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Evaluation of some analytical methods for determination of calcium oxalate in Amorphophallus muelleri flour

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Abstract

The presence of calcium oxalate is the major obstacle in flour processing of Amorphophallus muelleri tuber since the calcium oxalate can induce skin irritation and is harmful to kidneys. The development of a rapid analytical method to detect calcium oxalate in Amorphophallus flour is required. This research was intended to evaluate the use of some analytical methods (FTIR, SEM-EDS, XRD, XRF, and titration methods) in calcium oxalate detection in Amorphophallus muelleri flour prepared from different treatments (soaking in water (W), solution of sodium bisulfite 1000 ppm (B), solution of sodium chloride salt 3% (S), solution of sodium bisulfite 1000 ppm and sodium chloride salt 3% (BS)). Results showed that the presence of oxalate in Amorphophallus flour can be detected in the FTIR spectra from the C=O group at a wavenumber of 1610 cm-1. SEM images confirmed that calcium oxalate in Amorphophallus flour existed as raphide crystals in which their quantity can be estimated by the EDS feature of SEM. The presence of calcium oxalate crystals in Amorphophallus flour can be differentiated from other salts present in the flour by XRD. XRF can be used as a rapid analytical tool to detect the presence of calcium oxalate in Amorphophallus flour. The potassium permanganate titration technique can be used as a reference method for other rapid analytical methods in detecting calcium oxalate in Amorphophallus flour.

Keywords: calcium oxalate crystal; analytical methods; Amorphophallus muelleri flour.

Practical Application: The investigated methods can be used to determine the calcium oxalate content in Amorphophallus muelleri flour in glucomannan industry.

1 Introduction

Most people in South East Asia consume rice as their staple food. However, consuming rice may not be suitable for some persons since it is classified as high glycemic index food which can induce high blood sugar (Kim et al., 2003). Moreover, older people should be more aware of the negative impact of daily and high rice consumption (Golozar et al., 2017). People with diabetes and other degenerative diseases should also avoid rice consumption since high blood sugar may induce organ or cell damage (Al-Ishaq et al., 2019). Therefore, alternative food with a low glycemic index should be introduced as a new staple food for people of South East Asia.

Amorphophallus tubers have been known as a source of food in Japan and China. People of Japan and China has consumed shirataki noodles prepared from the tuber as part of their daily food. However, food product from the tuber has not been widely known for people in South East Asia. Amorphophallus tuber contains a high percentage (up to 55% on a dry basis) of a precious carbohydrate substance namely glucomannan (Yanuriati et al., 2017). Glucomannan is classified as a soluble dietary fiber compound that contains tons of functional benefits for human health. It has the ability to lower the risk of developing hemorrhoids and small pouches in the colon (diverticular disease). It shows to lower cholesterol levels (Keithley et al., 2013) and helps control blood sugar levels (Shah et al., 2015). Therefore, food products developed from glucomannan of Amorphophallus tuber will be beneficial for health.

Most Amorphophallus tubers cannot be consumed directly since they contain calcium oxalate, an anti-nutrient compounds, that can irritate skin and is harmful to kidneys (Singh et al., 2018; Chairiyah et al., 2016; Siener et al., 2021). Therefore, a processing step should be carried out to remove the calcium oxalate from the flour of Amorphophallus tuber before it can be further processed to be a food product. Calcium oxalate can be removed from the flour of Amorphophallus tuber by means of separation techniques such as water soaking (Coronell Tovar et al., 2019) producing safe Amorphophallus flour (Witoyo et al., 2021; Kumar et al., 2017; Witoyo et al., 2020).

To date not so many analytical methods have been developed to evaluate calcium oxalate content in food. The most common methods to determine the calcium oxalate are titration and spectrophotometric methods (Mishra et al., 2017; Alavi & West, 1983; Ilarslan et al., 1997; Burrows, 1950; Fiske & Adams, 1931) and chromatographic method (Minocha et al., 2015; Huang & Tanudjaja, 1992). These methods require a long time for sample

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preparation and need a higher amount of toxic chemicals. Therefore, the development of a quick analytical method to estimate the calcium oxalate content in food samples specifically in Amorphophallus flour is required. This research was aimed to evaluate the use of some analytical methods in the determination of calcium oxalate in Amorphophallus muelleri flour.

2 Materials and methods

2.1 Materials

Amorphophallus muelleri tuber was purchased from a local farmer in the district of Subang, province of West Java, Indonesia. Sodium bisulfite and sodium chloride salt were purchased from the local market. All chemicals for analysis were analytical grade.

2.2 Sample preparation

Samples of Amorphophallus muelleri flour were prepared by the following procedure. Amorphophallus muelleri corms were washed, peeled, shredded, and soaked in different solutions including water (W), sodium bisulfite 1000 ppm (B), salt of sodium chloride 3% (S), sodium bisulfite 1000 ppm, and salt of sodium chloride 3% (BS) for 30 min. Subsequently, the sample was rinsed with 10 liters of water and dried finally dried in an oven at 55 °C for 12 h (UM500; Memmert, Germany). The dried sample was ground by a grinder (HR2115; Philips, Indonesia) and sieved by a laboratory test sieve (D-42757; Retsch Gmbh, Germany) with an aperture of 150 µm. The control sample (C) was prepared from the tuber without soaking in any solutions. All samples were prepared from triplicate treatments.

2.3 FTIR analysis of samples

The FTIR spectra of samples were assayed by using an FTIR Spectrometer ALPHA II (Bruker instrument, Billerica, MA-USA). For each sample, reading was taken three times. The FTIR spectra were processed according to the method of Goodacre et al. (1998) with some modifications. All data were baseline corrected by using Origin Pro Software 2016.

2.4 Morphological properties and mineral analysis by scanning electron microscope- energy dispersive X-ray spectroscopy (SEM-EDS) method

The morphological properties of samples were observed by a Scanning Electron Microscope (SEM) (JEOL JSM IT300, Japan). The mineral content of samples was estimated by surface map analysis using the Energy Dispersive X-Ray Spectroscopy (EDS) feature of the SEM. The sample was mounted on a metal stub then it was coated with gold. An accelerating voltage of 2 kV was used during observation.

2.5 The crystalline structure analysis

The crystalline structures of samples were assayed by using XRD (X-ray Diffraction,, Bruker, Germany) technique using a method of Nakorn et al. (2009) with modifications. The diffractogram of the sample was reported in the 2θ range of 5 to 70°.

2.6 X-ray fluorescence analysis of samples

The calcium content of samples was also evaluated by using the XRF method (XRF Portable Thermo Scientific, type of Niton XL3t 500 analyzers, Thermo Scientific, USA).

2.7 Determination of calcium oxalate by titration method

Calcium oxalate in the sample was determined by the protocol of Mishra et al. (2017) with some modifications. H_2SO_4 of 0.5 N with an amount of 30 mL was added into the sample of 0.5 g in a test tube, then it was heated in a water bath (water bath shaker type 1086; Gesellschaft für Labortechnik (GFL), Germany) at 100 °C for 15 min. The sample then was filtered with Whatman filter paper of number 41 and rinsed with an aqua distillate of 30 mL. Then, 10 mL of filtrate was mixed with 40 mL of H_2SO_4 0.5 N and heated at 100 °C for 5 min. The sample then was immediately titrated with $KMnO_4$ 0.05 N until the titration endpoint was reached as indicated by light red color. Prior to analysis, the $KMnO_4$ 0.05 N was standardized. $C_2H_2O_4.2H_2O$ of 0.0405 gr was mixed with 30 mL of H_2SO_4 0.5 N in a test tube, subsequently, it was heated at 100 °C for 5 min. The standard sample then was titrated with $KMnO_4$ 0.05 N until the titration end point was reached as indicated by light red color. The calcium oxalate content was calculated according to the following Equation 1:

$$\text{Calcium oxalate content}[\%] = \frac{\text{titration volume}(\text{mL}) \text{ x Normality of } KMnO_4 \text{ x Molecular weight of Calcium oxalate x dilution factor}}{\text{weight of sample}(\text{gr})} \quad (1)$$

3 Results and discussion

3.1 FTIR spectra of sample

The FTIR spectra of all samples are presented in Figure 1. According to Derrick et al. (1995), the presence of oxalate salts in a sample can be identified by the FTIR technique. The identity peak of oxalate functional groups including C=O appears at a wavenumber of 1600-1700 cm^{-1} and C-O shows at a wavenumber of 1200 cm^{-1}. The result of this experiment showed that the identity peak of oxalate in four different Amorphophallus flour samples including the C=O group appeared at 1610 cm^{-1} and the peak C-O was observed at 1322 cm^{-1} (Figure 1). Nurlela & Arizal (2020) reported that the C=O groups in the glucomannan structure (Figure 2) show two identity peaks at a wavenumber

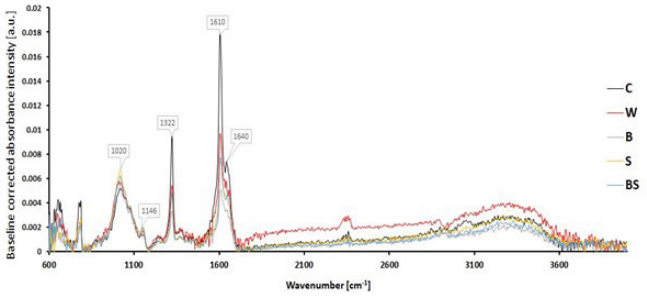

Figure 1. FTIR spectra of Amorphophallus muelleri flour from different treatments (Untreated (C), soaked in water (W), soaked in sodium bisulfite solution (B), soaked in sodium chloride solution (S), soaked in sodium bisulfite-sodium chloride solution (BS).

1739 cm^{-1} and 1736 cm^{-1}. Nurlela & Arizal (2020) also noted that the presence of the C-O group was seen at a wavenumber of 1230 cm^{-1} and 1247 cm^{-1}. Based on the FTIR spectra of oxalic acid (Figure 3), the group of O-H, C=O, and C-O can be identified at wavenumber 3424 cm^{-1}, 1685 cm^{-1}, 1263 cm^{-1}, and 1126 cm^{-1}, respectively (Spectral Database for Organic Compounds, 1999). Shifting of FTIR peaks might be occurred due to the manifestation of gradual changes in the IR frequency associated with a specific chemical bond under the influence of molecular interactions (Ryu et al., 2010).

Figure 1 indicates that the peak intensity of the control sample was the highest among the samples. Meanwhile, the peak intensity of Amorphophallus flour from BS treatment was the lowest one. These results implied that the oxalate content of the BS sample was the lowest among the samples. In terms of preparation of Amorphophallus flour with low oxalate content, the BS treatment can be suggested as the best practice to remove oxalate from the flour compared to other treatments such as soaking in sodium chloride solution (Rofi'ana et al., 2018) and soaking in sodium metabisulfite solution (James et al., 2013). Moreover, a rapid quantitative analysis to determine the oxalate content in Amorphophallus flour might be applied by using an FTIR technique if the calcium oxalate standard is available (Sarifudin et al., 2021).

3.2 Scanning electron micrograph of samples

The SEM images of Amorphophallus muelleri flour samples are shown in Figure 4. Some particles with irregular shapes are observed which could be the agglomerates of flour components including glucomannan, protein, amyloplast, and starch (Takigami et al., 1997). Needle-shaped or raphide crystals are detected in the images of all treatments as indicated by arrows in the image. The length of the raphide is about 150 µm with a width of about 5 µm. Images of samples C and W show a higher number of the raphide crystals compared to those of samples B, S, and BS. Chairiyah et al. (2016) reported four shapes of calcium oxalate crystal found in amorphophallus tuber including styloid, prism, druse, and raphide shapes. Moreover, the raphide shapes of calcium oxalate can be seen by the microscope technique (Ramos et al., 2020). Therefore, in tandem with the microscopy technique, an image of SEM can be an early quick analytical technique to observe the presence of calcium oxalate in the Amorphophallus flour sample (Takigami et al., 1997).

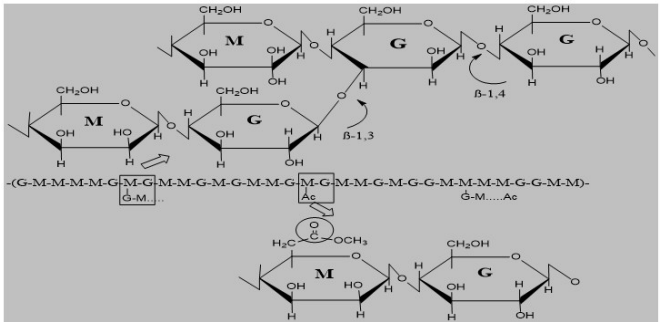

Figure 2. Chemical structure of glucomannan (M: Mannose, G: Glucose) (Behera & Ray, 2016).

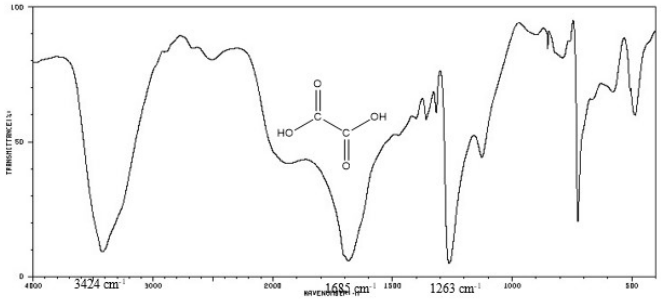

Figure 3. The FTIR Spectra of Oxalic Acid (Spectral Database for Organic Compounds, 1999).

3.3 EDS analysis of samples

The elemental analysis was performed by using an energy-dispersive X-ray spectrometer (EDS) which is a feature of SEM analysis. The reported trace elements are minerals that are concerned in this study including calcium and sodium as shown in Table 1. As expected, the percentage of calcium in the control sample was the highest among the samples. This result indicated that the calcium mineral in Amorphophallus flour is contributed by calcium oxalate. Treatments S and BS left sodium mineral residue in the sample. In terms of calcium oxalate removal, treatment S was found as the most effective one as indicated by the lowest value of calcium. Overall, the result followed the treatments in that the sodium residue were higher in B, S, and BS samples compared to that of C and W samples, whereas the calcium residues of C and W samples were higher than that of B, S, and BS samples (Nurlela & Arizal, 2020; Rofi'ana et al., 2018; Witoyo et al., 2020). Therefore, EDS analysis can be used to evaluate the presence of calcium oxalate in the Amorphophallus flour sample.

3.4 The crystalline structure of samples

X-ray diffractogram patterns of samples are presented in Figure 5. Overall, all samples exhibit X-ray diffractogram pattern of A-type starch as indicated by the presence of its identity peaks at 2θ of 17°, 18.1° and 23.3° (Li et al., 2013; Buléon et al., 1997). The percentage of starch is about 10-30% in Amorphophallus flour (Supriati, 2016). The presence of calcium oxalate crystal can

Table 1. EDS analysis of Amorphophallus muelleri flour from different treatments (Untreated (C), soaked in water (W), soaked in sodium bisulfite solution (B), soaked in sodium chloride solution (S), soaked in sodium bisulfite-sodium chloride solution (BS).

Sample	Na [%]	Ca [%]
C	0.00 ± 0.00*	1.76 ± 0.06
W	0.00 ± 0.00	1.34 ± 0.05
B	0.21 ± 0.06	1.2 ± 0.06
S	10.58 ± 0.16	0.32 ± 0.03
BS	8.64 ± 0.16	0.65 ± 0.04

*Average ± standard deviation.

be detected in the X-ray diffractogram of all samples. Identity peaks of calcium oxalate are shown by the X-ray diffractogram at 2θ of 15.0°, 15.4°, 24.5°, 30.2°, 31.6°, 36.1°, 38.4°, 40.1°, and 43.8° (Ahmed et al., 2012; Orlando et al., 2008). Lastly, the presence of sodium chloride residue from treatment S and BS is also detected by the identity peaks of NaCl crystal at 2θ of 27.4°, 31.7°, 45.5°, 56.5°, and 66.2° (Nickels et al., 1949). Based on this result, XRD can be suggested as a rapid analytical tool to detect the calcium oxalate in the Amorphophallus flour sample.

3.5 X-ray fluorescence analysis of samples

The result of the XRF analysis for the determination of calcium is presented in Figure 6. As expected, the control sample exhibited the highest calcium content (8.87%). Even though the results of calcium content determination by XRF were different from those of EDS, however, their trends were similar. The difference could be caused by the different analytical parameters among the analysis. The X-ray fluorescence is a

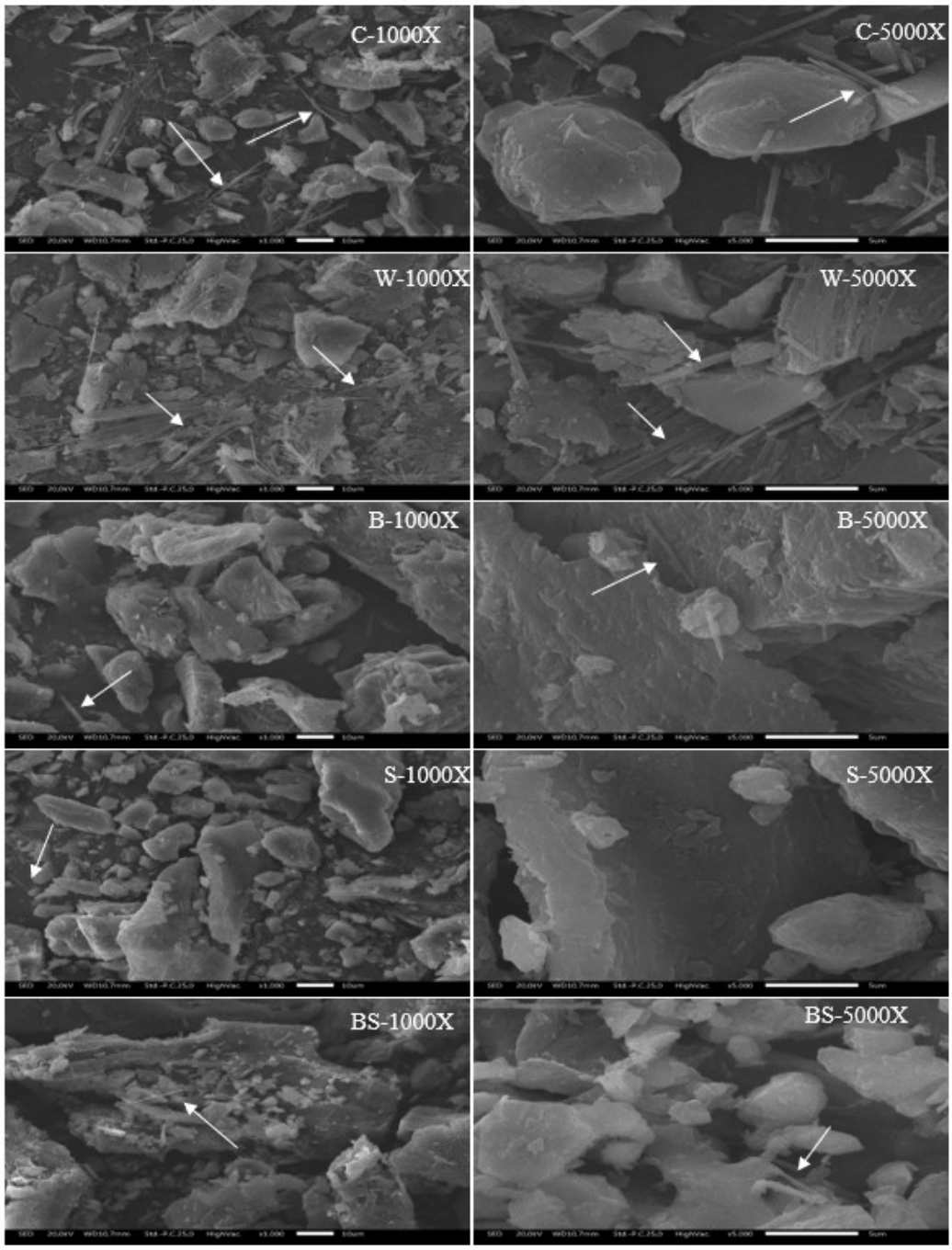

Figure 4. Micrograph of Amorphophallus muelleri flour from different treatments (Untreated (C), soaked in water (W), soaked in sodium bisulfite solution (B), soaked in sodium chloride solution (S), and soaked in sodium bisulfite-sodium chloride solution (BS) in different magnification levels (1000X and 5000X).

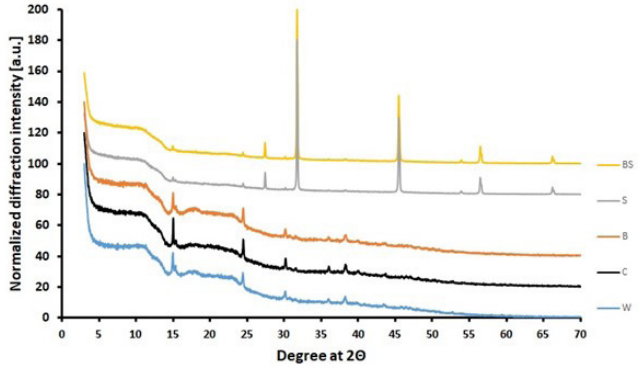

Figure 5. X-ray diffraction patterns of Amorphophallus muelleri flour from different treatments (Untreated (C), soaked in water (W), soaked in sodium bisulfite solution (B), soaked in sodium chloride solution (S), soaked in sodium bisulfite-sodium chloride solution (BS).

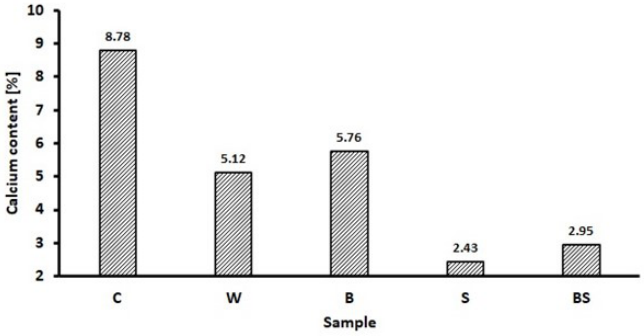

Figure 6. Calcium content of Amorphophallus muelleri flour from different treatments (Untreated (C), soaked in water (W), soaked in sodium bisulfite solution (B), soaked in sodium chloride solution (S), soaked in sodium bisulfite-sodium chloride solution (BS) using XRF method.

fast, low cost, and non-destructive method for determining the concentration of elements in a sample (Peruchi et al., 2014) This technique has been used for screening inorganic nutrients in soybean (Otaka et al., 2014); wheat flour (Peruchi et al., 2014); and cassava (Udoro et al., 2020). Therefore, XRF can be used as an analytical tool to determine calcium oxalate in the Amorphophallus flour sample.

3.6 Calcium oxalate content by titration method

The results of calcium oxalate determination by using the titration method are shown in Figure 7. As expected, the control sample contained the highest percentage of calcium oxalate residue (13.5%). Meanwhile, treatments of S and BS produced Amorphophallus flour with low calcium oxalate content of 6.2 and 6.4%, respectively. Potassium permanganate titration is the most common method to determine calcium oxalate content in food samples (Karamad et al., 2019; Naik et al., 2014). However, this method is limited due to time and chemical consumption. Therefore, other methods are developed in order to overcome the limitation of the titration method in calcium

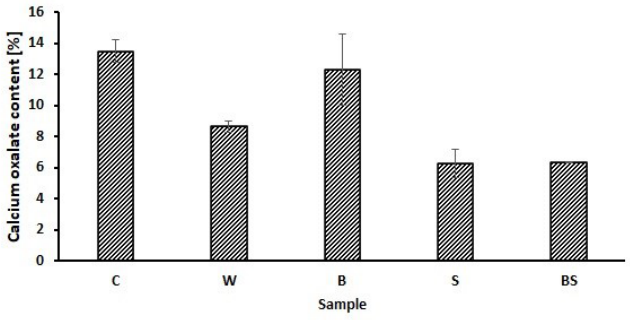

Figure 7. Calcium oxalate content of Amorphophallus muelleri flour from different treatments (Untreated (C), soaked in water (W), soaked in sodium bisulfite solution (B), soaked in sodium chloride solution (S), soaked in sodium bisulfite-sodium chloride solution (BS).

oxalate determination such as capillary electrophoresis (Trevaskis & Trenerry, 1996) and high-performance liquid chromatography (Martz et al., 1990). Despite many limitations of the potassium permanganate titration technique, this method might be used as a comparison method in detecting calcium oxalate in the Amorphophallus flour by other rapid analytical methods. This is because the method can determine total oxalic content in the Amorphophallus flour sample including soluble oxalic acid and non-soluble form of calcium oxalate (Karamad et al., 2019). Prior to the permanganate titration, all oxalic contents in the sample are being solubilized by a strong acid solution i.e. sulfuric acid. The titration method uses the redox principle in which the oxidating agent, i.e. $KMnO_4$, oxidizes the soluble oxalate through titration. The deviation between replicates usually comes from the titration endpoint. In fact, in nature oxalate compounds can be found in dissolved and undissolved forms. Dissolved oxalate is usually formed when oxalate bind with potassium (K^+) ions. In contrast, the undissolved form of oxalate will be formed if oxalate compounds bind with calcium (Ca^{2+}) ions (Chairiyah et al., 2016). Therefore, in this context, the titration method could be a more sensitive method compared to the other instrumental methods.

4 Conclusions

Some analytical instruments have been evaluated in the determination of calcium oxalate in Amorphophallus muelleri flour. The identity peak of oxalate in Amorphophallus muelleri flour can be identified by FTIR technique including C=O group which appeared at a wavenumber of 1610 cm^{-1}. Needle-shaped or raphide crystals of calcium oxalate in Amorphophallus flour can be observed by SEM. The peaks identity of calcium oxalate crystals in X-ray diffractogram were observed at 2θ of 15.0°, 15.4°, 24.5°, 30.2°, 31.6°, 36.1°, 38.4°, 40.1°, and 43.8°. XRF can be used to estimate the calcium oxalate content in Amorphophallus flour based on the determination of calcium minerals. The potassium permanganate titration technique can be used to determine total oxalic in the Amorphophallus flour sample including soluble oxalic acid and non-soluble form of calcium oxalate. Treatment S was found as the most effective

method to remove oxalate one as indicated by the lowest value of calcium content by all analytical methods.

Author contributions

Contributions of each author are listed below:

Achmat Sarifudin: funding acquisition, methodology, validation, writing, and editing.

Lia Ratnawati: supervision, review and editing.

Novita Indrianti: methodology, data curation.

Riyanti Ekafitri: methodology, validation, review, and editing.

Enny Sholichah: methodology, validation, review, and editing.

Nok Afifah: project administration, supervision, review, and editing.

Dewi Desnilasari: supervision, review, and editing.

Pramono Nugroho: supervision, review, and editing.

Annisa Dwi Yuniar: data curation, writing, and editing.

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References

Ahmed, J., Ojha, K., Vaidya, S., Ganguli, J., & Ganguli, A. (2012). Formation of calcium oxalate nanoparticles in leaves: significant role of water content and age of leaves. *Current Science*, 103, 293-298.

Alavi, Z. I., & West, D. B. (1983). Proposed method for the quantitative determination of oxalate in beer and wort. *Journal of the American Society of Brewing Chemists*, 41(1), 24-27. http://dx.doi.org/10.1094/ASBCJ-41-0024.

Al-Ishaq, R. K., Abotaleb, M., Kubatka, P., Kajo, K., & Büsselberg, D. (2019). Flavonoids and their anti-diabetic effects: cellular mechanisms and effects to improve blood sugar levels. *Biomolecules*, 9(9), 430. http://dx.doi.org/10.3390/biom9090430. PMid:31480505.

Behera, S., & Ray, R. (2016). Solid state fermentation for production of microbial cellulases: recent advances and improvement strategies. *International Journal of Biological Macromolecules*, 86, 656-669. http://dx.doi.org/10.1016/j.ijbiomac.2015.10.090. PMid:26601764.

Buléon, A., Gallant, D. J., Bouchet, B., Mouille, G., D'Hulst, C., Kossmann, J., & Ball, S. (1997). Starches from A to C: *Chlamydomonas reinhardtii* as a model microbial system to investigate the biosynthesis of the plant amylopectin crystal. *Plant Physiology*, 115(3), 949-957. http://dx.doi.org/10.1104/pp.115.3.949. PMid:9390431.

Burrows, S. (1950). A colorimetric method for the determination of oxalate. *Analyst*, 75(887), 80-84. http://dx.doi.org/10.1039/an9507500080.

Chairiyah, N., Harijati, N., & Mastuti, R. (2016). Variation of calcium oxalate (CaOx) crystals in porang corms (*Amorphophallus muelleri* Blume) at different harvest time. *American Journal of Plant Sciences*, 07(02), 306-315. http://dx.doi.org/10.4236/ajps.2016.72030.

Coronell Tovar, D., Chavez-Jauregui, R., Bosques-Vega, Á., & Lopez-Moreno, M. (2019). Characterization of cocoyam (*Xanthosoma* spp.) corm flour from the Nazareno cultivar. *Food Science and Technology*, 39(2), 349-357. http://dx.doi.org/10.1590/fst.30017.

Derrick, M., Stulik, D., & Landry, J. (1995). *Infrared spectroscopy in conservation science*. Los Angeles: The Getty Conservation Institute.

Fiske, C. H., & Adams, E. T. (1931). The determination of calcium by alkalimetric titration. I. *Journal of the American Chemical Society*, 53(7), 2498-2501. http://dx.doi.org/10.1021/ja01358a008.

Golozar, A., Khalili, D., Etemadi, A., Poustchi, H., Fazeltabar, A., Hosseini, F., Kamangar, F., Khoshnia, M., Islami, F., Hadaegh, F., Brennan, P., Boffetta, P., Abnet, C. C., Dawsey, S. M., Azizi, F., Malekzadeh, R., & Danaei, G. (2017). White rice intake and incidence of type-2 diabetes: analysis of two prospective cohort studies from Iran. *BMC Public Health*, 17(1), 133-133. http://dx.doi.org/10.1186/s12889-016-3999-4. PMid:28137245.

Goodacre, R., Timmins, É. M., Burton, R., Kaderbhai, N., Woodward, A. M., Kell, D. B., & Rooney, P. J. (1998). Rapid identification of urinary tract infection bacteria using hyperspectral whole-organism fingerprinting and artificial neural networks. *Microbiology*, 144(5), 1157-1170. http://dx.doi.org/10.1099/00221287-144-5-1157. PMid:9611790.

Huang, A. S., & Tanudjaja, L. S. (1992). Application of anion-exchange high-performance liquid chromatography in determining oxalates in taro (*Colocasia esculenta*) corms. *Journal of Agricultural and Food Chemistry*, 40(11), 2123-2126. http://dx.doi.org/10.1021/jf00023a016.

Ilarslan, H., Palmer, R., Imsande, J., & Horner, H. (1997). Quantitative determination of calcium oxalate and oxalate in developing seeds of soybean (leguminosae). *American Journal of Botany*, 84(8), 1042-1046. http://dx.doi.org/10.2307/2446147. PMid:21708659.

James, E. O., Peter, I. A., Charles, N. I., & Joel, N. (2013). Chemical composition and effect of processing and flour particle size on physicochemical and organoleptic properties of cocoyam (*Colocasia esculenta* var. esculenta) Flour. *Nigerian Food Journal*, 31(2), 113-122. http://dx.doi.org/10.1016/S0189-7241(15)30084-9.

Karamad, D., Khosravi-Darani, K., Hosseini, H., & Tavasoli, S. (2019). Analytical procedures and methods validation for oxalate content estimation. *Biointerface Research in Applied Chemistry*, 9(5), 4305-4310. http://dx.doi.org/10.33263/BRIAC95.305310. PMid:33927893.

Keithley, J. K., Swanson, B., Mikolaitis, S. L., DeMeo, M., Zeller, J. M., Fogg, L., & Adamji, J. (2013). Safety and efficacy of glucomannan for weight loss in overweight and moderately obese adults. *Journal of Obesity*, 2013, 610908-610908. http://dx.doi.org/10.1155/2013/610908. PMid:24490058.

Kim, W. K., Chung, M. K., Kang, N. E., Kim, M. H., & Park, O. J. (2003). Effect of resistant starch from corn or rice on glucose control, colonic events, and blood lipid concentrations in streptozotocin-induced diabetic rats. *The Journal of Nutritional Biochemistry*, 14(3), 166-172. http://dx.doi.org/10.1016/S0955-2863(02)00281-4. PMid:12742545.

Kumar, A., Patel, A. A., & Gupta, V. K. (2017). Reduction in oxalate, acridity, phenolic content and antioxidant activity of *Amorphophallus paeoniifolius* var. Gajendra upon cooking. *International Food Research Journal*, 24, 1614-1620.

Li, W., Shan, Y., Xiao, X., Luo, Q., Zheng, J., Ouyang, S., & Zhang, G. (2013). Physicochemical properties of A- and B-starch granules isolated from hard red and soft red winter wheat. *Journal of Agricultural and Food Chemistry*, 61(26), 6477-6484. http://dx.doi.org/10.1021/jf400943h. PMid:23756853.

Martz, F. A., Weiss, M. F., & Belyea, R. L. (1990). Determination of oxalate in forage by reverse-phase high pressure liquid chromatography. *Journal of Dairy Science*, 73(2), 474-479. http://dx.doi.org/10.3168/jds.S0022-0302(90)78694-8.

Minocha, R., Chamberlain, B., Long, S., Turlapati, S. A., & Quigley, G. (2015). Extraction and estimation of the quantity of calcium oxalate crystals in the foliage of conifer and hardwood trees. *Tree*

Physiology, 35(5), 574-580. http://dx.doi.org/10.1093/treephys/tpv031. PMid:25934989.

Mishra, D., Mishra, N., Musale, H., Samal, P., Mishra, S., & Swain, D. (2017). Determination of seasonal and developmental variation in oxalate content of *Anagallis arvensis* plant by titration and spectrophotometric method. *The Pharma Innovation Journal*, 105, 105-111.

Naik, V., Patil, N., Aparadh, V., & Karadge, B. (2014). Methodology in determination of oxalic acid in plant tissue: a comparative approach. *Journal of Global Trends in Pharmaceutical Sciences*, 5, 1662-1672.

Nakorn, K. N., Tongdang, T., & Sirivongpaisal, P. (2009). Crystallinity and rheological properties of pregelatinized rice starches differing in amylose content. *Stärke*, 61(2), 101-108. http://dx.doi.org/10.1002/star.200800008.

Nickels, J. E., Fineman, M. A., & Wallace, W. E. (1949). X-ray diffraction studies of sodium chloride-sodium bromide solid solutions. *The Journal of Physical and Colloid Chemistry*, 53(5), 625-628. http://dx.doi.org/10.1021/j150470a003. PMid:18130456.

Nurlela, A. D., & Arizal, R. (2020). Extraction of glucomannan from porang (*Amorphophallus muelleri* Blume) flour using Ethanol. *Sains dan Terapan Kimia*, 14(2), 88. http://dx.doi.org/10.20527/jstk.v14i2.8330.

Orlando, M. T. D., Kuplich, L., de Souza, D. O., Belich, H., Depianti, J. B., Orlando, C. G. P., Medeiros, E. F., da Cruz, P. C. M., Martinez, L. G., Corrêa, H. P. S., & Ortiz, R. (2008). Study of calcium oxalate monohydrate of kidney stones by X-ray diffraction. *Powder Diffraction*, 23(S1), S59-S64. http://dx.doi.org/10.1154/1.2903738.

Otaka, A., Hokura, A., & Nakai, I. (2014). Determination of trace elements in soybean by X-ray fluorescence analysis and its application to identification of their production areas. *Food Chemistry*, 147, 318-326. http://dx.doi.org/10.1016/j.foodchem.2013.09.142. PMid:24206725.

Peruchi, L. C., Nunes, L. C., de Carvalho, G. G. A., Guerra, M. B. B., de Almeida, E., Rufini, I. A., Santos, D., Jr., & Krug, F. J. (2014). Determination of inorganic nutrients in wheat flour by laser-induced breakdown spectroscopy and energy dispersive X-ray fluorescence spectrometry. *Spectrochimica Acta. Part B, Atomic Spectroscopy*, 100, 129-136. http://dx.doi.org/10.1016/j.sab.2014.08.025.

Ramos, A., Verçosa, R., Teixeira, S., & Teixeira-Costa, B. (2020). Calcium oxalate content from two Amazonian amilaceous roots and the functional properties of their isolated starches. *Food Science and Technology*, 40(3), 705. http://dx.doi.org/10.1590/fst.18419.

Rofi'ana, R., Suedy, S. W. A., & Parman, S. (2018). Effect of soaking of NaCl solution on reduction of calcium oxalate and size of Amylum on Purple Yam (*Dioscorea alata* L.). *Niche Journal of Tropical Biology*, 1(1), 1-6.

Ryu, S. R., Noda, I., & Jung, Y. M. (2010). What is the origin of positional fluctuation of spectral features: true frequency shift or relative intensity changes of two overlapped bands? *Applied Spectroscopy*, 64(9), 1017-1021. http://dx.doi.org/10.1366/000370210792434396. PMid:20828438.

Sarifudin, A., Sholichah, E., Setiaboma, W., Ekafitri, R., Afifah, N., Ratnawati, L., Pudiyanto, E. A., & Achyadi, N. S. (2021). Adulterated powdered white pepper products by tapioca flour sold in Indonesian's online market investigated by simple FTIR analytical method. *Journal of Food and Nutrition Research*, 9(6), 297-303. http://dx.doi.org/10.12691/jfnr-9-6-5.

Shah, B., Li, B., Wang, L., Liu, S., Li, Y., Wei, X., Weiping, J., & Zhenshun, L. (2015). Health benefits of konjac glucomannan with special focus on diabetes. *Bioactive Carbohydrates and Dietary Fibre*, 5(2), 179-187. http://dx.doi.org/10.1016/j.bcdf.2015.03.007.

Siener, R., Seidler, A., & Honow, R. (2021). Oxalate-rich foods. *Food Science and Technology*, 41(Suppl. 1), 169-173. http://dx.doi.org/10.1590/fst.10620.

Singh, A. K., Chaurasiya, A. K., & Mitra, S. (2018). Oxalate content in elephant foot yam (*Amorphophallus paeoniifolius* Dennst-Nicolson) dry and fry cubes. *Journal of Pharmacognosy and Phytochemistry*, 7, 2905-2909.

Spectral Database for Organic Compounds – SDBS. (1999). *Spectral Database for Organic Compounds (SDBS) of oxalic acid.* Japan: National Institute of Advanced Industrial Science and Technology (AIST).

Supriati, Y. (2016). Biodeversity of Iles-Iles (*Amorphophallus* spp.) and its potency for functional food, cosmetics, and bioethanol industries. *Jurnal Litbang Pertanian*, 35(2), 69-80. http://dx.doi.org/10.21082/jp3.v35n2.2016.p69-80.

Takigami, S., Takiguchi, T., & Phillips, G. O. (1997). Microscopical studies of the tissue structure of konjac tubers. *Food Hydrocolloids*, 11(4), 479-484. http://dx.doi.org/10.1016/S0268-005X(97)80046-X.

Trevaskis, M., & Trenerry, V. C. (1996). An investigation into the determination of oxalic acid in vegetables by capillary electrophoresis. *Food Chemistry*, 57(2), 323-330. http://dx.doi.org/10.1016/0308-8146(95)00228-6.

Udoro, E. O., Anyasi, T. A., & Jideani, A. I. O. (2020). Characterization of the root and flour of South African Manihot esculenta Crantz landraces and their potential end-use properties. *International Journal of Food Properties*, 23(1), 820-838. http://dx.doi.org/10.1080/10942912.2020.1759625.

Witoyo, J. E., Argo, B. D., Yuwono, S. S., & Widjanarko, S. B. (2021). A pilot plant scale of Yellow Konjac (Amorphophallus muelleri Blume) flour production by a centrifugal mill using response surface methodology. *Potravinárstvo*, 15, 199-209. http://dx.doi.org/10.5219/1455.

Witoyo, J., Ni'maturohmah, E., Argo, B., Yuwono, S., & Widjanarko, S. (2020). Polishing effect on the physicochemical properties of porang flour using centrifugal grinder. *IOP Conference Series: Earth and Environmental Science*, 475, 012026. http://dx.doi.org/10.1088/1755-1315/475/1/012026.

Yanuriati, A., Marseno, D., Rochmadi, R., & Harmayani, E. (2017). Characteristics of glucomannan isolated from fresh tuber of Porang (Amorphophallus muelleri Blume). *Carbohydrate Polymers*, 156, 56-63. http://dx.doi.org/10.1016/j.carbpol.2016.08.080. PMid:27842852.