Alternative Use of Tamarind Pretreatment in Making Eggshell Flour

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Abstract

The use of acid pretreatment in the manufacture of eggshell flour is an alternative way to speed up drying time, reduce product odor, and facilitate the absorption of nutrients when consumed. Tamarind is one type of acidifying agent that is widely available around, both sold commercially and naturally. This study aims to examine the differences in the use of various types of tamarind pretreatment in the process of making eggshell flour. The type of acid used as pretreatment was Tamarind in Lombok, Kalimantan, and Java. The method of this research is to soak egg shells with various types of tamarind mentioned above for 3 (three) hours. Parameters observed were proximate analysis (water content, ash, fat, protein, crude fiber, carbohydrates) and minerals (calcium and phosphorus), pH and yield. The results show that there are differences in the administration of acid pretreatment to the observed parameters. The results of the treatment showed that the best proximate value was obtained in the Lombok tamarind pretreatment and the main control, as well as the yield and calcium levels. The best value of phosphorus content was in the use of Lombok tamarind pretreatment and the main control.

Key word : acid pretreatment, eggshell flour, tamarind

Introduction

Eggs are an almost perfect source of animal protein. Chicken eggs are perfect food ingredients that contain nutrients such as protein (12.8%) and fat (11.8%). In 100 grams of whole eggs also contain vitamin A of 327.0 SI and minerals of 256.0 mg. Eggs contain high-quality protein because they have a complete composition of essential amino acids and have a high biological value, which is 100% (Kurniawan et al., 2014). Thus, one of the sources of animal food that is cheap and easily available is eggs. Egg waste is the impact of community consumption activities. Egg shell is part of the waste that has not been handled properly. The contribution of waste from egg shells also comes from hatcheries and food processing industries (Mittal et al., 2016).

Egg shell waste is one of the waste that has not been handled. The hatchery industry in America produces the largest shell waste of 140,000 tons/year (Mahmud et al., 2015). Potential uses of eggshell waste include handicrafts (Glatz et al., 2011), animal feed (Abiola et al., 2012; Nurlaela et al., 2014), fertilizer (Ernawati et al., 2019) and calcium fortification in cookies (Rahmawati & Nisa, 2015). Egg shell waste has a negative impact, if there is no treatment. Environmental pollution, odors, flies, and problems with eggshell waste disposal and zoonoses can occur, namely diseases that are transmitted from animals

to humans and vice versa (Glatz et al., 2011; Khairiyah, 2016, Orrico et al., 2020,) which pose a threat for human health.

Acid pretreatment is a pre-treatment method by soaking food ingredients in a solution of acidifying agents. Acid pretreatment has many benefits, namely being able to speed up drying time, improve organoleptic materials, remove inhibitor compounds, and especially egg shells can eliminate fishy odors and bacteria present. The building block in this research is tamarind. Tamarind is widely found in the surrounding environment. Tamarind fruit (*Tamarindus indica L.*) has long been used as a spice in cooking, beverage products, sweeteners, and traditional medicines that are useful for health. Tamarind fruit has a health function for the body because it acts as an antidiabetic, antihyperlipidemic, antioxidant, and content that inhibits bacterial growth, namely the active substances of saponins, flavonoids, alkaloids and tannins (Utami, 2011).

This study aims to see the difference in the nutritional value of eggshell flour which is dried with acid pretreatment, namely tamarind as an introduction. The type of acidifying agent in the acid pretreatment in this study was tamarind from several regions: Lombok, Kalimantan and Java.

Materials and Methods

The study was conducted from August to October 2022. The production of eggshell flour with tamarind pretreatment, pH measurements and yield were carried out at the Livestock Production Laboratory and Agricultural Production Laboratory, Antakusuma University Pangkalan Bun, Central Kalimantan. Tests for proximate, calcium and phosphorus minerals were carried out at the Laboratory of Animal Science Production, Lambung Mangkurat University, Banjarmasin. The type of acid used as an acid pretreatment in this study was tamarind originating from Lombok, Java and Kalimantan.

The research was carried out by collecting egg shells and cleaning the dirt attached to the egg shells with running water. The egg shells were then boiled at a boiling temperature of 100^{o} C for 30 minutes. This serves to eliminate salmonella microbes. Boiled egg shells are then drained and soaked. Acid pretreatment is soaking egg shells with an acid solution, namely tamarind from Lombok, Kalimantan and Java with a concentration of 0.5%. Then, each treatment was soaked for 3 hours. After acid pretreatment, the egg shells were drained and cooled. The egg shells were then dried in the oven for 4 hours at 95^{o} C. Refinement of size is done by blending the flour and sifting through an 80 mesh sieve. The last stage is to shell eggs in the form of flour packaged by means of a sealer.

The process of making eggshell flour with tamarind pretreatment starts from cleaning, boiling, drying process to size refinement. The boiling process aims to eliminate salmonella sp. Boiling at 100^{o} C for 15-20 minutes is effective enough to kill salmonella sp. (Rahayu, 2017). The drying stage is a method for removing or removing some of the water from a material by evaporating the water using heat energy. Drying is the process of reducing the water content in food as a result of heat and mass transfer of water either by conduction, convection or radiation (Maisnam et al., 2016). In general, the advantage of this preservation is that the material becomes durable with a small volume of material making it easier to transport. The purpose of drying is to reduce the water content of the

material to a limit where microorganisms and enzyme activities that can cause spoilage will stop, thus the dried material can have a long shelf life (Riansyah et al., 2013).

Results and Discussion

The results of the proximate analysis can be seen in Table 1.

Table 1. Analysis of Proximate Levels of Eggshell Powder

Variable (%)	Eggshell Flour *Pretreatment* Tamarind Java Lombok	Eggshell Flour *Pretreatment* Tamarind Java	Eggshell Flour *Pretreatment* Kalimantan Tamarind	Eggshell Flour Control
Crude Fat	0.33 ± 0.0057 [a]	0.500 ± 0.0000 [b]	0.433 ± 0.0577 [b]	0.580 ± 0.0026 [c]
Crude Protein	3.2367 ± 0.0152[a]	3.4433 ± 0.3512[b]	3.2733 ± 0.6658[a]	3.9933 ± 0.1528[v]
Crude Fiber	1.7233 ± 0.0152[a]	1.8733 ± 0.2082[b]	1.7400 ± 0.3606[a]	1.8900 ± 0.1000[b]
Ash	79.31 ± 0.900[a]	81.3067 ± 1.04314[b]	79.9667 ± 1.5631[b]	91.2767 ± 0.5462[c]
Carbohydrat	15.3867 ± 0.0750[d]	12.9600 ± 1.01681[c]	14.6233 ± 1.5567[c]	2.2953 ± 0.553[a]
Water content	0.3100 ± 0.0100[a]	0.3667 ± 0.1528[b]	0.3533 ± 0.0577[b]	0.4900 ± 0.0100[c]

Note: The average value marked with a different letter in the same line shows a significant difference ($P<0.05$)

Moisture content is affected by drying during oven. Oven will reduce the water content so that the material gains a constant weight, namely through the release of free water on the material that evaporates and leaves bound water. According to Hariyadi (2018), free water is present in tissues, while bound water is usually found in cells. Other factors that play a role are the physical and chemical properties of the material being dried, the size of the material, the shape of the material and the initial composition of the moisture content (Nurmala et al., 2021). Measurement of water content is one of the main parameters in the manufacture of flour, this is because the water in the food ingredients greatly determines the freshness and durability of the material because the water content is related to the development of microorganisms in the product (Winarno, 1997). Thus, eggshell flour will have a long shelf life due to its low water content. This is because the low water content will make it difficult for microbes to grow so that eggshell flour becomes durable. The standard used in determining the water content of eggshell flour is (Indonesian National Standart) SNI 01-3751-2006 regarding wheat flour, the maximum moisture content is 14.5%. From the observation, it can be seen that the water content of eggshell flour has met the standard. The drying process which aims to reduce the water content in eggshell flour will affect the content of other ingredients. The explanation of Sani (2000) supports this statement, namely that the reduction in water content will have

an impact on the concentration of protein, carbohydrates, fats and minerals in eggshell flour. On the contrary, the drying process in the manufacture of eggshell flour will then have an impact on the lower levels of vitamins and dyes.

Table 2. Calcium and Phosphorus Levels in Eggshell Flour

Variable (%)	Eggshell Flour *Pretreatment* Tamarind Java Lombok	Eggshell Flour *Pretreatment* Tamarind Java	Eggshell Flour *Pretreatment* Kalimantan Tamarind	Eggshell Flour Control
Calcium (%)	85,0100 ± 0,1652[a]	86,6200 ± 3,3645[b]	85,2300 ± 2,2517[a]	91,0833 ± 0,4649[c]
Phosphorus (%)	0,8467 ± 0,0550[b]	0,8667 ± 0,0577[b]	0,7833 ± 0,2082[a]	0,9567 ± 0,0577[c]

Note: The average value marked with a different letter in the same line shows a significant difference($P<0.05$)

Table 3. Yield of Eggshell Flour

Variable (%)	Eggshell Flour *Pretreatment* Tamarind Java Lombok	Eggshell Flour *Pretreatment* Tamarind Java	Eggshell Flour *Pretreatment* Kalimantan Tamarind	Eggshell Flour Control
Yield (%)	86,6300 ± 0,2951[a]	88,4500 ± 0,5294[b]	88,4700 ± 0,1179[b]	91,8100 ± 0,5624[c]

Note: The average value marked with a different letter in the same line shows a significant difference ($P<0.05$)

Table 4. Eggshell Flour pH

Variable (%)	Eggshell Flour *Pretreatment* Tamarind Java Lombok	Eggshell Flour *Pretreatment* Tamarind Java	Eggshell Flour *Pretreatment* Kalimantan Tamarind	Eggshell Flour Control
Ph	8,2967 ± 0,3055[a]	8,4867 ± 0,1528[c]	8,3900 ± 0,1000[b]	9,2500 ± 0,3606[d]

Note: The average value marked with a different letter in the same line shows a significant difference ($P<0.05$)

Drying in the oven for ± 4 hours produced different water content, the lowest water content was tamarind (Lombok) pretreatment eggshell flour, which was 0.31% and the highest was 0.49% in the control. Acid pretreatment causes the cell wall pressure of the material to increase so that cell damage occurs and causes the eggshell cell wall to become porous. This makes water evaporation easier (Prasabani et al., 2013). The water content is closely related to the acid pH content of the acidifying agent used as an acid pretreatment. Table 4 regarding pH levels shows that the lowest pH of eggshell flour was in tamarind pretreatment egg shell flour (Lombok) (pH 8.2967) and the highest was in control (pH 9.2500). The lower the pH of the acidifying material, the greater the level of cell wall damage in the material so that the evaporation process takes place faster and the resulting water content will be smaller.

Ash content represents the amount of mineral content in a material (Nurlaela et al., 2014). Ash content testing is carried out because ash is an inorganic residue from burning organic matter. Its contents and composition depend on the nature of the material being burned and the method of ashing. Ash content in food is determined by weighing the dry

mineral residue of organic matter heated at high temperatures, which is about 550^{o}C (Pomeranz & Meloan 1994). Many factors affect the ash content of eggshell flour. Sudarmadji et al., (1997) stated that the ash content depends on the type of material, the method of ashing, the time and temperature used during drying. In the process of making eggshell flour using drying. The length and time of drying and the high drying temperature will cause the ash content to be higher and the water content in the eggshell flour to come out. The results showed that the ash content of eggshell flour ranged from 79.31%, which was the lowest in tamarind pretreatment (Lombok) to 91.2767%, which was the highest in the control. This indicates the mineral content of eggshell flour is very high. Research Setiawan et al., (2021) explained that egg shells contain minerals such as sodium (Na), magnesium (Mg), silica (Si), phosphorus (P), chloride (Cl), potassium (K), and calcium (Ca) which is the highest content. Calcium in eggshells is used as a constituent of the palisade layer which serves to strengthen the eggshell, so the higher the calcium, the harder or compact the layer (Shen and Chen, 2003). The presence of phosphorus and silica also helps strengthen the palisade layer.

The protein content of egg shells had different values in each treatment. The lower value is possible that the by-products have been lost during the pre-treatment process (Setiawan et al., 2021), namely in this study in the form of acid pretreatment. Table 3 shows that the highest protein content of eggshell flour is 3.9933% in control and the lowest is tamarind (Lombok) which is 3.2367%. This result is somewhat different in the study of Setiawan et al., (2021) which examined the protein content of eggshell waste. The results showed that eggshell had a protein content of 5.7731%. The high protein content in eggshells is because eggshell waste still has a shell membrane. This is in line with the research by Adawyah (2007) which explains that with the heating process, it causes a decrease in water content, causing the protein content of food to increase. Or in other words, the drier the eggshell flour, the higher the protein content. The presence of acid pretreatment will cause the eggshell flour protein to be hydrolyzed which can produce metals, carbohydrates, phosphates and lipids (Lidiasari et al., 2006). The lower acid pretreatment protein content compared to the control is in line with Syarif's (2017) statement explaining that the decrease in protein content can be caused by several factors, one of which is the changing amino acid structure due to heating, and acid. Acid pretreatment causes protein degradation, which results in the formation of short chain peptides, amino acids and volatile ammonia.

Measurement of fat content showed differences in the treatment. The lowest fat content was found in tamarind pretreatment (Lombok) which was 0.33%, while the eggshell flour pretreatment tamarind (Java and Kalimantan) was not significantly different, namely 0.50% and 0.433%. The fat content in the material will decrease along with the high temperature during the drying process. High fat content in food can cause the material to easily oxidize and become rancid so that the shelf life is not durable. Zuhra et al., (2012) added that fat is a compound formed as a result of the esterification reaction between glycerol and fatty acids. The drying process in the study caused the heat from the oven to break the double bonds in the fat, so that the fat would be decomposed into glycerol and fatty acids. Deddy and Nurhaeni (1992) explained that the heating process causes the breakdown of fat components into volatile products, such as aldehydes, ketones, alcohols, acids, and hydrocarbons.

The use of acid pretreatment can reduce fat content during processing and storage. Prabisini (2013) explained that the immersion time in acid pretreatment will make the cell tissue more damaged and perforated, so that the fat in the cell diffuses out of the cell. Added by Leufven et al., (2007) stated that one of the causes of food damage is oxygen. All gas components contained in the air, oxygen is an important gas in terms of food processing. Oxygen can accelerate fat breakdown, namely by oxidative rancidity in fatty foods. The oxidation process can be controlled by lowering the oxygen concentration in the package. The smaller the volume of oxygen in the package, the slower the rancidity process. The things mentioned above can be minimized by acid pretreatment. Acid pretreatment will reduce the effect of oxidative reactions because the fat content in the material is smaller than the material without the acid pretreatment process.

The lowest fiber content in eggshell flour was in the tamarind treatment (Kalimantan) which was 1.7233% and the highest was in the control 1.8900%. This result is higher than the research by Chiu and Wei (2011) which stated that the crude fiber content in egg shells is relatively low ranging from 0.82-1.39 so that with low fiber content, these raw materials can be used as poultry feed.

The carbohydrate content test showed that the highest carbohydrate content was found in tamarind (Lombok) egg shell flour pretreatment which was 15.3867% and the lowest was in the control 2.2953%. Carbohydrate content in eggshell flour is obtained from the effect of the proportion of water content, ash content, protein content and fat content of eggshell flour or in other words using the by differance method. This is in accordance with Fatkurahman et al., (2012) that carbohydrate content is calculated by difference influenced by other nutritional components, namely protein, fat, water, and ash. The heating process will increase the carbohydrate content of eggshell flour due to reduced water in the material due to high temperatures during the oven drying process, although with the presence of acid pretreatment the value is still somewhat lower than the control. In line with Ranken (2000) that reduced water due to the heating process at high temperatures can cause an increase in the amount of fat, protein and carbohydrate content. The results showed that the values mentioned above were found in tamarind (Lombok) pretreatment eggshell flour; This causes the carbohydrate content of tamarind (Lombok) pretreatment eggshell flour to have the highest carbohydrate percentage (Muchtadi and Ayustaningwarno, 2010).

Measurement of minerals in eggshell flour includes minerals calcium and phosphorus. Calcium is one of the most abundant minerals in egg shells. Calcium needs in humans can be met from various sources of calcium derived from animal and vegetable food products. Calcium can also be met from food waste such as poultry egg shells (Yonata et al., 2018). Calcium testing can be seen in the results of table 2, namely the lowest calcium content in the tamarind pretreatment treatment (Lombok) of 85.0100% and the highest in the control of 91.0833%. This is as stated by Nurlaela et al., (2014) that egg shells contain calcium carbonate minerals ranging from 94-97% (Nurlaela et al., 2014). Drying followed by acid pretreatment will cause the breakdown of the bonding components of water molecules (H_2O) and also increase the sugar, fat, mineral content, resulting in an increase in ash content.

Marzuki et al., (2013) added that in addition to calcium, eggshell waste is also rich in phosphorus. The results of the analysis in table 3 show that the lowest phosphorus content is in tamarind egg shell flour pretreatment (Kalimantan) which is 0.7833% and

the highest is in control, which is 0.9567%. Phosphorus (P) is a micronutrient that ranks second after calcium in the total body content (Siswanto et al., 2014). The main function of phosphorus is to provide energy and strength for fat and carbohydrate metabolism, to support healthy teeth and gums, to synthesize DNA and to absorb and use calcium (Emilia, 2009) and to develop muscle and red blood cell function (Moniaga, 2013).

Although eggshell flour has a high calcium content that has the potential to be utilized, the calcium contained in eggshell flour is still in the form of calcium carbonate ($CaCO_3$) which still needs to be broken down. This potential needs to be continuously developed, considering that the human body is not able to synthesize the mineral calcium, so it must be provided through food. Calcium is one of the macro minerals that is needed by the human body, among others, plays a role in the growth and development of bones and teeth, regulates blood clotting, catalyzes biological reactions and muscle contractions (Marzuki et al., 2013). Research on the absorption of calcium from foods containing chicken eggshell flour (45.59%) was not different from (39.88%) $CaCO_3$ which was used as a calcium supplement (Burn et al., 2013). So that the use of acid pretreatment is an alternative way to decompose calcium carbonate in eggshell flour, so that it can be used more optimally. This study was supported by Yonata et al., (2017) who used various chemical solvents in eggshell flour on calcium levels and physical characteristics of eggshell flour. Some chemical solvents that have been widely used are CH_3COOH, HCl and NaOH.

The use of various pretreatments of tamarind from various regions which results in differences in the mineral content of eggshell flour, namely calcium and phosphorus, has the same mechanism, which causes the pores of the shell to open, so that the spaces formed are easier to reach by the solvent, which results in compounds that are produced by the solvent. binds to minerals easily separated with optimum (Suptijah, 2009). The use of acid pretreatment as a soak also improves the physical structure of the shell and reduces harmful components and removes organic compounds from the shell (Aminah and Wulandari, 2016), this is because eggshell flour often still has a fishy smell and has an unattractive color so it is less desirable when used as a preservative. foodstuffs.

Both calcium and phosphorus levels of acid pretreated eggshell flour with control showed that control eggshell flour had a higher value than tamarind pretreatment eggshell flour. The decrease is as in the study of Yonata et al., (2018). Tamarind has a sour and sweet taste because it is associated with a high content of tartaric acid and reducing sugars (De Caluwé et al., 2010). The decrease in calcium and phosphorus levels was caused by the exponential value of the dissociation equilibrium constant of each solution. The dissociation equilibrium value of the solution is related to the pH value. The lower the dissociation equilibrium value of a solution, the stronger the acidity and alkalinity of the solution. In this condition, the solubility of calcium and other mineral components will increase (Vogel. 1985). Soaking with acid pretreatment will decompose calcium carbonate ($CaCO_3$). During the immersion process, some of the CO_3 will dissolve and some will settle to form a white atmosphere of $CaCO_3$ in the form of crystals (Vogel. 1985). It was further explained that the use of an acid solution as an immersion medium resulted in minerals that were initially complex (bonded with other components) turned into simple forms (ions), so that they would be more soluble. In this case, tamarind and the content in it act as enhancers, namely molecules or compounds that affect the shape and degree of solubility of minerals (Clydesdale, 1998).

The yield test in table 3 shows that the lowest yield was in acid-acid pretreated eggshell flour. Yield measurement serves to see the level of efficiency and effectiveness of the drying process so as to produce good flour. The higher the yield value of a treatment, the more effective and efficient the process is (Said et al., 2013). Several factors that greatly affect the yield of foodstuffs are the water content in the food, the temperature during drying, and the size of the dried product (Ramelan and Parnanto, 2016).

The highest yield was found in control egg shell flour, which was 91.8100 and the lowest yield was tamarind egg shell flour pretreatment (Lombok) 86.630%. The type of solvent and the interaction of the shell with the solvent had a very significant effect on the yield of eggshell flour. Yonata et al., (2018) who stated that immersion with distilled water produced shell flour with the best yield. The high yield of eggshell flour is because the components that make up the eggshell are several types of minerals. The yield that tends to decrease when acid pretreatment is carried out is due to several types of mineral components that make up the shell easily hydrolyzed. Heating can cause chemical bonds in the material to break and stretch, so that the material becomes more easily destroyed during the milling process (Nurlaela et al., 2018). The drying process also causes the evaporation of carbon dioxide (CO_2) so that the percentage of yield will be reduced (Saidi et al., 2013). Moreover, the use of acid pretreatment which functions to speed up the drying process will be able to reduce the water content in egg shells during the drying process. In the control, without acid soaking, the denaturation and demineralization process will be slower, so the yield is higher than the acid pretreated eggshell flour (Litayy and Joko, 2013).

Acid pretreatment has an effect on heat absorption and water loss. Some types of acidifying agents are able to speed up the drying time of the control, so that it has a lower moisture content than the control during the drying process. The less water content in the material, the lower the weight of the final material so that the weight and percentage yield will also be smaller. This is as stated by Yonata et al., (2017) that the processing of poultry eggshells can affect the yield of eggshell flour produced, and Masita et al., (2017) and Martunis (2012) stated that the water content in an ingredient affects the yield of flour produced. The high water content in the material will cause the resulting flour yield to be more (Ramlan et al., 2016). This is evident in the control water content of the results of the study which has the highest level of 0.49% causing less evaporated water content so as to produce yields and produce the highest percentage of yield, which is 91.81%. The lowest water content was found in tamarind (Lombok) egg shell flour, which was 0.31%, which resulted in a lower yield of 86.63%. It was further stated by Mastiani et al., (2018) that the yield is influenced by the level of ease of the material to be crushed, which in turn affects the level of fineness, milling method and flour yield.

The results of observations in table 4 show that the pH of the acid pretreatment eggshell flour was different between each treatment. The lowest pH was found in tamarind pretreatment eggshell flour which was 8.2967 and the highest was 9.2500 control pH. pH describes the degree of acidity expressed by the degree of freedom or acidity in a material. The acidity of tamarind is influenced by the content of citric, tartaric and malic acids as well as the organic matter in it. Different types of tamarind exhibit different pH, and various effects on nutritional content. This is as stated by Thursina (2010) that differences in soil types will affect the nutrient content contained in the plants

that grow on it. In addition to soil conditions and types, rainfall, genetic factors in the use of fertilizers and harvesting factors can also affect the mineral content in the soil. Mulyanto et al., (1995) added that pH also has an impact on differences in material content. The difference in initial pH for various types of tamarind will affect the final product in the form of flour. Egg shells that were not subjected to acid soaking had a pH value of control flour which was 9.25. Soaking with acid pretreats makes the acid content in various types of tamarind enter the material, namely egg shells, so that it absorbs and participates in drying. The drying process will evaporate most of the water vapor, and the pH of the food.

Conclusion

There are differences in the use of acid pretreatment on proximate levels, minerals (calcium and phosphorus), yield and pH. The highest value was found in the control and the lowest acid pretreatment, namely tamarind (Lombok) and the best using tamarind (Java).

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