MAKALAH ILMIAH

THE MICROWAVE-ASSISTED EXTRACTION OF INULIN FROM DAHLIA SP. TUBERS IN WATER

OLEH:

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The Microwave-Assisted Extraction of Inulin from Dahlia Sp. Tubers in Water

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Abstract. The Inulin is well-known as a valuable chemical since its structure contains fructose and glucose. Through extraction, inulin can be derived from Dahlia Sp. Tubers. Mostly, the acid and high temperature are used conventionally to extraction inulin from tubers. Instead of using acid and conventional hydrolysis, water and microwave-assisted extraction show promising. In term of the feasibility of the process, Dahlia Sp tubers are attractive alternative inulin feedstock as Dahlia Sp. tubers are abundant and cheap. This work is to find the optimum condition of inulin extraction in the aqueous medium based on the kinetic aspect. The Dahlia tuber powder was poured into microwave tubes filled with Milli-Q water in room temperature. Subsequently, the aqueous solution was added. Some tubes were located in a particular rack in the microwave reactor which was run on fixed temperature. The reaction time is in the range of 0-60 minutes. At diverse extraction times, a tube was withdrawn from the microwave reactor and speedily cooled down by the system to terminate the reaction. The reaction mixture was pipetted out, then the liquid product was separated from the mixture by centrifugation around 5-10 minutes. To identify and quantify, the liquid product was diluted then injected into vials to be analyzed with HPLC. A MALDI-TOF apparatus was also employed to identify the inulin structure. Variables investigated are temperature, loading concentration and reaction time. It can be concluded that (1) the optimum condition was influenced by temperature, loading concentration and reaction time; (2) the optimum condition providing the highest yield of 98.96 mole% was achieved at temperature reaction was 50 °C, loading concentration was 0,1 g/mL and reaction time was 50 minutes.

INTRODUCTION

Dahlia plant is one of the popular decorative plants which is easily cultivated almost all around the world. The Dahlia tubers also have higher economic value. Since the tubers are rich with carbon, the Dahlia tubers show promising as carbon feedstock to produce platform biobased chemicals. It is reported in the literature that Dahlia tubers are rich in inulin [1–3].

Inulin is a polysaccharide and has the chemical structure of the fructose-glucose unit and or fructose-fructose unit. The inulin structure is simply described in figure 1. Inulin is well-known considered a striking polysaccharide for biobased chemicals such as HMF for fuel and fructose for pharmacy industries [4–7]. Inulin is abundant and can be found easily from 30,000 plants all around the world [1,2,8]. Table 1 gives information about selected plants containing inulin [2].

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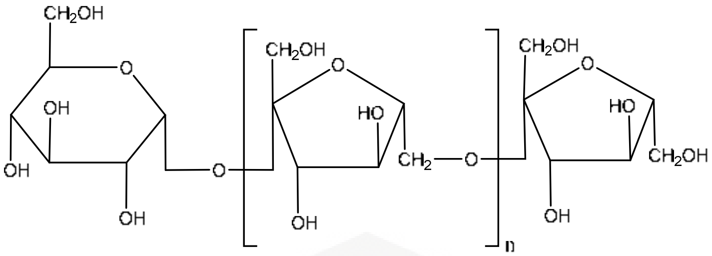

FIGURE 1. The chemical structure of inulin.

TABLE 1. Inulin content of some plants [2]

Source	Edible parts	Dry solids content	Inulin content
Onion	Bulb	6-12	2-6
Jerusalem artichoke	Tuber	19-25	14-19
Chicory	Root	20-25	15-20
Leek	Bulb	15-20	3-10
Garlic	Bulb	40-45	9-16
Artichoke	Leaves heart	14-16	3-10
Banana	Fruit	24-26	0.3-0.7
Rye	Cereal	88-90	0.5-1 (estimated value)
Barley	Cereal	not available	0.5-1.5 (estimated value)
Dandelion	Leaves	50-55 (estimated value)	12-15
Burdock	Root	21-25	3.5-4.0
Camas	Bulb	31-50	12-22
Murnong	Root	25-28	8-13
Yacon	Root	13-31	3-19
Salsify	Root	20-22	4-11

To produce inulin from the Dahlia tubers, the extraction process is obviously needed. The common method to extract inulin is conventional solvent extraction such as soxhlet and maceration. These methods are time and energy-consuming process which can provide the decomposition process of inulin. The fructose content in the inulin structure increases the reactivity of inulin. In order to avoid the inulin decomposition, this work performed the microwave-assisted extraction (MAE) of inulin from Dahlia Sp tubers. The MAE has been mentioned as one of powerful extraction methods. Due to fructose-glucose and or fructose-fructose units inside of its structure, inulin is soluble in water. This leads to the reason why water has been chosen as a solvent in this work. This work is to find the optimum condition of inulin extraction from Dahlia Tubers Sp in the aqueous medium based on the kinetic aspect.

EXPERIMENTAL METHODS

Materials

Dahlia Sp tuber was purchased from planters in Bandung, West Java, Indonesia. Inulin, Fructose (≥99%) and Glucose ((≥99%) were purchased from Sigma-Aldrich (Steinheim, Germany). Sulfuric acid (98%) was obtained from Merck KGaA (Darmstadt, Germany).

Extraction

The Dahlia tuber powder was fed into microwave tubes at room temperature around 20ºC. Subsequently, the aqueous solution was added. A sequence of tubes was positioned in a specific rack in the microwave reactor which set on constant temperature. The extraction time is in the range of 0-60 minutes. At diverse extraction times, a tube was withdrawn from the reactor and immediately cooled down by the system to terminate the reaction. The reaction mixture was pipetted out and fed into the centrifugation tube around 5-10 minutes to isolate the liquid product from the reaction mixture.

Analysis

To identify and quantify, the liquid product was diluted then injected into vials to be analyzed with HPLC of Agilent Technology 1200 series with an isocratic pump, a Waters 410 differential refractive index detector and a Bio-Rad Aminex HPX-87H organic acid column (300 mm x 7.8 mm) which was installed at 60ºC. The 5 mM of sulfuric acid in water was employed as the eluent at a fixed rate of 0.55 ml/min. Individually, the sample was investigated for 60 minutes. A MALDI-TOF apparatus was also employed to identify the inulin structure. Variables investigated are temperature (23-57ºC), loading concentration (0.016-0.184 g/mL) and reaction time (3-37 minutes).

RESULT AND DISCUSSION

Microwave-assisted extraction of Dahlia Sp tubers was performed in microwave tubes in temperature (23-57ºC), a loading concentration (0.016-0.184 g/mL) and extraction times (3-37 min). The influence of temperature, loading concentration and reaction time on the extraction yield was quantitatively observed. The yield is stated in wt% and marked as (weight of inulin/weight of Dahlia Sp tuber powders) x 100 %.

Effect of Process Variables on Inulin Yield

The effect of process variables on extraction yield was observed by performing 20 experiments with 6 repetitions in center point (C=0.1 g/ml, T=40ºC and t= 20 minutes). The result can be seen in Table 2. The highest extraction yield of 90 wt% was obtained at T= 40ºC, t=20 min and C=0.1 g/mL. Meanwhile, the effect of extraction time and loading concentration on inulin yield at constant temperature can be described as seen in Figure 2. As extraction time and loading concentration increases, the inulin yield also increases.

The longer extraction is performed, the number collision between solid particle (Dahlia Sp powder) and solvent molecules are higher. Figure 2 also informs that a higher temperature process leads to higher inulin yield. At 40 ºC,the yield is above 80 wt%, meanwhile at 30ºC, the yield is below 80 wt%. This finding fits with the Arrhenius theory stating at higher temperature, the reaction rate becomes faster.

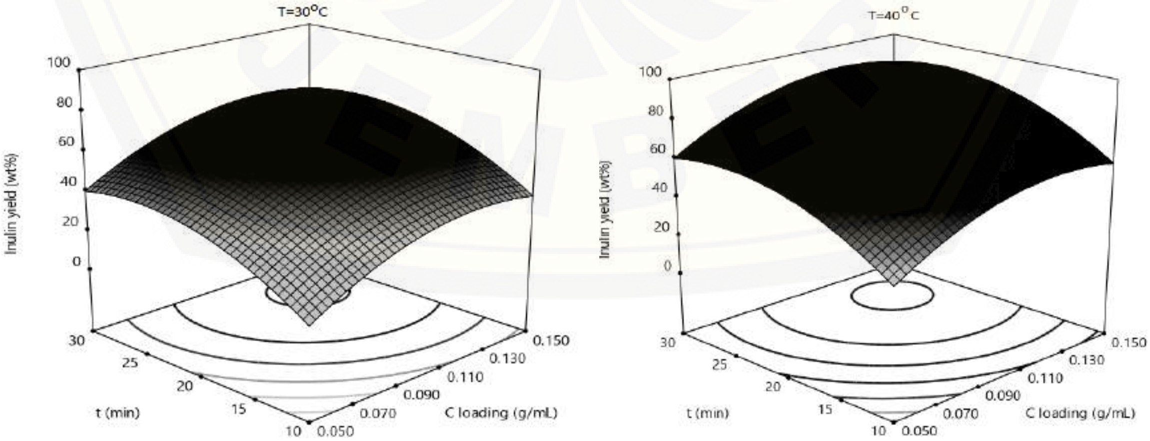

FIGURE 2. The effect of extraction time (10-30 mins) and loading concentration (0.05-0.15 g/mL) on constant temperature (left: T=30ºC; right: T=40ºC)

TABLE 2. Overview of experiments for the inulin extraction from Dahlia Sp tubers

Run	C, g/mL	t, min	T,ºC	Inulin yield,
1	0.1	20	40	90
2	0.1	20	57	79
3	0.05	10	50	35
4	0.15	10	30	42
5	0.05	30	30	41
6	0.184	20	40	82
7	0.1	37	40	84
8	0.1	20	40	90
9	0.1	20	23	30
10	0.1	20	40	85
11	0.016	20	40	29
12	0.1	20	40	87
13	0.15	10	50	55
14	0.15	30	50	80
15	0.15	30	30	55
16	0.05	30	50	57
17	0.1	3	40	22
18	0.1	20	40	85
19	0.1	20	40	86
20	0.05	10	30	17

C : loading concentration
t : extraction time
T : temperature

Kinetic Extraction

The kinetic extraction in this work follows the diffusion-controlled model. Since the inulin is strongly soluble in water, thus, the diffusion-controlled process takes places in one phase. In this work, it is assumed that the initial concentration of inulin at the initial time is zero and extraction takes places as diffusion occurs inside the cell wall of Dahlia Sp tuber powder. In this study, a mass balance equation can be expressed in equation 1.

$$\frac{1}{De}\frac{\partial C}{\partial t} = \frac{\partial^2 C}{dr^2} + \frac{2}{r}\frac{\partial C}{\partial r} \qquad (1)$$

C and t refer to the concentration of inulin at r position and time. The initial condition is as follows: C(r,0) = C_o; C(R,t) = 0.

The variables separation method was employed to solve equation (1). This gives an analytical solution for inulin fraction, as seen in equation (2).

$$x_{cal} = 1 - \frac{6}{\pi^2}\sum_{n=1}^{n=\propto}\frac{1}{n^2}e^{(-\beta\pi^2 t)} \qquad (2)$$

$$\beta = \frac{\pi^2 De}{R^2} \quad (3)$$

De refers to diffusion coefficient (m^2/min), R = radius of Dahlia Sp tubers powder, and t = diffusion time (min).

Figure 3 shows the yield profile of inulin extraction from Dahlia Sp tuber powder. This informs that inulin was extracted at the beginning of reaction (up to ~ 25 min) but decomposed to other fraction such as HMF. This phenomenon is in line with Fachri et al., [4–7,9,10] who reported that inulin is easily decomposed to fructose and consecutively goes to HMF.

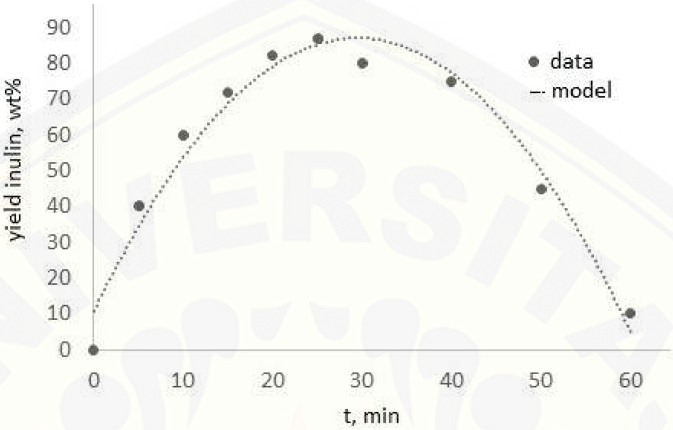

FIGURE 3. The profile of extraction curve

In the meantime, the diffusion coefficient (De) is 5.21 x 10^{-11} m^2/sec. This value is slightly in agreement with Figure 3 indicating that the extraction occurs faster. Herein this work, the diffusion coefficient represents the mass transfer process from solvent to wall cell and the solvent to inulin.

Inulin Characterization

In this study, the MALDI-TOF method was applied to characterize the molecular weight distribution of inulin. Figure 4 shows that the MALDI-TOF can successfully identify the inulin structure based on its molecular weight distribution. This method is well-suited for molecular weight determinations of oligosaccharides and polysaccharides [11,12].

Figure 4 also informs that Mn was found to be 2560 and the Mw was found to be 3680. This finding typically indicates a degree of polymerization of about 16. Roberfroid [2] reported that the degree of polymerization of inulin differs conferring to plant species, climate conditions, and the physiological age of the plant. In the chicory plant, the degree of polymerization values is in a range from 2 to 65. Characteristically, the value is 15. Roberfroid [2] also informed that the degree of polymerization of inulin from onion is in the range of 2-12, and less than 40 for Jerusalem artichoke. Clearly, this finding is in reported values in the literature.

Even figure 4 informs the inulin structure, however, the additional analysis using HPLC is needed to identify both fructose and glucose. In this issue, mild extraction of inulin sequenced by HPLC analyses of the liquid product phase was conducted. This work confirmed the inulin from Dahlia Sp tubers was structured by fructose and glucose. The chromatogram profile was described in Figure 5. For the meantime, the HPLC analysis concluded the fructose yield was 94 mol%, the balance being glucose, leading to a ratio of fructose to glucose of 15 to 1. The fructose content expressed in yield (mol%) is in adjacent agreement with the literature for Dahlia tubers (94.1 – 96.7 mol%). This result approves that the inulin sample consists principally of d-fructoside units which are chained with glucosic bond plugged with a d-glucose molecule, in line agreement with literature data [1,2].

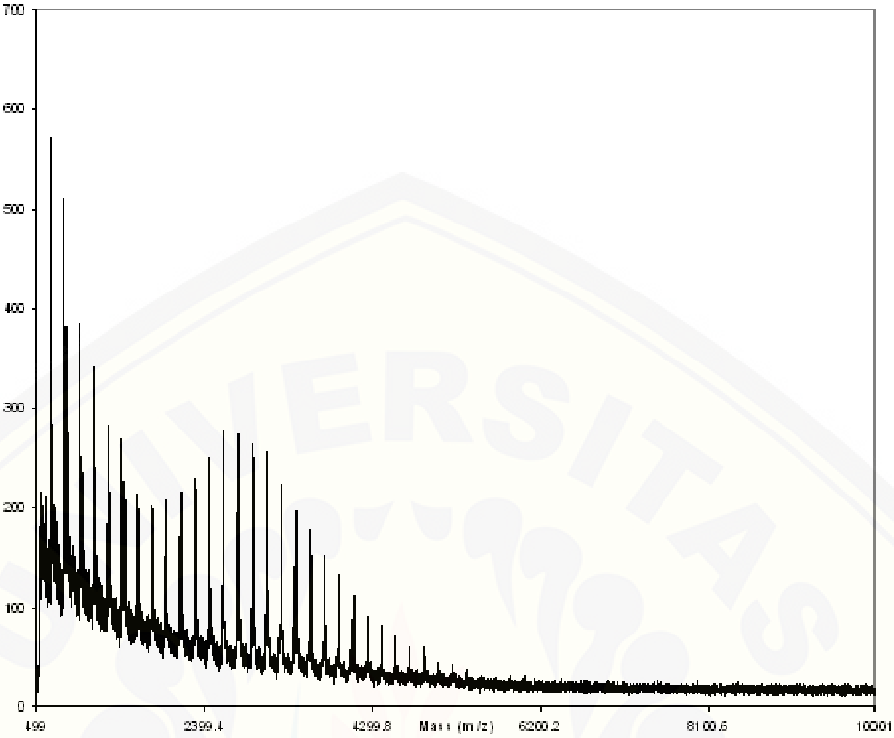

FIGURE 4. The spectrum of Inulin from Dahlia Sp Tuber by MALDI-TOF analysis

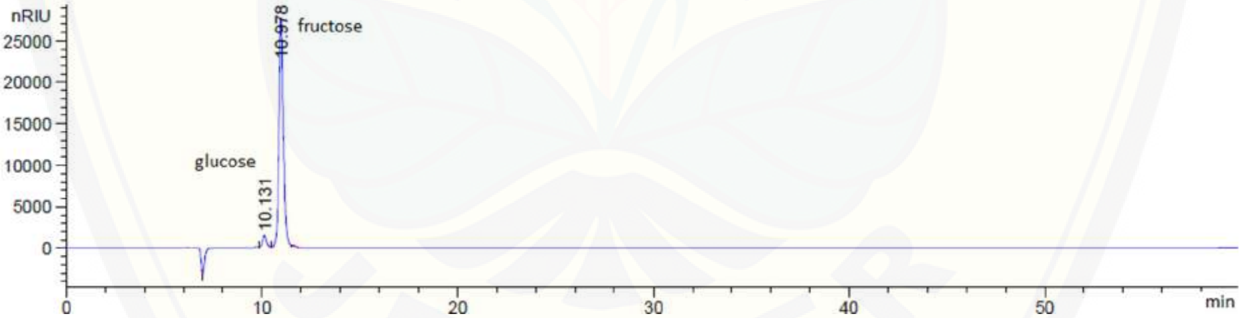

FIGURE 5. The chromatogram profile of Inulin extraction from Dahlia Sp Tubers powder by HPLC analysis

As the structure of inulin was successfully identified both by MALDI-TOF and HLPC, the mechanism extraction can be proposed as in figure 6. Thus, inulin extraction from Dahlia Sp tuber is strongly thermal-sensitive reaction providing the self-decomposition of inulin to fructose and glucose.

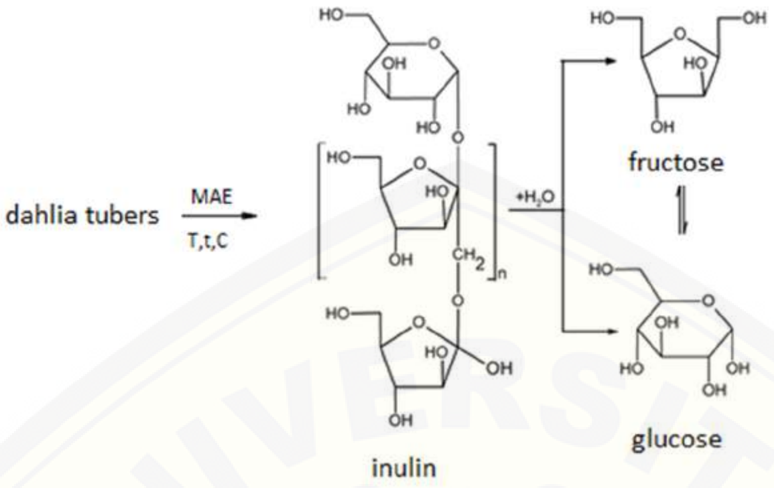

FIGURE 6. The proposed reaction mechanism in inulin extraction

CONCLUSION

The microwave-assisted extraction of inulin from Dahlia Sp tubers in aqueous medium was studied in range of reaction conditions, involving variations in temperature (23-57ºC), a loading concentration (0.016-0.184 g/mL) and extraction times (3-37 min). The highest inulin yield was of 90 wt% was obtained at T= 40ºC, t=20 min and C=0.1 g/mL. Due to the investigation of the effect of process variables on inulin yield, the temperature has a profound effect on extraction yield. and was also investigated and indicates Based on kinetic study which is approached by diffusion-controlled model, the diffusion coefficient was found to be 5.21 x 10-11 m^2/sec.

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The authors declare that they have no competing interests.

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