PHYSICOCHEMICAL PROPERTIES OF NYPA SALT EXTRACTED FROM DEHYDRATION AND CALCINATION METHOD.

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Abstract

The article explores the extraction of Nypa salt from Nypa frond using two methods, ashing and dehydration method. The dehydration method yielded 15% of salt by heating the fronds at 60°C under a pressure of 60 mbar, while the calcination method yielded 30% of salt by heating the fronds at 60°C and calcinating them overnight at 900°C. The mineral content of the salt extracted through the dehydration and calcination methods showed differences, with the dehydration method having Na (17.7%), Mg (4.4%), Al (4.7%), P (2.8%), Si (7.1%), Cl (44.1%), K (18.6%), and Ca (1.9%), and the calcination method having Na (17.5%), Mg (4.4%), Al (4.8%), P (2.6%), Si (7.0%), Cl (48.9%), K (18.9%), and Ca (2.2%). The colour of the salt produced by both methods was visually distinct, with the dehydration method producing salt with an L* value of 31.934, a* value of 11.327, and a b* value of 23.574, and the calcination method producing salt with an L* value of 52.567, a* value of 7.826, and a b* value of 13.009. The NaCl level obtained from the dehydration method was lower (42.5%) than that of ashing method (44.2%), but the solubility of the salt extracted through the calcination method was higher than that of the salt extracted through the dehydration method. The mass of input material used for extraction was a crucial variable in determining the yield of the product or process.

Keywords: Nypa salt, calcination, dehydration, mineral profile

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1. Introduction

Salt, composed of sodium chloride, is a naturally occurring mineral found in several foods, including milk, eggs, and shellfish [1][2]. Its significance to human civilization is rooted in its use as a flavor enhancer and preservative in food products [3]. Salt has been produced through the boiling of brine sourced from various natural sources such as saltwater, wells, lakes, and salt springs, and mining of rock salt [4][5]. Sodium, an essential element found in salt, plays a crucial role in regulating extracellular fluid volume, nerve conduction, and muscle function [3] [6][7]. However, it is worth noting that while sodium is essential to the body, excessive intake of salt can lead to health complications such as high blood pressure. The World Health Organization (WHO) recommends that adults consume no more than 2 g of sodium per day; however, most adults exceed this amount due to salt being already prevalent in packaged goods, equivalent to about 5 g of salt per day [8][9].

There are several varieties of unrefined edible salts available in the market today, sourced from various geographical origins ([10][11]. These salts often possess unique properties and are associated with magical health effects. However, there is limited information about their composition, which can lead to incorrect and fraudulent information being circulated online. The chemical composition of these salts varies based on their geographical origin and synthesis methods, which can be utilized for classification purposes. For instance, Nypa salt is a traditional Sarawak dish, locally known as "garam apong," which is made using ancestral methods and Nypa fruticans. However, there is limited knowledge about the physical and chemical properties of Nypa salt, and no standard extraction methods have been established. This study aims to address this gap by investigating the physical and chemical properties of Nypa salt and presenting a traditional and scientific method of extracting salt from Nypa frondlet.

The research involves the extraction of Nypa salt using two methods, calcination (ashing) and dehydration, and the investigation of the physicochemical properties of the extracted salt. The obtained data will serve as a basis for further research into the use of Nypa salt in various fields, including food, cosmetics, and others. Additionally, the study will recommend a standard and scientific method of extracting salt from Nypa frondlet. Thus, the primary objective of this research is to provide scientific information on the physical and chemical properties of Nypa salt, which will be beneficial for future studies on its usage in different fields

2. Materials and Methods

2.1 Extraction of Salt

Two methods of salt extraction will be employed in this study; ashing method, dehydration method.

2.1.1 Calcination (Ashing) method

Mature Nypa frondlets will be harvested and cut into smaller chunks, and oven-dried for 24 hours. Once dried, the frondlets chunks will be burned in furnace overnight, at 550°C. The ashes produced then will be boiled in a beaker of water. The debris floats to the surface will be removed using strainer as the liquid boils. The liquid will then be constantly stirred to evaporate the water. The salt will solidify at the bottom of the beaker forming a cake of salt.

2.1.2 Dehydration method

Mature Nypa frondlets will be harvested. Using sugarcane juicing machine, the 'Nypa juice' will be extracted. Once the juice is collected, it will then be filtered using strainer to remove the debris and foreign matters. Then, the juice will be boiled. During boiling process, the juices will be constantly stirred. The juice will then be filtered using a 500 micron mesh cloth and filter paper number 4 and dried at 60°C for 24 hours

2.2 Determination of Yield (%)

Yield of salt will be calculated using the formula; Yield (%) = (weight of extracted salt/weight of frondlet) x 100

2.3 pH Analysis

The pH of salt solution (1% w/v) will be determined using pH meter.

2.4 Mineral Profile Analysis

Following microwave-assisted acid digestion, the mineral content was determined using inductively coupled plasma mass spectrometry (ICP-MS). All glassware was cleaned with a 10% (v/v) HNO3 solution for one day before being rinsed with ultrapure water. 1.0 g of each sample was digested in PTFE vessels with 4.0 mL of 65% (v/v) HNO3 and 0.5 mL of 35% (v/v) H2O2. The vessels were placed in the microwave system (MARS 5, CEM). In the same way, a blank digest was performed. Digestion conditions for the microwave system used were as follows: up to 120°C for 15 minutes and then constant for 10 minutes; up to 160°C in 20 minutes and constant for 15 minutes; and finally, a cooling stage (30 minutes) was performed to 22°C and diluted to 50 mL with deionized ultrapure water. This solution was then used for elemental analysis using an ICP-MS equipped with a concentric Nebulizer, a quartz torch with quartz injector tube, and a cyclonic spray chamber. The concentrations of 8 elements (Na, Mg, Al, P, Si, Cl, K and Ca) in Nypa salt samples were determined. All samples were analysed in duplicate, and each sample was measured in triplicate using ICP-MS detection.

2.5 Solubility Analysis

100 mg of salt samples will be dissolved in 10 mL of deionized water, and then the salt solutions will be mixed for overnight in water bath at 25°C and 200 rpm. After dissolving, the solutions will be passed through the filter paper (Whatman No. 3, Whatman International Ltd.) preweighed. The filter papers will be dried in dry oven at 80°C and was reweighed. The solubility will be calculated from the residue content in the filter paper to the salt content in the filtrate.

2.6 NaCl Content Analysis

The analysis of NaCl follows the previously described method [12]. The analysis will be carried out by putting the sample into furnace until turning into ashes. 250 mg of samples will be washed with 10 mL of distilled water and will be transferred to 250 mL Erlenmeyer flask. 3 mL of 5% potassium chromate (K_2CrO_4) solution will then be added and the mixture will then be titrated with silver nitrite ($AgNO_3$) 0.1 M. The endpoint of the titration is reached when the orange colour arises. NaCl level will be calculated by the following formula: salt (NaCl) (%) = (T x M x 58.4)/W (mg) x 100% T = Standard solvent volume $AgNO_3$ 0.1 M M = Molarity of silver nitrite W = Sample weight

2.7 Colour Analysis

The colour of each sample will be determined using a colourimeter (Minolta Chroma meter CR-210, Minolta Ltd., Japan; illuminate C, calibrated with a white plate, L*=+97.83, a*= −0.43, b*=+1.98). Lightness (CIE L*-value), redness (CIE a*-value), and yellowness (CIE b*-value) values will be recorded

2.8 Statistical Analysis

All tests will be done at least three times for each experimental condition and mean values will be reported. One way analysis of variance will be performed on all the variables measured. Duncan’s multiple range test ($p<0.05$) will be used to determine the differences among treatments.

3. Result and Discussion

The extraction of salt from Nypa frond has been successfully carried out using two different methods: dehydration and calcination. Table 1 shows the physicochemical properties of Nypa salt extracted from dehydration and calcination method.

Table 1: Physicochemical properties of Nypa salt extracted from dehydration and calcination method.

Physicochemical Properties	Extraction Methods	
	Dehydration	Calcination (ashing)
Yield (%)	15.0	30.0
pH	8.37	9.11
Mineral content		
Na	17.7	17.5
Mg	4.4	4.8
Al	4.7	4.4
P	2.8	2.6
Si	7.1	7.0
Cl	4.1	48.9
K	18.6	18.9
Ca	1.9	2.2
Solubility	97.6	96.8
NaCl level (%)	42.5	44.2
Colour		
L*	31.934	52.567
a*	11.327	7.826
b*	23.574	13.009

3.1 Yield (%)

Referring to the Table 1, the dehydration method involved heating the frond at 60°C and under a pressure of 60 mbar, which yielded 15% of salt. On the other hand, the calcination method involved heating the frond at 60°C and then calcinating it overnight at 90°C, which yielded 30% of salt.

Generally, the yield percentage of extracted salt is directly proportional to the amount of starting material used for the extraction process. Therefore, if a larger amount of starting material is used, the yield percentage will also increase.

Both method utilized mature Nypa fronds with the similar size. In the case of the calcination method, the starting material used is the mature Nypa fronds, which are burned overnight at 900°C. This burning process will reduce the mass of the starting material and increase the concentration of salt in the remaining ash. On the other hand, the dehydration method used the Nypa juice squeezed from the Nypa frond, which has a smaller starting material compared to the mature Nypa fronds used in calcination. The smaller starting material may have contributed to the lower yield percentage obtained from the dehydration method.

The mass of input material is a crucial variable in determining the yield of a product or process. [13] demonstrated that the yield of Citronella Oil obtained by the Microwave Hydrodistillation (MHD) method was affected by several parameters, including microwave power, material size, and extraction time. The smaller size of the material resulted in a lower yield of oil obtained, while the longer extraction time resulted in higher yields.

3.2 Mineral Content

The mineral content of the salt extracted through dehydration and calcination methods showed some differences as shown in Table 1. The mineral content of the salt extracted through the dehydration method was Na (17.7%), Mg (4.4%), Al (4.7%), P (2.8%), Si (7.1%), Cl (44.1%), K (18.6%), Ca (1.9%). In comparison, the mineral content of the salt extracted through calcination method was Na (17.5%), Mg (4.8%), Al (4.4%), P (2.6%), Si (7.0%), Cl (48.9%), K (18.9%), Ca (2.2%).

The differences in mineral content can be attributed to the differences in extraction methods used. The calcination method involved heating the sample at high temperature of 900°C for an extended period, leading to the release of some volatile minerals like magnesium and sodium, as well as other minerals from the salt. The dehydration method involves heating the sample at a lower temperature of 60°C, which is not enough to release volatile minerals like magnesium and sodium.

The calcination process may have caused a reduction in micro mineral concentration in the extracted salt. This reduction in micro mineral concentration may be due to the high temperature and prolonged calcination time, which can lead to the breakdown of mineral compounds and the loss of volatile components [14].

Moreover, the variation in the mineral contents could also be due to the differences in the salt matrix's chemical composition. The matrix's chemical composition may affect the solubility of some minerals, leading to their variation in the final product. This is in agreement with the finding [15]. The study investigated the physicochemical properties of salt samples collected from four saline lakes and surrounding playas in the Qaidam Basin, focusing on the chemical compositions and hygroscopicity. The salt samples included brines, lakebed salts, crust salts, and crystallized brines. The results showed that the natural solid salts, including lakebed salts and crust salts, had very distinct composition differences compared to the complex mineral compositions of brines.

3.3 Colour

In the dehydration method, the salt has an L* value of 31.934, a* value of 11.327, and a b* value of 23.574 as depicted in Table 1. In contrast, the calcination method produced salt with an L* value of 52.567, a* value of 7.826, and a b* value of 13.009. The difference in colour values indicates that the salts produced by both methods are visually distinct.

The colour difference observed in the two salts may be attributed to the production process. The calcination method involves heating the sample at high temperature for an extended period, which may result in the destruction of pigments present in the sample, leading to a change in colour. On the other hand, the dehydration method involves heating the sample at a lower temperature and pressure, which may not significantly affect the pigments present in the sample [16]. In a study investigating the effect of pigments on the strength of ceramic bricks, it was found that the introduction of a pigment in the amount of 2–4% results in a rich dark brown brick. At the same time, the intensity of the colour increases with an increase in the pigment concentration from 2 to 4% and a brick firing temperature from 800 to 950°C. It has been established that with the introduction of pigment, the strength of the obtained brick samples decreases, as well as the frost resistance [16]. Another study investigated the effect of processing temperature on pigments and colour of spinach. The study found that colour measurements indicated a difference of 4.1 units between the controls and the samples processed at 280°F, and 11.6 units between the controls and the samples processed at 240°F. The pigment changes indicated a progressively smaller change in [17]. Therefore, it can be concluded that the production process, specifically the temperature and duration of heating, can significantly affect the pigments present in a sample, leading to a change in colour.

3.4 pH

As shown in Table 1, the pH of the salt extracted using the dehydration method was 8.37, while the pH of the salt extracted using the calcination method was 9.11.

In the case of the salts extracted from Nypa fronds, it is likely that the difference in pH is due to differences in the mineral content of the salts. The calcination method may have produced a salt with a higher concentration of alkaline minerals, such as potassium and magnesium, which could have contributed to the higher pH value. Conversely, the dehydration method may have produced a salt with a lower concentration of alkaline minerals and a higher concentration of acidic minerals, such as chloride, which could have contributed to the lower pH value. This trend is in line with the aforementioned mineral content (Table 1) in Nypa salt extracted from both method in which the calcium, magnesium and potassium were higher in the salt produced from calcination method.

Calcium, magnesium, potassium, and iron can have alkaline properties and can contribute to the alkalinity of a solution or environment [18,19,20,21,22,23]. Magnesium and calcium, which are alkaline earth metals, and iron and magnesium, which are common metals, have a great effect on reforming [18]. Both mineral also provide buffering effects upon pH which can contribute to the alkalinity of a solution [19] and commonly found in metal ion-binding proteins [20]. On the other hand, plant biomass naturally contains alkali and alkaline earth metals, including potassium, sodium, phosphorus, calcium, and magnesium, which can affect biomass degradation and product distribution [21]. Moreover, a study suggests that the alkaline properties of calcium, sodium bicarbonate, and potassium in a snack can cause an increase in saliva pH [22]. Calcium, sodium, potassium, and alkaline earth metal oxides in the micropores of concrete can generate strong alkaline hydroxide with water in porous by chemical reactions, which can contribute to the alkalinity of the environment around rebar [23].

3.5 NaCl level

The difference in NaCl level (Table 1) obtained from the dehydration method (42.5%) and the ashing method (44.2%) could be due to the different extraction efficiencies of the two methods.

The ashing method involves the complete combustion of the sample, which could lead to the release of bound NaCl that may not be extracted by the dehydration method . This is in agreement with two studies, both suggesting that the salt treated with higher temperature resulted in higher level of NaCl. Therefore, the ashing method may be a more accurate method for determining the NaCl content in food samples [24,25].

3.6 Solubility

The solubility of salt extracted using the dehydration method was found to be 97.6% at 1% concentration and 25°C, while the solubility of salt extracted using the calcination method was 96.8% at the same concentration and temperature . The difference in solubility level of salt extracted from both methods can be attributed to the difference in the chemical composition of the salt. The dehydration method involves the removal of water from the salt, resulting in a higher concentration of salt. On the other hand, the calcination method involves heating the salt to high temperatures, which can cause the salt to decompose and form new compounds, resulting in a lower solubility level. In a study discussing on the influence of heat treatment on the physicochemical property and mineral composition of various processed salts, the lowest solubility of bamboo salt was attributed to the generation of oxide salts due to high temperature treatment above melting point of NaCl [24].

4. Conclusion

It can be concluded that both dehydration and calcination methods can be used for the extraction of salt from Nypa frond. The calcination method produced a higher yield of salt at 30% compared to the dehydration method's yield of 15%. The mineral content of the salt extracted through the calcination method showed a higher concentration of chloride and calcium than the dehydration method. However, the dehydration method showed a higher concentration of magnesium and sodium than the calcination method. The colour of the salt produced by the two methods was visually distinct, with the calcination method producing salt with a lighter colour than the dehydration method. The differences in mineral content and colour can be attributed to the differences in the extraction methods used. Therefore, the choice of extraction method should be based on the desired characteristics of the extracted salt. Furthermore, the yield percentage and mineral content of the extracted salt are directly proportional to the amount of starting material used.

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