The Effect of Sonication Time on Physicochemical Profiles of The Nanocalcium from Snake-Head Fish Bone (Channa striata)

AB Tawali¹, N Wakiah¹, K Qanitah¹, M Asfar¹, NK Sukendar¹ and Metusalach²

¹Food Science and Technology Study Program, Faculty of Agriculture
²Fisheries Resources Utilization Study Program, Faculty of Marine Science and Fisheries Hasanuddin University, Makassar, Indonesia

E-mail: wakiahn13g@student.unhas.ac.id

Abstract. Snake-Head Fish bone is one of the fishery wastes that is potentially used as the raw material for calcium supplement products. In order to make the calcium products easily absorbed and utilized rapidly by human body, supplement products need to be made in nano form. The aims of this study were to determine the effect of the sonication time in the production of Snake-Head Fish bone nano calcium on the physical profile of the resulted nano calcium, and to obtain the best sonication time. The development of nanocalcium utilized sonication method with 405 Powersonic Ultrasonic Cleaner. The result showed that the best particle size distribution was found on 45 minutes sonication treatment which was 842.3 nm with the highest particle size ranging from 461.6 to 487.5 nm. The average value of the polydispersity index is 1.527. The yield of nano calcium produced from whole fish is 1.331%. The resulting nano calcium has an average white degree of 86.622% and average density of 0.73 g / ml. Some other mineral content also showed a not significant difference.

1. Introduction

Snake-Head fish is a type of carnivorous fish that is commonly found in Indonesian freshwater. According to statistics data, the production of Snake-Head fish was farmed in 2013 as much as 14,250 tons and increased in 2014 to reach 17 847 tons, while from fishing in 2013 as many as 61 401 tons and increased in 2014 amounted to 63 409 tons [1]. The use of these fish is very high, especially in the food and pharmaceutical industries. Snake-Head fish is used in the food industry as raw material for making fillets and it is commonly used as an albumin supplement extracted from Snake-Head fish meat.
Figure 1. Snake-Head Fish (Channa striata)[2]

The development of the Snake-Head fish processing industry causes increase in the product of waste, especially fish bones. Snake-Head fish, which is rarely used but rich in minerals, especially calcium, also rich in phosphorus, collagen and amino acids [2]. Snake-Head fish bone has a natural source of calcium, for example raw material for making calcium mineral source supplements products. Calcium is generally available in the form of micro (µ) which is not optimally absorbed in the body's metabolism. In order to make the calcium products easily absorbed and utilized rapidly by human body, supplement products need to be made in nano form.

Sonication is one method of making nanocalcium. It is one of the top down methods which used to produce materials with nanometer (nm) form. The principle of sonication method is using ultrasonic waves with a frequency range of 20 MHz to 10 MHz which is fired into a liquid medium to produce cavitation vesicle that can make particles have a diameter on the nanoscale [3]. Using ultrasonic waves is very effective in making nano materials. This method is quite simple and requires a relatively short time. Based on these, The aims of this study were to determine the effect of the sonication time in the production of Snake-Head fish bone nano calcium on the physical profile of the resulted nano calcium, and to obtain the best sonication time.

2. Materials and Method

2.1. Snake-Head Fish Bone Powder Preparation
Making snake-head fish powder begins with cleaning snake-head fish and steamed for 15 minutes. Then the bone was pressure cooking using a Signora Pressure Cooker (t: 30 minutes). After that, the fish bone is dried with an oven blower (T: 65°C & t: 2.5 hours). Bones that have been dried, and smoothed using a grinder [4].

2.2. Preparation of Snake-Head Fish Bone Powder Nanocalcium Form
Fish bone powder was dissolved in distilled water using 1:10 then homogenized and sonicated using 405 Powersonic Ultrasonic Cleaner for 15, 30, 45, and 60 minutes. Then dried with freeze drying for 16 hours. After that, it smoothed with a grinder and sifted with a 100 mesh sieve.

2.3. Psychochemical Analysis
The physicochemical properties test was carried out on the best sonication time samples (Particle Size Analyzer (PSA) while the physicochemical testing included the determination of the yield of whole fish, Bulk density of using a measuring cup, the whiteness using a colorimeter, moisture and mineral content (AOAC).

3. Result and Discussion
Particle size is the most important part of nanoparticle. It plays an important role in determining particle distribution in vivo, biological conditions, toxicity and capabilities target of the system, and affect the calibration system and its stability [4]. The best time of sonication in making nanocalcium is based on the size of the smallest particles produced. The test results can be seen in figure 02. Figure 02 shows the best time treatment is in samples with 45 minutes sonication time. The average particle size distribution is 682.1 nm with the largest particle size of 487.5 nm. The size of particles can be classified as nanoparticles because nanoparticles are particles measuring 10-1000 nm [4].

Based on these results, sample Q1 (15 minutes) has the lowest PI value while sample Q1(0 minute) has the highest PI value. The value obtained shows the distribution of particles are not homogeneous because of the average value obtained is > 0.7. Based on these results, the homogeneity of the sample is occurrence of aggregation or clumping. Aggregation becomes a very important factor in the measurement process using the PSA method. In the measurement of PSA, the particle measured in liquid media which is dispersed in dispersant. This dispersion condition greatly affects the PSA reading. In
addition, the imperfect drying process also affecting nanocalcium which not completely dispersed in the dispersant of distilled water. So that occurs results in inconsistent particle size with greater readability in PSA testing.

![Figure 2. Average Size of Nanocalcium Particle Distribution](image)

The polydispersity index is a parameter used to describe the level of non-uniform particle size distribution. This parameter is closely related to testing of particle distribution, which value is closer to 0.0 then the sample will be more uniform. The polydispersity index value of 0.0 indicates that the sample has a perfectly uniform particle size [5]. The range of values from 0.1 to 0.25 shows a narrow size distribution, while a value of more than 0.7 shows a broad distribution.

![Figure 3. Polidispersity Index of Nanocalcium Particle](image)

The yield of fish bone nanocalcium is the initial weight of whole fish. The yield parameter is an important parameter for knowing the economic value and effectiveness of a product. Recovery Snake-Head Fish bone is clean after being removed from the rest of the meat, mucus, and the blood attached to the bone is 5.544% (bk). After being dried and mixed with the yield to 1.423% (bk) then after becoming a Snake-Head Fish nanocalcium, the yield becomes 1.387% (bk). These results indicate that the treatment yields by sonication for 45 minutes does not affect the yield of bone before it was made in nano form.
Whiteness analysis to measure the white color of a material. The white powder is generally more often used for fortification in various food products. Results The average value of white fish bone flour was 88.601% while bone fish meal was 86.622%. These results indicate that fish bone flour and nanocalcium flour have a color that is clearly seen as white powder. The whiteness of organic composition available in powder. High organic components will reduce the colors of powder [7].

Bulk density is a measure of the amount of mass of material per volume it occupies, including empty space between materials. If the density value is smaller, the material requires a larger volume for a smaller amount of material. Bulk foodstuffs will require a wider area compared to materials with a large density of fish for the same weight. [8]. The average yield of fish bone flour Bulk density value obtained is 0.97 g / ml while the density value of fish bone nanocalcium is 0.73 g / ml. Based on these results, fish bone nanocalcium flour has a smaller Bulk density value than fish bone flour. This is because the particle size of the nanocalcium is smaller and the mass is lighter so it will require a larger volume in the same weight.

![Figure 4. Whiteness of Fish Bone Powder](image)

![Figure 5. Bulk Density of Fish Bone Powder](image)
The moisture content per unit weight of material is indicated. The moisture content is the most fundamental parameter because it affects the shelf life of the product. Based on the results obtained by the water content of cork fish nanocalcium is 11.78% while the fish bone flour is 9.39%. Corkfish nanocalcium flour has a higher water content compared to cork fish bone flour without nano size. This is due to the different types of drying where bone flour uses an oven blower while the nanocalcium uses a freeze-drying dryer. Both of these results when compared with SNI 01-3158 1992 regarding fish bone flour for feed have a moisture content value above SNI which is a maximum of 8% [9].

About 4% of our body consists of minerals, which in the analysis of food ingredients are left as ash content, which is left over if a sample of food is burned perfectly in a furnace (muffle furnace) [10]. Based on the results obtained, calcium minerals in snake-head fish powder that have been made in nano size are greater than calcium in snake-head powder without nano size. Some other mineral content also showed a not significant difference.

![Figure 6. Mineral Content of Fish Bone Powder](image)

**Figure 6. Mineral Content of Fish Bone Powder**

4. Conclusion
It was concluded that the best sonication time for making snake-head bone fish powder in nanocalcium form is 45 minutes. It was presented 842.3 nm with the highest particle size ranging from 461.6 to 487.5 nm. The average value of the polydispersity index is 1.527. The yield of nano calcium produced from whole fish is 1.331%. The resulting nano calcium has an average white degree of 86.622% and average density of 0.73 g / ml. Some other mineral content also showed a not significant difference.

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