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Integration of physical and chemical treatment on the extraction of starch from *Canna edulis* Ker. rhizome

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ABSTRACT

The extraction of *Canna edulis* Ker. starch from its rhizome was performed using 2 different types of press (hydraulic press and screw press) and with the addition of Na-metabisulphite and NaOH (in the range of concentration 100 - 5000 ppm each). The optimum condition for this process was determined by Central Composite Design of experiment and the statistical calculation was solved by Design-Expert 7.0.0. The targets of the observed responses were high starch yield, low ash, low fiber, and high carbohydrate content. The results showed that the starch yield and the reduction of fiber were only influenced by the physical treatment whereas ash content in the product was influenced by both the NaOH concentration and physical treatment. The carbohydrate content in the extraction product was affected by NaOH, by the interaction between the concentrations of NaOH and $Na_2S_2O_5$ and also by the physical treatment. The hydraulic press gives much better responses compared to the screw press. But in the selected range of additives concentrations, the screw press gives a higher starch yield (30% - 52%).

Keywords: *Canna edulis* Ker.; Central Composite Design of Experiment; Hydraulic Press; Screw Press; Starch Extraction

1. INTRODUCTION

One of the tropical starch resources which have not been utilized for industrial application is *Canna edulis* Ker. rhizome, partly because of the difficulties in the extraction processing [1]. The high content of fiber and other trace elements are a major constraint in producing pure starch. A sample of 100 g of *Canna edulis* rhizome contains 125 mg phosphorus (P), 84 mg calcium (Ca) and 1.5 mg iron (Fe) mineral [2]. The presence of those elements in food products can be categorized as a nutritional value but it will be a disadvantage for a chemical starch-based product.

Salt solutions like NaCl, Na-bisulphite, and Na-metabisulphite are commonly used during extraction of starch from its natural source, to inhibit microbial growth and deactivate plant enzyme (amylase). The salt solution can dissolve the surface starch granule protein as well, but for the breakdown of the integral starch granule protein, stronger solutions are required, e.g. sodium dodecyl sulphate (SDS) [3,4] or alkaline solutions [5].

Lim *et al*. [6] have investigated that the use of 0.2% of NaOH as an extraction solution for rice starch could reduce more than 80% of the flour protein. This was confirmed also by Radosavljevic *et al*. [7] on the extraction of Amaranth starch; Mistry *et al*. [8] on the extraction of corn flour using 0.1% and 0.4% of NaOH. It was investigated also that sodium hydroxide (NaOH) can remove phosphorus up to 70% - 90% from wheat starch [9].

Since the properties of starch are, to some extent, different from the fiber (cellulose), a relatively low cost method for isolation of the starch is physical treatment. The goal of the research reported here is to determine the optimum condition of the integration of physical and chemical treatments in producing pure starch. For this purpose, a Central Composite Experimental Design method (CCD) was applied, statistical calculations were made using Design expert 7.0.0 software.

2. MATERIALS & METHODS

2.1. Materials

Freshly harvested *Canna edulis* Ker. rhizome (locally known as Ganyong) was supplied by the farmers union "Mekar Sari" at the Kulon Progo region (Central Java) Indonesia. Analytical grade of sodium metabisulphite and NaOH were purchased from Sigma Aldrich. α-amylase was supplied by Novozyme and Anthrone reagent by Merck.

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2.2. Design of the Experiment

To determine the optimum condition from 2 numerical factors (the Na-metabisulphite and NaOH concentrations) and 1 categorial factor (type of mechanical treatment or type of press instrument), the Central Composite Design was applied. This results in 26 runs with variations as shown in **Table 1**.

2.3. Starch Extraction

Fresh *Canna edulis* was peeled and washed, then milled with a cross beater mill. The pulp was mixed with water at a weight ratio 1:10. Half of mixture was filtered with the hydraulic press and the rest with the screw press, to separate the crude fiber.

The free crude fiber mixture which consists of the filtrate from each press was divided into 13 portions that were mixed with different concentrations of Na-metabisulphite and NaOH solutions as stated **Table 1**. After 12 hours the precipitated starch was separated from the liquor and dried in a tray drier for about 12 hours at 45˚C until constant weight. The dried starch was kept in closed containers to be analyzed further.

Table 1. Overview of experimental runs.

	Factor 1	Factor 2	Factor 3
Run	[$Na_2S_2O_5$]	[NaOH]	Type of treatment*
	ppm	ppm	
1	2550	2550	Level 2
2	2550	2550	Level 2
3	100	100	Level 2
4	2550	2550	Level 1
5	2550	2550	Level 2
6	5000	100	Level 2
7	100	5000	Level 1
8	2550	2550	Level 2
9	5000	5000	Level 2
10	5000	5000	Level 1
11	2550	2550	Level 1
12	100	2550	Level 1
13	100	2550	Level 2
14	2550	2550	Level 1
15	5000	2550	Level 1
16	5000	2550	Level 2
17	2550	2550	Level 1
18	2550	2550	Level 1
19	2550	5000	Level 2
20	2550	2550	Level 2
21	100	5000	Level 2
22	100	100	Level 1
23	2550	100	Level 1
24	2550	5000	Level 1
25	2550	100	Level 2
26	5000	100	Level 1

*Level 1—hydraulic press and level 2—screw press.

2.4. Fiber Content

The fiber content was measured by heating a mixture of 5 g of starch and 50 mL of water until 90˚C. Then 4 mL of α-amylase enzyme was added and the total was kept at 90˚C for a further 30 minutes. After that 50 mL of water was added and the sample was cooled to room temperature.

This method was based on the characteristic property of the α-amylase enzyme which degrades carbohydrates to produce shorter chain molecules such as glucose, which is soluble in water. The cooled mixture was filtered using Whatman 42 filter paper under vacuum conditions to separate the fine fiber. The Fiber content (FC) was calculated using **Eq.1**.

$$\mathrm{FC}(\%) = \frac{w_1}{w_2} \times 100\% \quad (1)$$

Where w_1 is the weight of fine fiber and w_2 is the weight of starch sample (dry basis).

2.5. Ash Content

Ash content measurement was also based on a gravimetric technique. 1 g of starch sample was combusted in a furnace at 600˚C for 1 hour then cooled in desiccator and weight. This step was repeated until the weight was constant. Ash content (AC) was calculated using **Eq.2**.

$$\mathrm{AC}(\%) = \frac{w_3}{w_4} \times 100 \quad (2)$$

Where w_3 is the weight of ash and w_4 is the weight of starch sample (dry basis).

2.6. Carbohydrate Content

The carbohydrate content was analyzed by anthrone reagent [10]. The anthrone method was started by making a standard graph which was made by diluting 10 mg of glucose into 100 mL of water. The standards solution was taken 0, 0.2, 0.4, 0.6, 0.8 and 1 mL (0 serves as blank) and made up to 1 mL with distilled water. Then 4 mL of anthrone reagent was added.

Anthrone reagent was made by diluting 200 mg anthrone in 100 mL of ice-cold 95% H_2SO_4. All of the standard solutions were heated for eight minutes in a boiling water bath and then cooled rapidly. Carbohydrate content was measured with a spectrophotometer at 595 nm wavelength. A calibration graph was made by plotting concentrations of the standard solution on the X-axis against and the absorbance on the Y-axis.

Starch must be hydrolyzed prior to the treatment with anthrone solution. Therefore, 100 mg of a sample of starch with 5 mL of 2.5 N HCl added, was boiled in a water bath for three hours. When sample temperature

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was back to room temperature, it was neutralized with solid sodium carbonate (Na_2CO_3) until the effervescence ceases. The solution was then made up to 100 mL and centrifuged and 0.1 mL of the supernatant was used for analysis. The rest of the procedure is the same for the standard solution. The amount of carbohydrate (CC) in the sample tube was then calculated from the absorbance in the spectrophotometer and the calibration graph by **Eq.3**.

$$CC(\%) = \frac{0.9 \times [C] \times 1000}{w_5} \times 100 \quad (3)$$

where [C] is the glucose concentration measured from the calibration graph whereas w_5 is the weight of starch sample (dry basis).

2.7. Starch Yield

Starch yield was measured by comparing the weight of obtained starch (dry basis.) with the weight of dry matter sample (*Canna edulis* rhizome). The starch has been free from dirt 100. Starch Yield (SY) was determined by **Eq.4**.

$$SY(\%) = \frac{w_6}{w_7} \times 100 \quad (4)$$

where w_6 is the weight starch (d.b.) whereas w_7 is the weight of the dry matter original sample.

3. RESULTS AND DISCUSSION

The analysis results from every run can be seen in **Table 2**.

3.1. Starch Yield

Based on the ANOVA of the values obtained for the starch yield (**Table 3**) it can be seen that the additives concentrations, both $Na_2S_2O_5$ and NaOH do not affect the yield of starch. This is also proved by the probability value (P value) from both additives which are above 0.005. The yield obtained is apparently more determined by the method of physical treatment.

Between the two types of press used, the higher starch yield was found when the screw press was used for separation (30% - 52%) compared to the hydraulic press (25% - 41%). However, this fraction contains 79% ± 6% carbohydrate which is slightly lower than the product from the hydraulic press 82% ± 8%).

3.2. Carbohydrate Content

Based on the ANOVA of carbohydrate content (**Table 4**) it can be seen that the NaOH concentration and the interaction between the concentrations of $Na_2S_2O_5$-NaOH affect the amount of carbohydrate obtained. Since the p-value of BC ≈ 0.05, so it can be predicted that there is a relation between the physical treatment and the additive (NaOH).

From **Figure 1**, it can be seen that at the high concentration of $Na_2S_2O_5$ and the high concentration of NaOH, within the selected ranges of this research the carbohydrate content reached the maximum value.

However, if the 3D surface plot is observed for each physical treatment (**Figures 2** and **3**), the effect of concentration of additives on the carbohydrate content gives a different profile. The separation process using the screw

Table 2. Results of the experimental runs.

Run	Fiber %	Ash %	Carbohydrate %	Yield %
1	3.7	4.0	76.0	14.1
2	2.4	1.9	76.4	33.7
3	2.9	0.7	76.9	49.7
4	1.6	1.0	82.2	35.0
5	3.3	1.5	82.0	40.7
6	2.7	1.5	74.5	44.0
7	2.0	2.7	88.9	31.7
8	2.6	1.6	82.1	40.9
9	3.8	3.8	92.0	64.9
10	2.4	3.5	88.7	47.0
11	1.9	1.3	94.0	31.5
12	2.5	1.3	81.3	35.1
13	3.2	2.0	82.3	43.2
14	2.3	2.9	76.7	25.2
15	2.8	1.7	73.8	33.9
16	3.2	2.3	77.0	57.6
17	2.5	1.2	74.2	35.0
18	2.9	3.0	81.1	11.1
19	2.7	4.3	66.5	30.0
20	2.7	2.7	81.8	34.6
21	3.2	3.6	75.4	45.7
22	1.4	0.4	82.7	39.0
23	2.6	0.5	78.7	37.4
24	2.4	2.6	93.6	30.4
25	3.2	1.0	77.7	39.2
26	1.4	0.7	65.1	38.7

Table 3. ANOVA for starch yield response.

Factor	SS	DF	MS	F Value	p-Value
[$Na_2S_2O_5$]	144.47	1	144.47	3.38	0.18
[NaOH]	0.24	1	0.24	2	0.95
Treatment	442.65	1	442.65	3.37E-003	0.02

Table 4. ANOVA for carbohydrate content response.

Factor	SS	DF	MS	F Value	p-Value
A:[$Na_2S_2O_5$]	22.39	1	22.39	0.69	0.42
B:[NaOH]	204.67	1	204.67	6.28	0.02
Treatment	62.86	1	62.86	1.93	0.18
AB	165.91	1	165.91	5.09	0.04
BC	132.74	1	132.74	4.07	0.06

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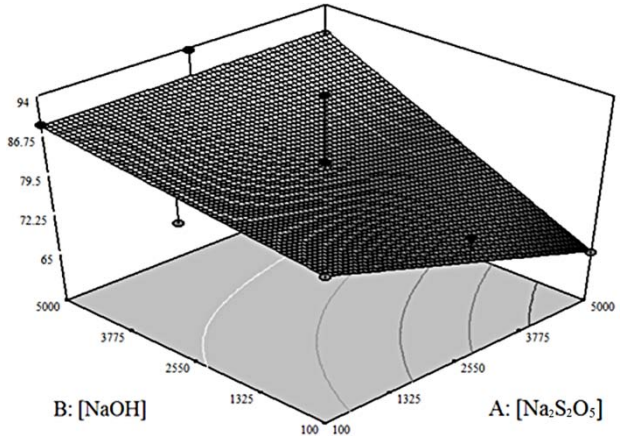

Figure 1. 3D plot of carbohydrate content.

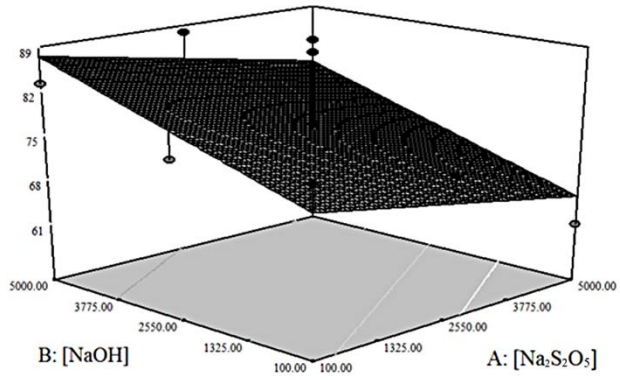

Figure 2. 3D surface of carbohydrate content using the hydraulic press.

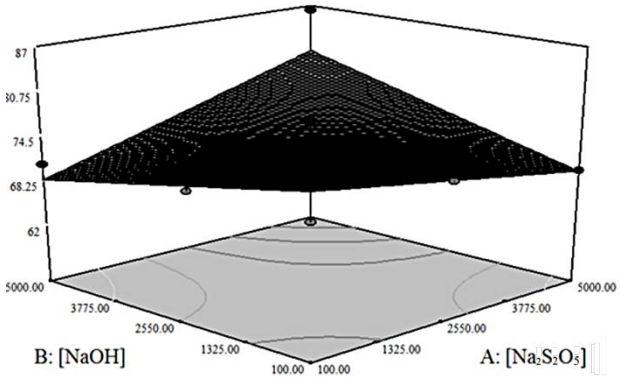

Figure 3. 3D surface of carbohydrate content using the screw press.

press is slower than the hydraulic press. This results in the need for the higher concentration of Na-metabisulphite (5000 ppm) to achieve high carbohydrate content as well as to prevent microbial growth in the slurry which can destroy the carbohydrate. This is a different result compared to the hydraulic press in which it is enough to use 100 ppm to gain a high carbohydrate yield.

The models resulting from the statistical methods illustrate the relation between the carbohydrate content and the significant factors as followed:

For the hydraulic press

$$CC = 83.2 - 3.7\times10^{-3}\left[Na_2S_2O_5\right] + 1.1\times10^{-3}\left[NaOH\right] + 7.6\times10^{-7}\left[Na_2S_2O_5\right]\left[NaOH\right] \quad (5)$$

For the screw press

$$CC = 81.1 - 1.3\times10^{-3}\left[Na_2S_2O_5\right] + 1.6\times10^{-3}\left[NaOH\right] + 7.6\times10^{-7}\left[Na_2S_2O_5\right]\left[NaOH\right] \quad (6)$$

3.3. Fiber Content

Based on the ANOVA calculations (see **Table 5**), the additives concentration, both $Na_2S_2O_5$ and NaOH do not affect the reduction of fiber content. This is also proved by the P values for both additives (>0.05). So, the physical treatment determines the amount of fiber in starch after extraction. This result is the same as for the starch yield.

3.4. Ash Content

Ash content shows the presence of inorganic component in the starch. These can originate from the rhizome, but also from the chemicals added during processing. From the ANOVA calculations (see **Table 6**) it can be seen that the concentration of NaOH and the physical treatment affect the amount of inorganic material left in the starch. This seems due to the solubility level of $Na_2S_2O_5$ in water which is higher than NaOH.

On **Figure 4**, it is shown that although $Na_2S_2O_5$ concentration does not affect the ash content significantly, there is a tendency that the higher concentrations of additives, both $Na_2S_2O_5$ and NaOH, result in a higher ash content in the starch.

4. CONCLUSIONS

This study showed that the integration of physical and chemical is a promising technology for the extraction of starch from *Canna edulis*. It is a relatively simple and low cost process and it produces a good quality starch.

Table 5. ANOVA for fiber content response.

Factor	SS	DF	MS	F Value	p-Value
[$Na_2S_2O_5$]	0.089	1	0.089	0.41	0.5261
[NaOH]	0.43	1	0.43	2.00	0.1717
treatment	4.54	1	4.54	21.18	0.0001

Table 6. ANOVA for ash content response.

Factor	SS	DF	MS	F Value	p-Value
[$Na_2S_2O_5$]	0.63	1	0.63	1.54	0.2284
[NaOH]	20.18	1	20.18	48.80	<0.0001
Treatment	2.55	1	2.55	6.18	0.0210

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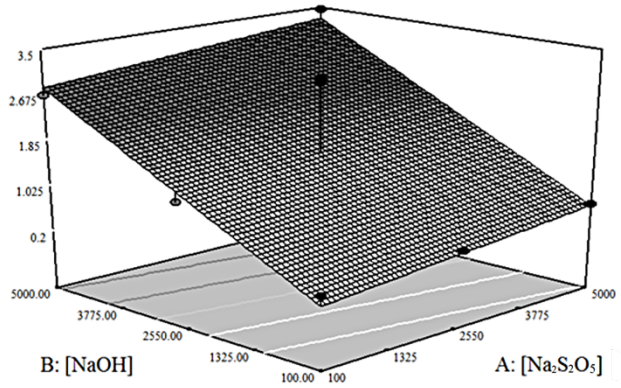

Figure 4. 3D surface of ash content.

The use of the screw press in the separation process of the fibers after the chemical extraction produce gives a higher starch yield compared to the use of hydraulic press, but the purity is lower. Therefore, the hydraulic press is perhaps the most suitable method for preparing starch that has to be a feed material for chemical modification processing.

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