

The Extraction of Oxalate Acid from Porang Flour (*Amorphophallus oncophyllus*) using Microwave-Assisted Solvent Extraction

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Abstract

Porang (*Amorphophallus oncophyllus*) is one of the local annual plants that contain high levels of oxalate. Oxalates are undesirable compounds in *porang* flour, and their levels must be removed or reduced to obtain high quality *porang* flour as a food ingredient. In this research, the extraction of oxalate acid was conducted using microwave-assisted solvent extraction and mechanical separation methods. This study aims to analysis the parameters that influence the extraction of oxalate acid using microwave-assisted solvent extraction such as the effect of microwave power, extraction time, feed-to-solvent ratio (F/S) and material size. The mechanical separation process will be studied the effect of material size for extraction of oxalate acid. The optimum yield obtained for oxalate acid extraction with microwave-assisted solvent extraction was microwave power of 600 W, extraction time of 30 min, feed-to-solvent ratio of 0.05 g/mL and porang size of 100 mesh obtained by oxalate acid yield of 24.78%. Porang size of 100 mesh was the optimum yield obtained for extraction of oxalate acid using mechanical separation method. The result of physical properties test using SEM-EDX and FTIR shows that extraction of oxalate acid from porang using microwave extraction method could produce oxalic acid which has good quality. In addition, extraction using microwave offers an environment-friendly extraction method, which can accelerate and increase the oxalate acid extraction yield.

Keywords: *Amorphophallus oncophyllus*; porang flour; microwave; extraction; oxalate acid

1. INTRODUCTION

Oxalate acid was first synthesized in 1776 by Schleele, with the oxidation of sugars and nitric acid. Oxalate acid ($H_2C_2O_4$) is a relatively strong organic acid compound, 10000 times stronger than acetic acid. Oxalate acid is a colorless crystalline substance with a strong acid taste. Oxalate acid at high concentrations can be a dangerous toxic. Many patents and papers concern the process of producing oxalate acid with the oxidation of nitric acid from cellulose. Most of these processes involve hydrolysis from cellulose to glucose, then oxidized using nitric acid or sulfuric acid to

produce oxalate acid ¹. Oxalate acid is widely available in the form of potassium and calcium salts found in many leaves, roots and rhizome from a variety of plants. In addition oxalate acid is also found in the urine of humans and animals in the form of calcium salts that are partly in the kidneys ².

Oxalate acid has been widely used as a bleach or cleaning agent. In the textile and wood industry, oxalate acid has been used as a bleach. In addition, oxalate acid can be used as rust remover for metal industry and water treatment. Asia is becoming the biggest consumer of oxalate acid in

the world, especially China is a major consumer, manufacturer as well as exporter of oxalate acid. Currently, Indonesia is still importing oxalate acid from abroad to meet the needs of oxalate acid in the country. The need for oxalate acid for industry in Indonesia in 2016 amounted to 1,469 tons. High oxalate components can found in plants such as *amaranthus*, *colocasia* (taro), *orach*, *beet*, *spinach*, *tetragonia*, *oxalis*, *rheum*, *rumex*, *portulaca* ² and *porang* (*Amorphophallus oncophyllus*) ³.

Porang (*Amorphophallus oncophyllus*) is a perennial plant that generally grows in the forests of Indonesia and includes plants with root tubers. *Amorphophallus oncophyllus* was originally found in the tropics from Africa to the Pacific, then spread to temperate climates such as China and Japan. The spread of *porang* from the Andaman islands of India, spreading eastwards through Myanmar then entering Thailand and Indonesia ⁴. The *porang* flour (*Amorphophallus oncophyllus*) is rich in glucomannan soluble fiber, glucomannan content in *porang* form of about 70-90% ⁵ where glucomannan is better known as Manan. *Porang* flour has a dark brown color and causes itching, so it is not safe to eat. This is due to the high content of oxalate in *porang* tubers ⁶. But oxalates are undesirable compounds in *porang* flour, and their levels must be removed or reduced to obtain high quality *porang* flour as a food ingredient. Removal of oxalate in *porang* flour can be conducted using extraction methods.

Solvent extraction has been used extensively for isolation of important compounds and for qualitative and quantitative analysis in areas such as environmental analysis, food and agricultural analysis, pharmacological drugs and herbal medicines ⁷. The disadvantage of extraction using a solvent when applied to *porang* flour is that the solvent can contaminate the *porang* flour which is used as a food ingredient, the solvent extraction process is relatively longer so it is inefficient and requires expensive costs to provide the solvent ⁸. Currently, one of the methods that have been developed to speed up the extraction process is extraction using a microwave (microwave-assisted extraction method). Microwave-assisted extraction method is a relatively new extraction technique that combines microwave and soxhlet extraction method or can be called microwave-assisted solvent extraction ⁹. In recent years extraction with the help of microwave has attracted interest, as it allows speed up the extraction process. Microwave heating is a radiation heating that has several advantages such as: heating occurs without direct contact, reduces thermal gradient, heat rapidly and the heating effect stop rapidly, heat start from inside the material, transfers energy based on the

radiation heat transfer ¹⁰. Another advantage gained when using microwave extraction method is higher yield, environmentally friendly, lower energy consumption and economical compared to conventional methods ¹¹.

Production of oxalate acid with microwave use can reduce energy consumption and speed up extraction time because microwave radiation can propagate through the solvent and allow the heating process to be more effective and the extraction process can be done faster ⁹. The purpose of this research is to study various parameters that affect the extraction of oxalic acid from *porang* flour using microwave-assisted solvent extraction. These parameters include microwave power, extraction time, and the raw material ratio, also known as the feed-to-solvent ratio. These parameters need to be studied because this can affect the oxalate acid extraction process of *porang* flour using microwave-assisted solvent extraction.

2. RESEARCH METHODS

Material and chemicals

The dried *porang* chips are obtained from Nganjuk district, East Java, Indonesia. The dried *porang* is then stored at room temperature until use. Pro-analytical chemicals used: sodium bicarbonate (NaHCO_3); calcium chloride (CaCl_2); sulfuric acid (H_2SO_4); 96% ethanol; and potassium permanganate (KMnO_4) and aquadest.

Determination of total oxalate using mechanical separation and solvent extraction

Mechanical separation method does in two stages, grinding process and sieving stage. The grinding process, the *porang* in the form of chip in the puree uses a disk mill (Power 1.5 Hp/1100W, 220V) with dimensions of 60 cm x 95 cm x 35 cm and a maximum capacity of 20 kg material. Furthermore, the finely sieved *porang* uses a vibrating screen of 60, 80 and 100 mesh. The smoothed *porang* is stored in a sealed container with room temperature of 25°C until use.

Samples of 5 grams each, with particle sizes of 60, 80, and 100 mesh, were combined with 200 ml of sodium bicarbonate solution. The mixture was heated using a heating mantle (as shown in Figure 1) for 15 minutes, followed by centrifugation to separate the filtrate from the precipitate. The filtrate is added with sulfuric acid (H_2SO_4) and then titrated in a warm state using KMnO_4 solution until a pink change occurs for 30 seconds. Oxalate acid (ppm) can be calculated using equation (1):

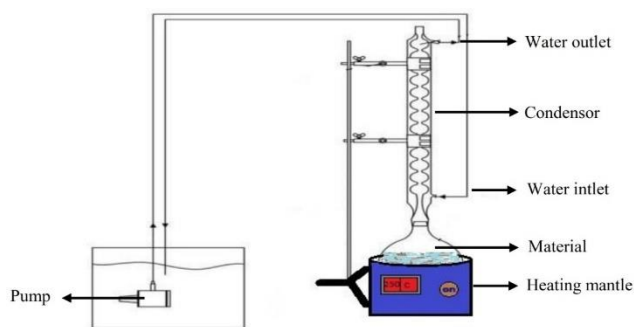


Figure 1. Schematic apparatus for oxalate acid extraction using solvent extraction method.

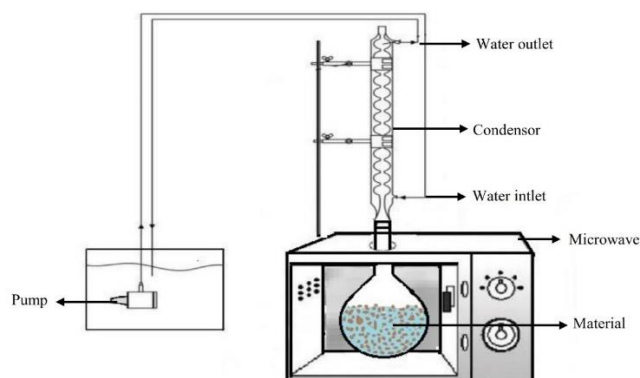


Figure 2. Schematic apparatus for oxalate acid extraction using microwave-assisted solvent extraction method.

$$X = \frac{V \times N \times BE}{m} \times 1000 \quad (1)$$

where X is the oxalate acid (ppm), V is the titration volume (ml), N is the normality of KMnO_4 , BE is the equivalent weight of oxalate and m is the weight or mass of the *porang* flour (g).

Microwave-assisted solvent extraction method

In the process of extracting *porang* flour using the microwave-assisted solvent extraction method, a domestic microwave oven (EMM-2007X, Electrolux) with the following specifications is employed: it has a capacity of 20 liters and can deliver a maximum power of 800 W. The microwave oven operates at a wave frequency of 2450 MHz. To facilitate the extraction process, the microwave cavity is lined with PTFE (polytetrafluoroethylene), and the microwave dimensions measure 46.1 cm in length, 28.0 cm in width, and 37.3 cm in height. As part of the modification to adapt the microwave oven for the extraction process, holes are drilled at the top of the oven. A round-bottom flask with a 1000 ml capacity is placed inside the oven, and it is connected to a reflux condenser through one of these holes. This setup, as illustrated in Figure 2, allows for the efficient and controlled extraction of *porang* flour using microwave-assisted techniques.

Porang flour with feed to solvent ratio (0.05; 0.10; and 0.15 g/mL) is placed in a 1 L flask containing 200 mL of water and then added 20 g of sodium bicarbonate. The flask is arranged in a microwave cavity and connected to the reflux condenser at the top (outside the microwave). Microwave ovens operate at various power levels (300 W; 450 W; and 600W) with a time of 10, 20 and 30 min. After the extraction process is complete, the precipitate and filtrate are separated then CaCl_2 is added into the filtrate and decantation is obtained until calcium oxalate is obtained. Calcium oxalate is obtained by adding sulfuric acid and heated by hot plate until boiled, then the solution cooled. Furthermore, the solution is reheated until boiled, and the filtrate is cooled back to form a white crystalline precipitate as well as the filtration process. The crystalline precipitate obtained was washed using ethanol to remove the sulfuric acid content of the crystalline precipitate. The results of the washing of oxalate acid in the form of crystals and then stored into a vial bottle at room temperature until the analysis. The oxalate acid yield can be calculated using equation (2):

$$Y = \frac{W}{W} \times 100 \quad (2)$$

where Y is the yield of oxalate acid (% w/w), w is the weight or mass of oxalate acid extracted (g) and W is the weight or mass of *porang* flour (g).

Analysis of oxalate acid

SEM-EDX analysis of oxalate acid

The chemical composition of the oxalate acid surface is semi-quantitatively analyzed using scanning electron microscopy in combination with energy dispersive X-ray (SEM-EDX). The sample is prepared as an analysis using SEM. For semi-quantitative evaluation, it is assumed that the peak height is proportional to the mole fraction of an element. In small crystals with dimensions smaller than resolution, the spectrum of the apical surface next to the crystal serves as a reference to distinguish between oxalate acid and other materials that can form crystals.

FTIR analysis of oxalate acid

Fourier transform infrared spectroscopy (FTIR) can be used to analyze oxalate acid compounds in the sample. The FTIR spectra from the extracted sample were recorded using a FTIR spectrophotometer using the ATR sampling technique by recording 45 scans in% transmittance mode in the range of $4000\text{-}500\text{ cm}^{-1}$.

Table 1. Research on glucomannan purification using conventional methods.

Methods	Processing time	Yield	Refs.
Extraction using water bath heating and stirring. Solvent: HCl 0.125-1 M and ethanol 95%.	0.25-3 hours Optimum 1 hours	glucomannan 98.34% recovery 90.18- 91.03%	12
Wet extraction: <i>porang</i> tubers in 50-96% ethanol solvent. Room temperature	0.5-3 hours	glucomannan, 66.56%; calcium oxalate 0.56%.	13
Extraction was carried out in a flask in which stirring occurred at room temperature with HCl solvent: 0.1 - 0.9 M.	0.5-3 hours	glucomannan 95.85% at HCl 0.7 M; F:S ratio 1:5; temperature 70°C.	14

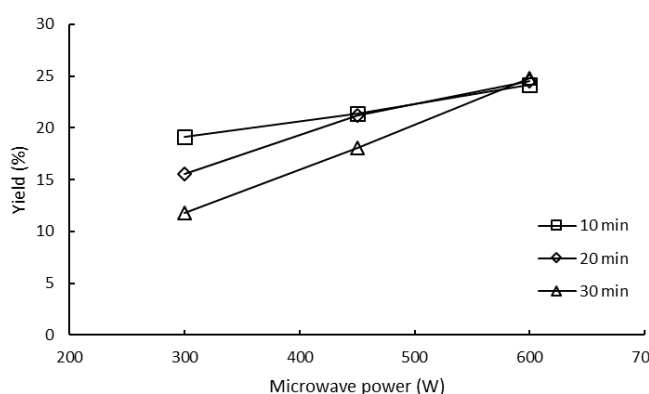


Figure 3. Effect of microwave power to yield of oxalate acid (F/S 0.05 g/mL, 100 mesh)

3. RESULTS AND DISCUSSION

Research on purifying glucomannan using conventional methods.

Table 1 provides a comprehensive overview of various established methods for the extraction of oxalate from a source material to yield highly purified glucomannan. Table 1. effectively illustrates that the most successful results in terms of both the quantity and quality of glucomannan are achieved when utilizing hydrochloric acid (HCl) as the solvent, accompanied by a relatively efficient processing time spanning from 0.5 to 3 hours. This indicates that HCl exhibits a strong affinity for oxalate, allowing it to effectively separate from the glucomannan flour, resulting in a higher level of purity. However, despite its efficacy, a significant drawback associated with HCl as a solvent emerges, namely its inherent toxicity concerning its suitability for food applications. Given the importance of ensuring the safety and edibility of food products, the use of HCl in this context is deemed problematic. Considering this concern, researchers and practitioners seek a more benign alternative. One promising solution proposed in the paragraph is the application of microwave-assisted

techniques. These methods leverage microwave energy to expedite the extraction process. This acceleration stems from the rapid and uniform heating of both the solvent and the sample. Such techniques hold promise for their potential to maintain or even enhance the effectiveness achieved with HCl while mitigating safety and environmental concerns. The choice of non-toxic and environmentally friendly solvents in conjunction with microwave-assisted techniques underscores the broader shift towards more sustainable and responsible practices in research and industry. These considerations emphasize not only the importance of yield and purity but also the essential criteria of safety and ecological impact in the pursuit of effective extraction methods for food-related applications. Further research and rigorous testing are expected to be pivotal in evaluating the feasibility and safety of these alternative approaches.

Therefore, it is important to choose properly the microwave power used to minimize the time it takes to reach the optimum temperature and avoid the bumping effect during the extraction process. From this study the increase in the amount of oxalate acid yield increases as microwave power is used. Figure 3 shows the oxalate acid obtained with microwave power 600W result high yield when compared to the oxalate acid yield obtained with microwave power 300W. This is because the higher the microwave power used will cause the polar molecules in the material to rotate rapidly (oscillation motion and impact).

The same phenomena also reported by Kusuma and Mahfud 2017¹⁷ for the extraction of patchouli oil, using high microwave power will increase yields as well and produce higher operating temperatures. Temperature rise is the result of the ability of materials and solvents to absorb energy from microwave, so it is important

to select the solvent used to interact with the material. Polar molecules and ion solutions (usually acid) can absorb microwaves energy due to permanent dipole moment. On the other hand when exposed to microwaves, nonpolar solvents such as hexane will not heat up. Therefore, by increasing the microwave power for oxalate acid extraction from *porang* flour then it will get higher oxalate acid yield.

Effect of ratio of feed to solvent (F/S) on microwave power

One important factor affecting extraction using microwave-assisted solvent extraction method is the type of solvent used. Selection of suitable solvents can make the extraction process run more efficiently. The solvent selection itself also depends on several things such as the solubility of the component to be extract, the penetration ability and its interaction with the matrix of the sample or the material and the dielectric constant¹⁸. In the extraction of oxalate acid using microwave-assisted solvent extraction method the solvent selection also needs to consider the capacity of the solvent to absorb microwave energy and its heating capability¹⁹. In this study, sodium bicarbonate solvent was employed for oxalate acid extraction from *porang* flour.

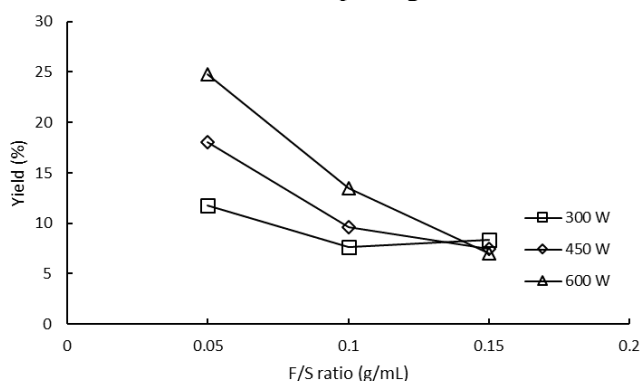


Figure 4. Effect of ratio of feed to solvent (F/S) to yield of oxalate acid (30 min, 100 mesh)

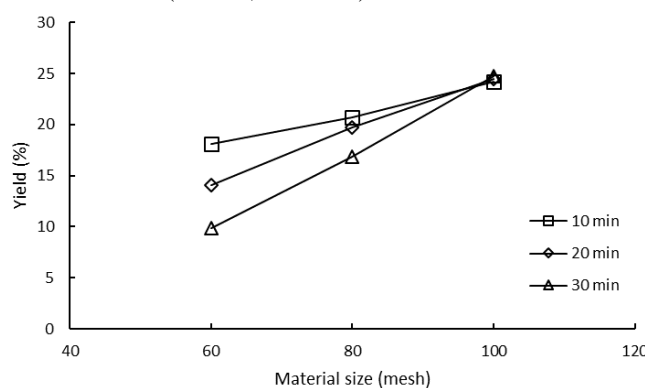


Figure 5. Effect of material size to yield of oxalate acid (F/S 0.05 g/mL, 600 W)

In addition to the solvent use effect, the ratio between the mass of the feedstock and the solvent volume (F/S) also affects the yield of oxalate acid extracted, this can be seen in Figure 2. The *porang* masses used in this study were 10, 20 and 30 grams with 200 ml solvent volume and extraction time of 30 min. Figure 4 shows that with the higher F/S ratio, yield of oxalate acid obtained will be lower. The optimum result for oxalate acid extraction from *porang* using microwave-assisted solvent extraction method is F/S ratio 0.05 g/ml. *Porang* flour extracted with a small F/S ratio will yield high yields due to the low density of the material in the flask. The density of the material is closely related to the large space between materials. The density of the material is too high to cause the formation of steam lines that can cause the yield of the yield²⁰.

These results are similar to Kumoro and Hartati 2015²¹ on the removal of *discorine* in *gadung* using microwave assisted extraction with high ratio (1:20) resulting in lower extraction. Chen yi 2007²² also reported the same thing that the high ratio (1:30) used for saponins extraction from *Ganoderma atrum* with 75% ethanol yield low extraction results. This is due to the increased mass of the material not followed by increasing and the volume of solvent used. Solvents with high dielectric constant will absorb more energy from microwaves. With many solvents being used, more microwave energy can be absorbed by the material. This makes the solvent experience a higher heating rate because of microwave radiation so using a feed to solvent ratio high will result in higher yields. Excessive solvent may also cause the removal of the desired compound and decrease the extraction selectivity to the desired compound²³.

Effect of raw material size on extraction time

The effect of *porang* flour size on the oxalate acid extraction results can be studied in terms of the size of the material used. In this study the material size used was 60, 80 and 100 mesh and the extraction time used was 10, 20 and 30 min with microwave power of 600W. The effect of the material size to the extraction time is if the smaller the material size to extraction it will cause the penetration of the microwaves to be more effective. Smaller-sized materials allow for more effective microwave penetration, which, in turn, increases the efficiency of the extraction process²⁴

Figure 5 shows that the yield of oxalate acid obtained reaches an optimum point with a material size of 100 mesh and at material size of 60 mesh the yield of oxalate acid obtained less. In

general, with the larger material size, the yield will be less. The same is also reported by Zhiyi at al 2006²⁵, the extraction rate increased with decreasing particle size because the intraparticle diffusion resistance was smaller for smaller particle size due to the shorter diffusion path. Large particle sizes provide a smaller contact area with microwaves emitted thus affecting mass transfer and not running efficiently, while the small size of the material provides a wider contact area to the microwave so mass transfer becomes easy.

Effect of extraction time on ratio of feed to solvent (F/S)

Based on the effect of the length of extraction time to oxalate acid yield for a various variable of plant material to solvent ratio, it can be seen in Figure 6. In this study the ratio of feed to solvent used was 0.05 g/mL and 0.01 g/mL while the extraction time used was 10, 20 and 30 min for microwave power of 600W and *porang* flour size of 100 mesh. In Figure 6 can be seen the relationship between the extraction time to the yield obtained from a various variable of plant material to solvent ratio using microwave-assisted solvent extraction method.

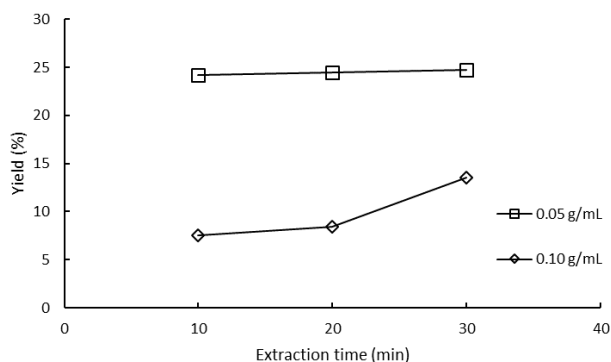


Figure 6. Effect of extraction time to yield of oxalate acid (100 mesh,600 W)

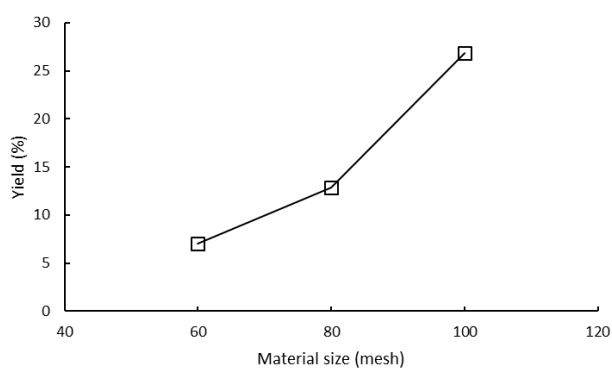


Figure 7. Effect of material size to yield of oxalate acid.

The yield of oxalate acid will increase as the extraction time increases at the solvent extraction microwave method. Because extraction processes performed under the action of microwave radiation are believed to be affected in part by polarization, volumetric and selective heating²⁶. Selective heating in microwave radiation can directly penetrate the transparent pumpkin (forwarding microwaves), so that the radiation can be directly absorbed by materials and solvents that are absorbing microwaves. Because microwave have a volumetric heating effect, much faster temperature increases can be obtained, depending on the microwave power and the dielectric loss factor of the material being irradiated²⁷. Because of this, the yield of oxalate acid is obtained faster when using microwave-assisted techniques.

From Figure 6, it can be seen that the extraction time of oxalate acid does not significantly affect the yield of oxalate acid extraction from *porang* flour. In theory, the longer the extraction time, the greater the yield obtained. Extraction time is also one of the factors that need to be considered. However, with the longer the extraction time, the increase in yield obtained becomes smaller²⁸.

Determination of oxalate acid yield using mechanical separation method

In this study, oxalate acid extraction in addition to using microwave-assisted solvent extraction method also uses the solvent extraction (conventional) method. After the extraction process with solvent extraction method was done, the solution of oxalate acid result extracted will titrate. Basically, the purpose of the titration is to determine the equivalent point, which is the point where the titration reaches the equilibrium point stoichiometry. This process includes a type of redox titration that uses potassium permanganate (pink or purple) as a titrant so that it does not need an indicator. When the titrant is reduced, the solution will become colorless. After reaching the equivalence point, there is an excess residual titrant in the solution. The equivalence point is identified when the first pink color appears (due to excess permanganate) in the solution being titrated²⁹.

In Figure 7, it is shown that the highest oxalate acid yield is obtained with a material size of 100 mesh, while a decrease in oxalate acid yield is observed with a material size of 60 mesh. This is because as the size of the material increases, the amount of glucomannan also increases, which, in turn, leads to less oxalate acid being extracted.

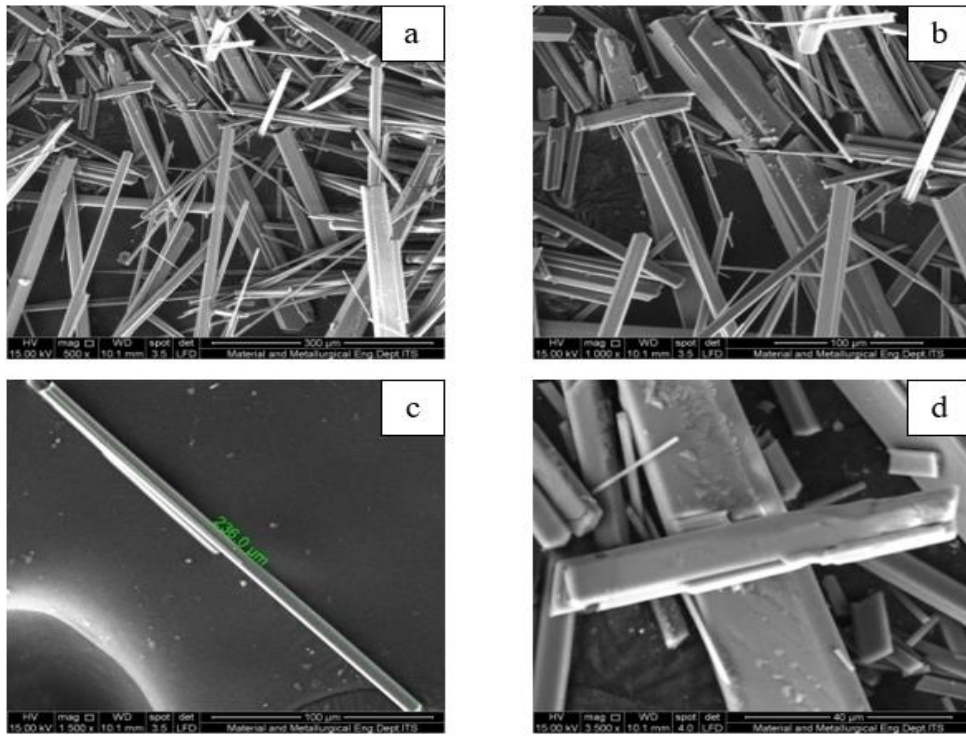


Figure 8. Results of SEM-EDX oxalate acid with magnification (a) 500, (b) 1000, (c) 1500 and (d) 3500 times

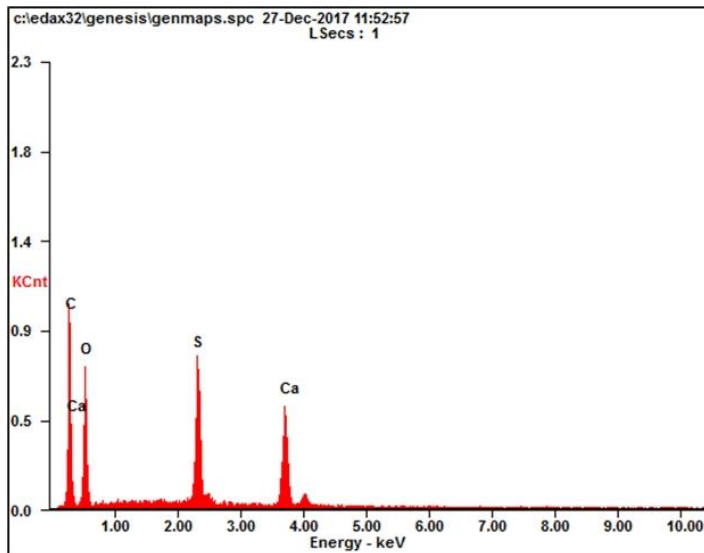


Figure 9. Result of EDX analysis for oxalate acid from *porang* tuber

Element	Wt%	At%
CK	29.91	44.01
OK	35.30	39.00
SK	15.00	08.27
CaK	19.78	08.72
Matrix	Correction	ZAF

Oxalate Acid Characterization Analysis of SEM-EDX

In SEM-EDX analysis the analyzed material is oxalate acid obtained from the extraction using microwave-assisted solvent extraction method. The result of SEM-EDX analysis obtained can be seen in Figure 8.

In Figure 8 it can be seen clearly that the oxalate acid component contained in needle-shaped *porang* tubers where the length of the needle can be measured at 1500x magnification of 236.0 μm as shown in Figure 8 (c). In addition to its morphology, EDX analysis is also used to

determine the components of the material contained in oxalate acid as in Figure 9.

In EDX analysis there are some elemental contents such as C and O which indicate oxalate acid. Whereas S and Ca indicate the presence of sulfate and calcium. The content of Ca comes from the components of *porang* where in *porang* there is calcium oxalate, from the results of EDX analysis the content of Ca is only 8.72%. While from the analysis of oxalate acid with EDX sulfate content of 8.27%. This is because the sulfuric acid contained in oxalate acid is still not washed clean as a result of the administration of sulfuric acid solution in the extraction process of sulfuric acid.

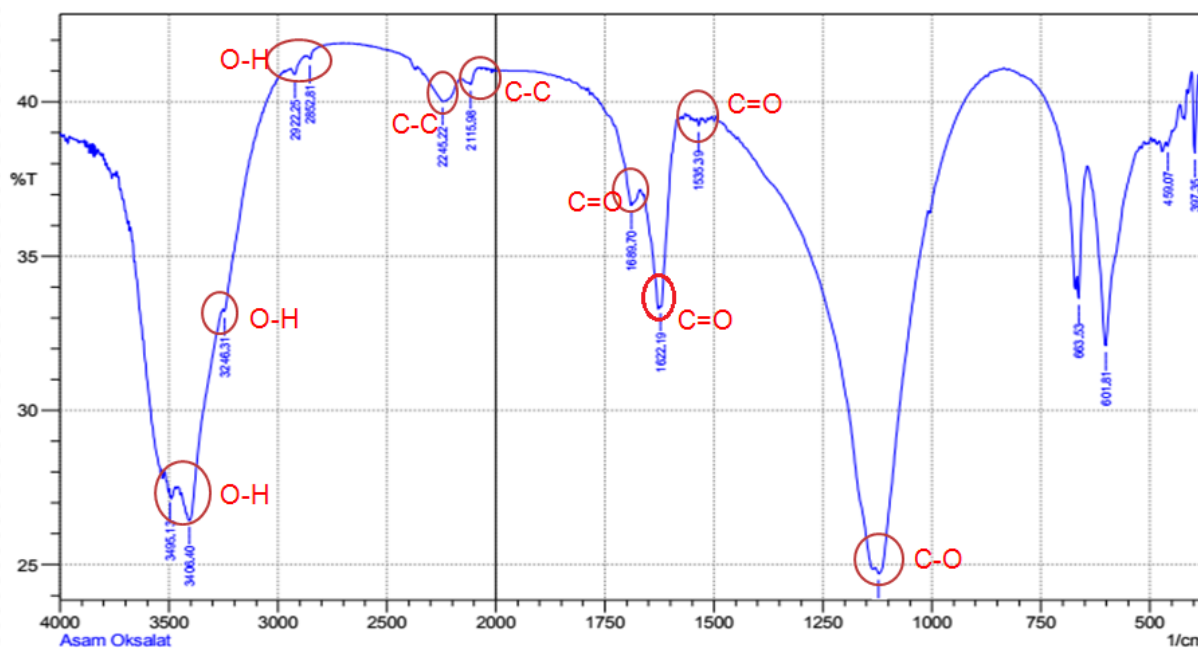


Figure 10. FTIR spectrum of oxalate acid from *porang* tubers (*Amorphophallus oncophyllus*) (O-H (alcohol), C = O (carboxylic), C-C (alkyne), C-O (ether))

Table 1. Oxalate acid functional groups extraction oxalate acid from porang

No	Functional group	Synthesized oxalate acid from porang (cm ⁻¹)
1	C-O	1122.61
2	C=O	1535.39
3	C=O	1622.19
4	C=O	1689.70
5	C-C	2115.98
6	C-C	2245.22
7	O-H	2852.81
8	O-H	2922.25
9	O-H	3246.31
10	O-H	3406.40
11	O-H	3495.13

Analysis of FTIR

FTIR can be used to analyze organic and inorganic compounds and can also be used for qualitative analysis including functional group analysis (the existence of 'peaks' of specific functional groups) and their patterns and quantitative analysis by looking at the strength of the absorption of compounds at certain wavelengths. The result of functional oxalate acid specific analysis on *Amorphophallus oncophyllus* (*porang*) tuber using FTIR is shown as Figure 10.

This analysis was conducted to compare the synthesis of oxalate acid from *porang* tuber with standard oxalate acid. Figure 10 shows the vibration of an alcohol group (O-H) strain with the FTIR spectrum for oxalate acid from the *porang* tuber present at wavelengths of 3495.13-397.35 cm⁻¹. The oxalate acid wavelength is most clearly seen in 2852.81-3495.13 cm⁻¹ which is a bond

between O-H (alcohol) in the oxalate acid chain. The wavelength 1200 cm⁻¹ at the ether function indicates the wavelength of 1122.61 cm⁻¹ shows the C-O bond. The C-C bonds on alkyne compounds are shown at wavelengths of 2115.98-2245.22 cm⁻¹, while for C = O bonds on carboxylic compounds are shown at wavelengths 1535.39-1689.7 cm⁻¹.

4. CONCLUSIONS

Extraction of oxalate acid from *porang* flour using microwave-assisted solvent extraction method with material size of 100 mesh, microwave power of 600 W, feed-to-solvent ratio (F/S) of 0.05 and extraction time for 30 min obtained optimum oxalate acid yield. The optimum extraction of oxalate acid using mechanical separation method is the smallest material size (100 mesh). SEM analysis produces morphological forms such as needles which indicate the content of oxalate acid

and EDX analysis produces elemental content which indicates the presence of oxalate acid. In the analysis using FTIR, the cluster groups contained in oxalate acid are C-C, C=O, C-O, and O-H groups. Extraction using microwave is an environment-friendly extraction method, which can accelerate and increase the oxalate acid extraction yield.

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