morphological image of cassava pulp after treatment (Fig. 2(b),(c),(d)). The biomass matrix was seen to be slightly degraded at 2% H_2SO_4 acid treatment; reaction temperature 107^{o}C; and a reaction time of 15 mins, possibly in the amorphous section. However, in this condition still visible that starch granules were bound to the fiber matrix. After acid treatment at 4% H_2SO_4 concentration; 117^{o}C; 30 minutes, the degradation process was more obvious, marked by the visible bundle of fibers that allegedly was the bundle of cellulose fibers. Cellulose fiber was the most resistant cassava pulp component to the degradation process, while the hemicellulosic component was more soluble and degraded (Hermiati, 2012). At optimum process conditions (6% H_2SO_4 concentration; temperature 127^{o}C; and a 45 mins reaction time), the biomass matrix degradation was very clear. This was characterized by the formation of fiber bundles with a regular structure. In this condition almost all starch was hydrolyzed and separated from the

fiber matrix, so that resulted in dietary fiber with a porous fiber structure. Microstructure (porosity, particle size, and fiber length) and processing conditions are important things that influence the physicochemical properties of dietary fiber related to water (Nelson, 2001).

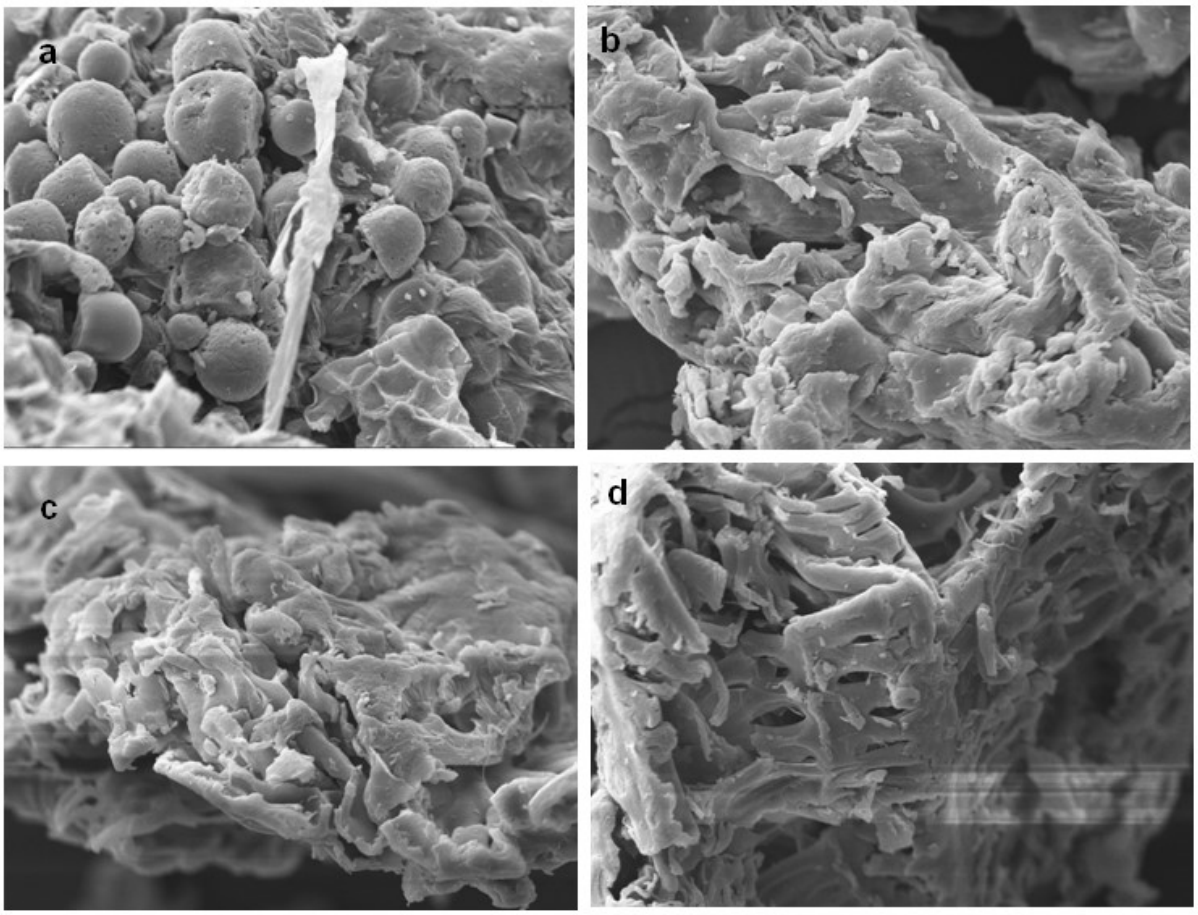

Figure 2. Scanning electron microscope image of : (a) untreated cassava pulp; (b) after treatment at 2% H_2SO_4; 107°C; 15 mins; (c) after treatment at 4% H_2SO_4; 117°C; 30 mins; (d) after treatment at 6% H_2SO_4; 127°C; 45 mins

Crystallinity

X-ray diffractogram of cassava pulp before and after treatment is shown in Figure 3. X-ray diffractogram of cassava pulp before treatment shows that cassava pulp has a high peak at 2Θ by 15°, 17°, 18° and 23°. This spectrum type is similar to the tapioca starch spectrum that having a strong peak at 15°, 17°, 18° and 23° as reported by Atichokudomchai et al. (2001). Starch and fiber are the main constituents of cassava pulp, each of which has crystallinity properties. The result of X-ray diffraction analysis shows that the crystallinity of cassava pulp used in this study was 45.13%. By treatment using 2% H_2SO_4; reaction temperature 107°C; and reaction time of 15 mins, the crystallinity of the cassava pulp decreased to 36.19% due to the hydrolysis of starch component. The spectrum under these conditions has the high peak at 21.4°, 22.4°, and 23.3°. In other conditions, 4% H_2SO_4 concentration; 117°C; 30 mins, crystallinity in the range 15° -18° decreased while the crystallinity in the range 21°- 23° increased. At optimum process conditions (6% H_2SO_4; 127°C; 45 minutes), crystallinity in the range 15° - 18° more decreased while the crystallinity in the range 21°- 23° more increased. It was possible that the starch component was almost completely hydrolyzed, so the contribution of crystallinity came from the fiber component. The spectrum under these conditions has the high peak at 21°, 21.8° and 22.4° with the crystallinity of 44.9%. By acid treatment, modification of the fiber structure was occurred and changed the crystalline structure into a more amorphous structure so as to increased its porosity and improved the physicochemical properties.

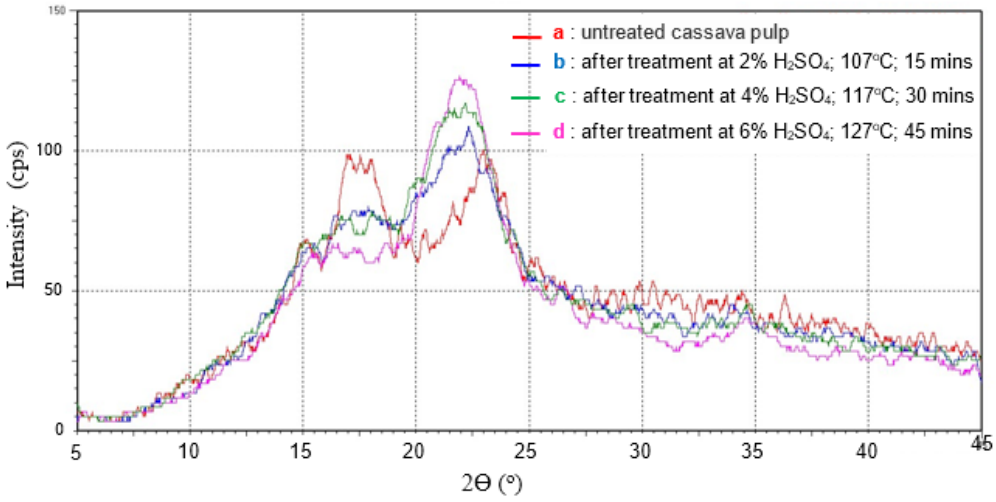

Figure 3. X-ray diffractogram of cassava pulp before and after treatment

CONCLUSIONS

The results of this study indicate that cassava pulp is a potential and valuable resource that can be converted to the dietary fiber using acid treatment. The acid treatment is effective for removing the starch component and increasing the TDF content. The optimum process conditions for the production of dietary fiber are at H_2SO_4 concentration 6% (v/w), temperature 127°C and reaction time 45 mins. Temperature is the variable that most influences the response of TDF, WHC and OHC. The results of this study also proved that the acid treatment can be used to modify the fiber structure and improve the physicochemical properties. Therefore, cassava pulp can be an excellent choice for the production of dietary fiber from biomass.

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REFERENCES

1. AACC. (2001). The definition of dietary fiber. *Cereal Foods World*. 46(3), 112–129.
2. Ahmed, S.Y., Ghildyal, N.P., Kunhi, A.A.M. & Lonsane, B.K. (1983). Confectioner's syrup from tapioca processing waste. *Starch/Stärke*. 35, 430–432.
3. Anderson, M.J. & Whitcomb, P.J. (2005). *RSM simplified – optimizing processes using response surface methods for design of experiments*. New York: Productivity Press.
4. AOAC. (1995). *Official Method of Analysis* (16th ed). Virginia: The Association of Official Agricultural Chemists.
5. Artati, E.K. & Andik, P.A. (2006). Effect of acid concentration on starch hydrolysis of banana. *Ekuilibrium*. 5(1), 8-12.
6. Atichokudomchai, N., Shobsngob, S., Chinachoti, P. & Varavinit, S. (2001). A study of some physicochemical properties of high-crystalline tapioca starch. *Starch/Stärke*. 53, 577-581.
7. Caprez, A., Arrigoni, E., Amado, R. & Neukom, H. (1986). Influence of different types of thermal treatment on the chemical composition and physical properties of wheat bran. *Journal of Cereal Science*. 4, 233-239. DOI: 10.1016/S0733-5210(86)80025-X.
8. Carvalho, A.F.U., Portela, M.C.C., Sousa, M.B., Martins, F.S., Rocha, F.C., Farias, D.F. & Feitosa, J.P.A. (2009). Physiological and physico-chemical characterization of dietary fibre from the green seaweed Ulva fasciata Delile. *Brazilian Journal of Biology*. 69(3), 969-977. DOI: 10.1590/s1519-69842009000400028.
9. CDMI. (2014). *Study of business potential and main actor of tapioca industry in Indonesia, 2015-2018*. Jakarta: PT. Central Data Mediatama Indonesia.
10. Chaikaew, S., Maeno, Y., Visessanguan, W., Ogura, K., Sugino, G., Lee, S.H. & Ishikawa, K. (2012). Application of thermophilic enzymes and water jet system to cassava pulp. *Bioresource Technology*. 126, 87-91. DOI: 10.1016/j.biortech.2012.09.020.

11. Choct M. (2002). Non-starch polyccharides: effect on nutritive value. In J.M. McNab & K.N. Boorman (Eds.), *Poultry Feedstuffs: supply, composition, and nutritive value* (pp. 221-237). Oxon: CABI Publishing.
12. Elleuch, M., Bedigian, D. & Roiseux O. (2010). Dietary fiber and fiber-rich by-products of food processing: characterization, technological functionality and commercial application: a review. *Food Chemistry.* 124, 411–421. DOI: 10.1016/j.foodchem.2010.06.077.
13. Fan, G., Han, Y., Gu, Z. & Chen, D. (2008). Optimization conditions for anthocyanins extraction from purple sweet potato using response surface methodology (RSM). *Food Science and Technology*. 41, 155-160. DOI: 10.1016/j.lwt.2007.01.019.
14. Gaman, P.M. & Sherrington, K.B. (1981). *The science of food* (2nd ed). New York: Pergamon Press. p:48-59.
15. Gómez-Ordóñez, E., Jiménez-Escrig, A. & Rupérez, P. (2010). Dietary fibre and physicochemical properties of several edible seaweeds from the northwestern Spanish coast. *Food Research International*. 43, 2289-2294. DOI: 10.1016/j.foodres.2010.08.005.
16. Hermiati, E. (2012). *Process engineering for cassava pulp hydrolysis using micro wave heating for ethanol production*. Doctoral disertation, Institut Pertanian Bogor, Bogor, Indonesia.
17. Hodge, D.B., Andersson, C., Berglund, K.A. & Rova, U. (2009). Detoxification requirements for bioconversion of softwood dilute acid hydrolyzates to succinic acid. *Enzyme and Microbial Technology.* 44, 309–316. DOI: 10.1016/j.enzmictec.2008.11.007.
18. Kinsella, L.E. (1976). Functional properties of protein in foods: A survey. *Journal of Food Science and Nutrition*. 7, 219-280. DOI: 10.1080/10408397609527208.
19. Kumari, K.S., Babu, I.S. & Rao, G.H. (2008). Process optimization for citric acid production from raw glycerol using response surface methodology. *Indian Journal of Biotechnology*. 7, 496-501. http://hdl.handle.net/123456789/2353.
20. Lacourse, N.L., Chicalo, K., Zallie, J.P. & Altieri P.A. (1994). U.S. Patent US5350593A. Wilmington: U.S. Patent and Trademark Office.
21. Lv, J-S., Liu, X-y., Zhang, X-p. & Wang, L-s. (2017). Chemical composition and functional characteristics of dietary fiber-rich powder obtained from core of maize straw. *Food Chemistry*. DOI: 10.1016/j.foodchem.2017.01.078.
22. Marsono, M.S. (2004). *Dietary fiber in perspective of nutrition science*. Yogyakarta: Universitas Gadjah Mada.
23. Matin, H.R.H., Shariatmadari, F. & Torshizi, M.A.K. (2013). Various physico-chemical properties of dietary fiber sources of poultry diets. *International journal of agriculture and crop sciences.* 6(18),1239-1245.
24. Montgomery, D. C. (2008). *Design and analysis of experiments* (7th ed). New Jersey: John Wiley & Sons.
25. Moorman, W.F.B., Moon, N.J. & Worthington, R.E. (1983). Physical properties of dietary fiber and binding of mutagens. *Journal of Food Science*. 48, 1010-1011. DOI: 10.1111/j.1365-2621.1983. tb14959.x.
26. Mussatto, S.I. & Roberto, I.C. (2001). Hydrolysate detoxification with activated charcoal for xylitol production by *Candida guilliermondii*. *Biotechnology Letters*. 23,1681-1684. DOI: 10.1023/A:1012492028646.
27. Nelson, A. L. (2001). *High-fiber ingredients: Eagan press handbook series*. St Paul, MN: Eagan Press.
28. Palmqvist, E. & Hagerdal, B.H. (2000). Fermentation of lignocellulosic hydrolysates. I: inhibition and detoxification. *Bioresource Technology*. 74, 17-24.
29. Paton, D. & Spratt, W. A. (1984). Component interactions in the extrusion cooking process conditions on the functional viscosity of the wheat flour system. *Journal of Food Science*. 49, 1380-1385. DOI: 10.1111/j.1365-2621.1984.tb14995.x.
30. Rattanachomsri, U., Tanapongpipat, S., Eurwilaichitr, L. & Champreda, V. (2009). Simultaneous non-thermal saccharification of cassava pulp by multi-enzyme activity and ethanol fermentation by Candida tropicalis. *Journal of Bioscience and Bio Engineering*. 107, 488-493. DOI: 10.1016/j.jbiosc.2009.06.012.
31. Srikanta, S., Jaleel, S.A., Ghildyal, N.P., Lonsane, B.K. & Karanth, N.G. (1987). Novel technique for saccharification of cassava fibrous waste for alcohol production. *Starch/Stärke.* 39, 234–237.

32. Sriroth, K., Chollakup, R., Chotineeranat, S., Piyachomkwan, K. & Oates, C.G. (2000). Processing of cassava waste for improved biomass utilization. *Bioresource Technology*. 71, 63-69. DOI: 10.1016/s0960-8524(99)00051-6.
33. Suzuki, T., Oshugi, Y., Yoshiem, Y., Shirai, T. & Hirano, T. (1996). Dietary fibre content, water holding capacity and binding capacity of seaweeds. *Journal of Fisheries Sciences*. 62, 445-446. DOI: 10.2331/fishsci.62.454.
34. Tufeanu, R. & Tița, O. (2016). Possibilities to develop low-fat products: a review. *Acta Universitatis Cibiniensis Series E: Food Technology.* 20(1), 3-19. DOI: 10.1515/aucft-2016-0001.
35. Vaithanomsat, P., Kosugi, A., Apiwatanapiwat, W., Thanapase, W., Waeonukul, R., Tachaapaikoon, C., Pason, P. & Moriet, Y. (2016). Efficient saccharification for non-treated cassava pulp by supplementation of Clostridium thermocellum cellulosome and Thermoanaerobacter brockii β-glucosidase. *Bioresource Technology*. 132, 383–386. DOI: 10.1016/j.biortech.2012.11.023.
36. Virunanon, C., Ouephanit, C., Burapatana, V. & Chulalaksananukul, W. (2013). Cassava pulp enzymatic hydrolysis process as a preliminary step in bio-alcohols production from waste starchy resources. *Journal of Cleaner Production*. 39, 273-279. DOI:10.1016/j.jclepro.2012.07.055.
37. Wahyudi, J., Wibowo, W.A., Rais, Y.A. & Kusumawardani, A. (2011). Effect of temperature on glucose levels formed and constants rate reaction on banana skin hydrolysis. Proceedings of the National Seminar on Chemical Engineering Kejuangan, 22 February 2011 (p.B09-1 –B09-5). Yogyakarta: Universitas Pembangunan Nasional.
38. Yasutmasu, K., Sawada, K., Moritaka, S., Nfisaki, M., Toda, J., Wada, T. & Ishi, K. (1972). Whipping and emulsifying properties of soybean products. *Agricultural and Biological Chemistry*. 36, 719–737. DOI: 10.1271/bbb1961.36.719.
39. Zhang, M., Xie, L., Yin, Z., Khanal, S.K. & Zhou, Q. (2016). Biorefinery approach for cassava-based industrial wastes: current status and opportunities. *Bioresource Technology*. 215, 50–62. DOI: 10.1016/j.biortech.2016.04.026.