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Processing of White Pepper Through the Combination of Soaking and Boiling Time Towards the Quality

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Abstract. The quality of white pepper depends on the processing process, and it can be done traditionally or semi-mechanically. The present work was to find out the characteristic and off-flavor compound of white pepper through a combination of soaking and boiling time. The research method was divided into several stages: threshing to separate berries and stalks, soaking in a container, boiling, decorticating, washing, and drying in an oven at 60°C. The experiment was designed as a Completely Randomized Design (CRD) with two factors and three replications. The first factor was the length of soaking time (A), A1 4 days, and A2 6 days. The second factor was boiling time (B), Bo: No boiling, B1: 5 minutes, B2: 10 minutes, B3: 15 minutes, and B4: 20 minutes. The results showed that the yield of white pepper from various treatments ranged from 23.82 to 32.96%. The quality of white pepper showed that 4 days of soaking time met the Indonesian Standard quality (SNI) requirement in quality 2. However, 6 days of soaking time required quality 1. Total Plate Count (TPC) levels tend to decrease slightly with the length of boiling time. The best treatment was given from 5 minutes of boiling and 6 days of soaking time. The off-flavor substances found within 4 days of soaking were propanoic, butanoic, hexanoic acids, 4-methylindole, and p-cresol (4-methylphenol). However, within 6 days of soaking, only found butanoic acid, hexanoic acid, and p-cresol.

Keywords: Boiling time; Processing; Semi mechanically; Soaking time; White pepper

1. Introduction

Black pepper (*Piper nigrum L.*) is one of Indonesia's main export commodities and is included in the Piperaceae family. The main product of these crops is black and white pepper which is very well known in the world trading market. Black pepper is harvested at 7-8 months, while white pepper is harvested at 8-9 months. The processing for black pepper is threshed, then directly dried in the sun or oven at 60°C. To improve the color, it can be blanched at 85°C for 3 minutes, then dried. Therefore, white pepper needs a long time to process, particularly soaking in water to make the outer skin of pepper berries soft and rotten (Megat *et al.,* 2020). Even after soaking in water, then it still needs to be followed by being decorticated, washed and dried (Kawachi *et al.,* 2015). Because of soaking time, the products are usually contaminated with molds, yeasts, and spore-forming bacteria. This

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can cause foodborne illnesses in consumers and spoilage of pepper containing foods (Schweiggert, Carle, and Schieber, 2008). The off-odor accumulation on pepper is due to prolonged and extended soaking in a river or stagnant water, which facilitates colonization by unknown microbes, including toxic coliform bacteria. Furthermore, the spread of a known microbial species as targeted decortication would be highly advantageous for ensuring high-quality white pepper.

The traditional method of processing white pepper is still used worldwide because the cultivation and processing of white pepper in most producing countries are done at the farm level. Processing by traditional process requires long soaking in the river or stagnant water and continued direct drying in the sun. The sun drying process is also relevant to further exploring the connection between humans, materials, and the environment as part of the ecological system (Harahap *et al.,* 2020). This will result in the development of characteristic fecal off-odor to the product (Sreekala, Meenakumari, and Vigi, 2019). Consequently, it will need a method to inactivate microorganisms in pepper processing. It has been reported that a decontamination level corresponds to a total plate count of less than 3 log colony forming units (CFU/g) for spices (Ferrentino and Spilimbergo, 2011).

Various studies have been developed by researchers to improve the white pepper processing method by reducing or accelerating the soaking time to soften the skin of the pepper berries to make them easier to peel. Steinhaus and Schieberle (2005) have improved the traditional method by using the ripe starting material with short fermentation under water and with frequent water exchange. Decortication by microbial fermentation has been carried out by Thankamani and Giridhar (2004), while enzymatic retting has been carried out by Usmiati and Nurdjannah (2006) (commercial pectinase). Ashari *et al.* (2014) combined an enzymatic process with an automatic machine which resulted in a significantly reduced soaking time of 5 days, and the quality was superior compared to soaking in tap water. For this reason, in this study, we will be tried the soaking days more and less than 5 days, that is, 4 and 6 days.

The processing of white pepper using a combination of soaking and boiling has not been found in the literature. The boiling process aims to accelerate the softening of the pepper skin so that the soaking time of the pepper berries can be shortened, and the off-flavor odor of white pepper can be minimized. The present work was to find out the characteristic and off-flavor compound of white pepper through a combination of soaking and boiling time.

2. Methods

2.1. Materials and Chemicals

Sample pepper was taken from Sukabumi with a maturity of 9 months. Chemicals are used only for the analysis process, such as standard piperine (Merck), 3,4-dimethoxy benzaldehyde (Sigma-Aldrich), methanol (Merck), NaCl, Buffered Peptone Water, and Plate Count Agar (Sigma-Aldrich).

2.2. Preparing Pepper Processing

The pepper was separated from the berries and stalk before the process. Then, berries weighed 2 kg for every treatment. Continually, the berries were soaked in 10 L of water in a container. The experiment was designed as a Completely Randomized Design (CRD) with two factors and three replications. The first factor was soaking time (A), A1 was 4 days, and A2 was 6 days. The second factor was boiling time (B), B0: No boiling, B1: 5 minutes, B2: 10 minutes, B3: 15 minutes, and B4: 20 minutes. After the boiling process, pepper berries were decorticated, washed then dried at a temperature of 50-60°C for 10-12 hours. The flow chart of the process is seen in Figure 1.

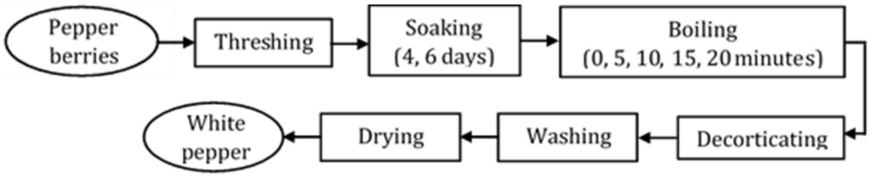


Figure 1 The processing of white pepper through the soaking and boiling process

The parameters observed were yield, color, aroma, off-flavor compound, and moisture in the examined material was determined by drying it to a solid mass at 105°C (Ocieczek, Makala, and Flis, 2021). Piperine content, TPC, and essential oil content refer to SNI 0004: 2013, method bulk density by Irtwange (2000).

2.3. Analysis of Off-flavor Substances by CG-MS (Vinod, Kumar, and Zachariah, 2014)

Weigh accurately 5.0 g of white pepper and spike with 3,4-dimethoxy benzaldehyde (5.1 mg) to the flask. Then the sample was extracted with a mixture of dichloromethane and methanol (2:1, v/v); after being filtered, the resulting filtrate was evaporated using an evaporator with reduced pressure. Extracts were analyzed by gas chromatography-mass spectrometry (Hewlett Packard Gas Chromatography Model (GC) 6890 Mass Spectrometry (MS) 5973), with the following settings:
Programmed initial temperature: 60°C-243°C / 3°C / minute; Inlet temperature: 220°C. Carrier gas: helium. Column: capillary with a length of 30 m, column diameter of 0.32 mm x 0.25 mol /L. Fill column: HD5 cross-linked 5% phenyl methyl siloxane.

2.4. Browning Index (BI)

The browning index (BI) was determined for the product according to Subhashree *et al.* (2017). L*, a*, and b* were measured as colors of white pepper using Chromameter (Ultra scan Pro, Hunter Lab, USA). The L* ranges from 0 to 100 (0 indicates black; 100 indicates white) and parameters a* (from green to red) and b* (from blue to yellow). The browning index was determined according to the following equation:

$$BI = \frac{100\ (x-0.31)}{0.17} \tag{1}$$

$$\text{Where } x = \frac{a*+1.75\ L*}{5.645\ L*+a*-3.012\ b*} \tag{2}$$

2.5. Determination of Total Plate Count (TPC)

A sample of 10 g was weighed and poured into a 90 ml BPW/Buffered Peptone Water solution, shaken until homogeneous, and allowed to stand until the sample settled. Pipette 1 ml of the solution and then put it into a test tube containing NaCl (9 ml). The dilution was carried out to 10^8. The solution was put into a petri dish, and agar medium (PCA/Plate Count Agar) with a temperature of 45°C was poured into a 12-15 ml petri dish, homogenized, and allowed to solidify. The petri dish was put in the incubator for 24-48 hours. Colonies growing on petri dishes were counted using a colony counter. The average result of the total microbial value in each g of the sample according to the formula below:

$$\text{TPC (CFU/g)} = \frac{\text{Numbers of colonies}}{\text{Dilution factors}} \tag{3}$$

3. Results and Discussion

3.1. Characteristic of White Pepper

The results showed that the yield of white pepper had significant differences between treatments (Table 1). For the 6 days of soaking time given, the high yield for every treatment was compared with 4 soaking days. The characteristics of white pepper, and the moisture of white pepper, have no significant differences between treatments. On the other hand, the moisture ranging from 9.10 to 11.25% has met the standard quality requirements. The light pepper for soaking 6 days meets the quality standard requirements 1; however, soaking time 4 days meets quality standard requirements 2. Possibly caused by one stalk, the maturity level of pepper fruit is not uniform, or fruit growth is not normal, so many produce light pepper. The density of the white pepper after 4 days of soaking time has not reached 600 g/l, except for the 15 minutes boiling time treatment, which reached 608.5 g/l (Table 1). This means the white pepper produced does not meet the SNI requirement (Indonesian Standard Quality) because the density is lower than 600 g/l. However, after 6 days of soaking time, the bulk density has a value of more than 600 g/l. The higher density of pepper cages with the same moisture content showed that the pepper is heavier and contains less light pepper (Purwanto, 2011). The minimum density standard for ASTA is 630 g/l, IPC quality I 600 g/l, and ISO 490 g/l. The dark color of the berries was also significantly different between treatments.

Table 1 Characteristics of white pepper quality

Treatment	Yield (%)	Moisture (%)	Bulk density (g/l)	Light berries (%)	Dark color berries (%)	Extraneous matter (%)
A_1B_0	28.54±3.54^{ab}	10.85±0.49^{a}	546.31±4.31^{a}	2.95±0.81^{bc}	0.90±0.14^{b}	0.99±0.75^{bc}
A_1B_1	27.65±3.7^{ab}	10.30±0.20^{a}	566.45±12.84^{ab}	3.39±0.64^{c}	0.92±0.17^{bc}	0.40±0.11^{ab}
A_1B_2	25.11±2.40^{a}	9.10±0.40^{a}	599.35±12.49^{b}	1.11±0.18^{ab}	0.62±0.07^{a}	0.38±0.47^{a}
A_1B_3	23.82±0.28^{a}	9.30±0.40^{a}	608.50±13.44^{b}	1.24±1.27^{ab}	0.36±0.24^{a}	0.25±0.20^{a}
A_1B_4	26.98±2.23^{ab}	10.25±1.10^{a}	596.39±12.91^{b}	2.00±1.10^{b}	1.40±0.21^{ab}	0.67±0.25^{a}
A_2B_0	32.96±5.03^{b}	10.10±0.59^{a}	598.70±12.59^{b}	1.04±0.18^{ab}	1.00±0.81^{ab}	0.37±0.04^{b}
A_2B_1	29.04±0.55^{ab}	9.75±3.30$^{a.}$	624.53±4.97^{bc}	0.34±0.20^{a}	0.66±0.55^{ab}	0.50±0.44^{ab}
A_2B_2	30.81±1.61^{b}	11.25±1.10^{a}	617.22±11.03^{bc}	0.40±0.14^{a}	1.02±0.54^{ab}	0.67±0.11^{b}
A_2B_3	28.83±0.98^{ab}	9.60±0.79^{a}	658.23±3.68^{c}	0.60±0.33^{a}	1.43±0.38^{c}	1.34±0.03^{c}
A_2B_4	28.64±0.66^{ab}	9.25±0.91^{a}	657.04±5.35^{c}	0.28±0.28^{a}	1.06±0.17^{bc}	1.06±0.42^{d}
Quality I*	-	13.00	600.00	Max. 1	Max.1	Max.1
Quality II*	-	13.00	600.00	Max.2	Max.2	Max.2

Note: The number followed by the same letter in the same column indicates no significant differences in Duncan's multiple range test ($p < 0.05$).

3.2. Piperine and Essential Oil Contents

Piperine content from the treatment of 6 days of soaking tended to decrease with the length of boiling time (Figure 2a) and had significant differences between treatments. Piperine is a bioactive compound with many beneficial health and therapeutic effects (Gorgani *et al.,* 2017). Piperine found in pepper has major pharmacological impacts on neuromuscular systems, exercises a sedative effect, and helps in digestion (Andrade and Ferreira, 2013).

The essential oil content was significantly different from the treatment of 6 days of soaking and boiling times of 15 minutes (Figure 2b). The essential oil of pepper is a mixture of many volatile chemical compounds in pepper (CBI, 2018). The boiling process will affect the aroma component of the pepper. If the boiling is longer than 30 minutes, the aroma and volatile oil components will evaporate. Volatile components of pepper, especially essential oil components, contain terpenoid compounds that have biological activity (Hao *et al.,* 2018).

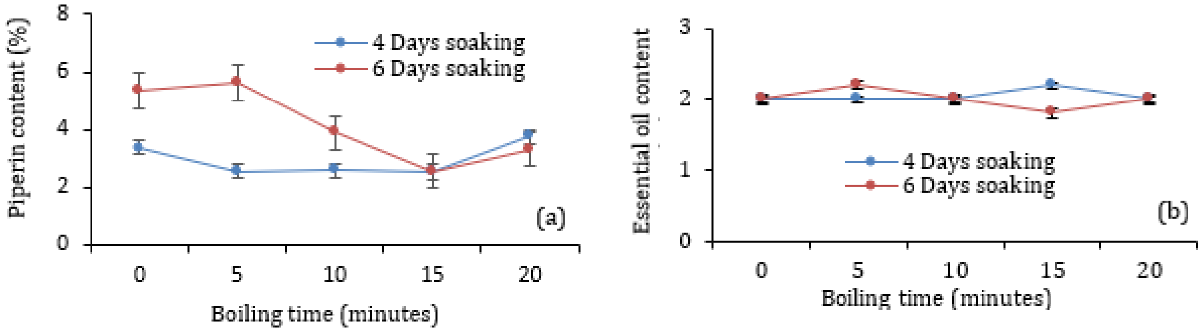


Figure 2 The piperine (a) and essential oil content (b) from variation treatment

3.3. Color

The product's color was slightly brownish, according to the L* value and degree of °hue (Table 2). The highest L* value resulted from the 6 days soaking treatment and the boiling time of 15 minutes (45.28).

Tabel 2 Color value of white pepper from a variation of boiling and soaking time

Treatment	L*	a*	b*	c*	°hue
A_1B_0	36.40±0.37^{b}	2.03±0.11^{c}	15.70±0.03^{a}	15.83±0.16^{a}	48.45±0.23^{a}
A_1B_1	32.92±1.23^{a}	0.80±0.32^{a}	16.77±0.80^{a}	16.79±0.35^{bc}	87.31±1.27^{d}
A_1B_2	43.41±0.19^{c}	1.12±0.13^{ab}	17.69±0.18^{a}	17.72±0.14^{de}	86.42±0.46^{d}
A_1B_3	44.13±0.41^{d}	1.08±0.39^{ab}	18.11±0.28^{a}	18.14±0.25^{de}	86.63±0.46^{d}
A_1B_4	43.08±0.90^{c}	1.31±0.33^{ab}	18.41±1.20^{a}	18.46±0.58^{de}	85.97±0.98^{cd}
A_2B_0	38.81±1.39^{b}	1.98±0.16^{bc}	17.18±2.72^{a}	17.29±0.10^{cd}	83.47±0.10^{b}
A_2B_1	45.14±0.32^{cd}	1.80±0.40^{bc}	16.40±1.85^{a}	16.50±0.46^{ab}	83.78±0.52^{b}
A_2B_2	43.23±0.74^{c}	1.78±0.35^{bc}	19.58±1.68^{a}	19.66±0.40^{f}	84.85±0.68^{bc}
A_2B_3	45.28±0.91^{cd}	0.85±0.47^{a}	17.67±1.46^{a}	17.69±0.35^{de}	87.29±0.25^{d}
A_2B_4	43.79±1.32^{c}	0.81±0.09^{a}	17.14±0.11^{a}	17.16±0.69^{cd}	87.34±0.39^{d}

The different maturity of each fresh pepper berries on a spike is caused by variations in color (Megat *et al.,* 2020). For example, the L* value for the 4 days of soaking pepper berries was 32.92±1.23, and on day 6 of soaking, its value increased to 45.14±0.32. This value shows that the samples turned white since it is a color measurement on the light-dark axis. The a* value was also increased during the soaking process from 0.80±0.32 to 1.98±0.16 color. For more details, the color resulting from the processing of white pepper is illustrated in Figure 3.

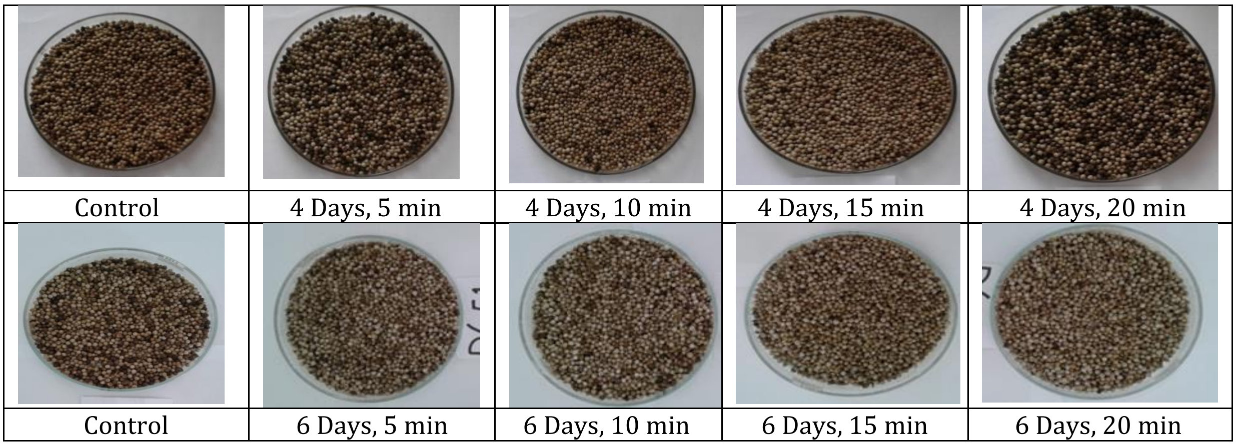


Figure 3 White pepper product from a variety of treatment

3.4. Browning Index (BI)

The browning index (BI) of 4 days of soaking tended to decrease by the length of boiling time; however, for 6 days of soaking, there was an increase to 10 minutes boiling time, then decreased until 20 minutes boiling time (Figure 4). The changes in BI were that soaked pepper berries were more likely to change color to black instead of a browning reaction.

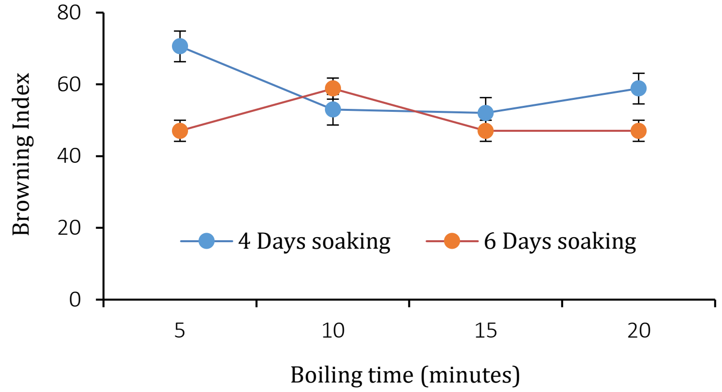


Figure 4 Browning index of white pepper product

In the soaking method in boiling water, the fragrance component will scatter, and the flavor becomes weak. Furthermore, the browning color is due to acid soot such as eluted polyphenol and chlorophyll (Namiki, Nakahara, and Abe, 2007).

3.5. Total Plate Content (TPC)

For TPC levels, 6 days of soaking tend to decrease with the length of boiling time (Figure 5). Otherwise, during the 4 days soaking time, TPC levels tend to decrease, then slightly increase with the length of boiling time.

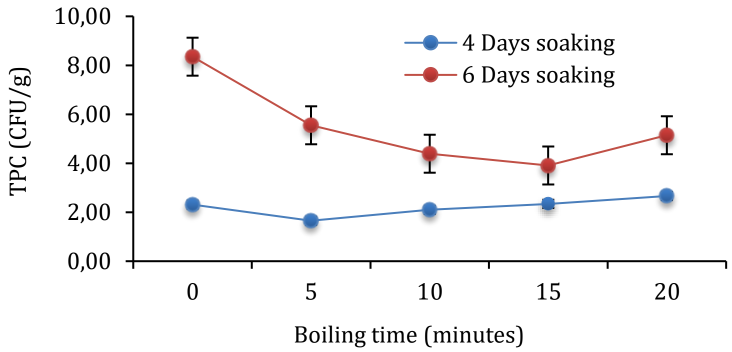


Figure 5 TPC value from variation treatment

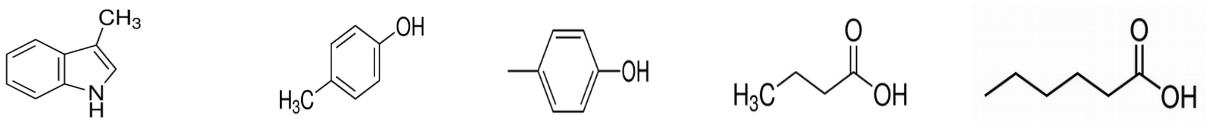
The length of soaking time showed an increase in the number of bacteria; however, it will be decreased by boiling. This is because the length of boiling time means the length of heating, which will affect the number of bacteria.

3.6. Off-flavor Substances

The chemical component was identified by the retention time of the component on the chromatogram and compared with the mass spectra of the MS database and also the fragmentation pattern in mass spectra of the NIST (National Institute of Standards and Technology) (Hao *et al.,* 2018). The chemical compounds were found, namely, 4-methylindole (fecal, swine-manure-like), 4-methylphenol (fecal, horse-like), and butanoic acid (cheese-like) found in white pepper formed during the retting time (Table 3 and Figure 6). The problem with pepper berries is that soaking in water for a long time may result in an accumulation of the above chemicals resulting in an off odor (Sreekala, Meenakumari, and Vigi, 2019). One of the major issues with the white pepper trade is its offensive fecal odor and the consequent consumer rejection (Vinod, Kumar, and Zachariah, 2014).

Tabel 3 The off-flavor substances from every treatment

Substances	Abundance (%)									
	A_1B_0	A_1B_1	A_1B_2	A_1B_3	A_1B_4	A_2B_0	A_2B_1	A_2B_2	A_2B_3	A_2B_4
Propanoic acid	0.49	0.48	-	0.45	0.50	-	-	-	-	-
Butanoic acid	3.91	-	1.49	6.53	2.43	1.01	-	-	-	-
4-Methylindole	-	-	0.30	-	-	-	-	-	-	-
4-Methyphenol	-	-	1.01	0.04	0.60	-	-	-	0.30	-
Hexanoic acid	0.62	-	0.98	-	0.38	-	0.37	0.91	0.71	-
p-Cresol										



4-Methylindole 4-Methylphenol p-Cresol Butanoic acid Hexanoic acid

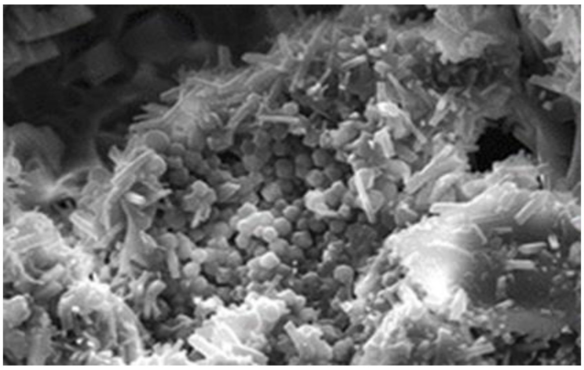
Figure 6 The structure of of-flavor

It has been reported by a previous study that soaking time for processing white pepper needs more than 6 days (Table 4). Nonetheless, the processing of white pepper still requires a long soaking, even though it has already been assisted by the addition of enzymes (Table 4).

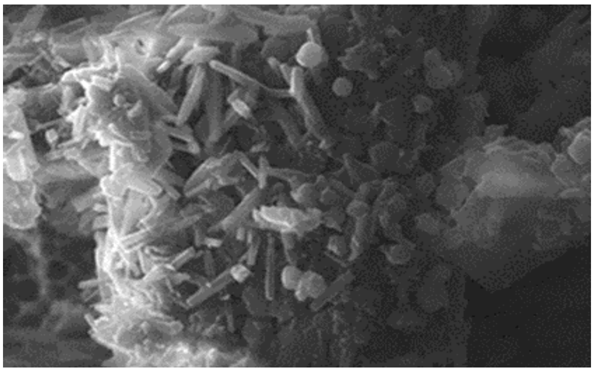
Table 4 The processing of white pepper has been reported

Methods	Results	References
Berries were placed in plastic barrels and soaked in water, and the water was exchanged daily	Off-odorant (3-methylindole and 4-methylphenol) formation could be suppressed until the fermentation time <7 days	Steinhaus and Schieberle (2005)
Producing white pepper using a combination of soaking in water and a decorticator	The white pepper processing requires soaking time for 7 days to get optimal quality	Rajesh *et al.* (2019)
Decorticating fresh berries or black pepper berries by specific bacterial	The white pepper obtained from bacterial decortication is free from the off-odor compound and does not affect essential oil content, oleoresin, and piperine content	(Vinod, Kumar, and Zachariah, 2014)
The enzymatic retting using the Viscozyme and Celluclast	The enzymatic retting at 42°C can fully soften the pericarp of pepper berries from 15 days to 7 days with non-blanched pepper berries	Rosnah and Chan (2014)
Processing white pepper by fermentation using *Bacillus subtilis* inoculum	The soaking time of pepper for 7 days produced better quality pepper than soaking for 5 days	Hernani, Yumna, and Aminingsih (2021)

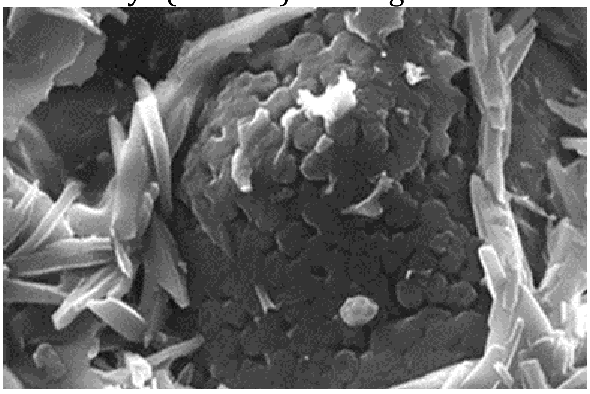
The morphology of pepper in variation treatment is shown in Figure 7. Based on SEM observations, there were many fragments on the surface of plant tissue. Microstructure changes were seen on several specimen surfaces. Microstructural changes in the length of boiling time were thought to be due to weak bonding between the matrix on the seed. For an illustration of morphology by SEM (Scanning Electron Microscope) of Pepper seed from 4 and 6 days soaking time and 20 minutes boiling time, respectively. Indentation occurs due to the process of shrinking or shrinking particles that occur during the process of drying and cooling (Sahlan *et al.*, 2019).



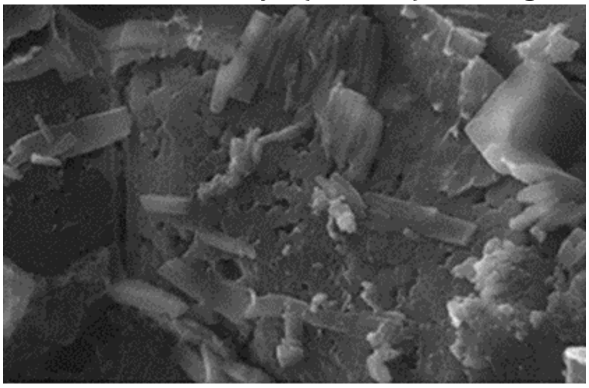
4 Days (Control) soaking



6 Days (Control) soaking



4 Days soaking and 20 minutes boiling times



6 Days soaking and 20 minutes boiling times

Figure 7 Morphological of pepper in 4 and 6 days soaking times and 20 minutes boiling time

It can be seen that the starch granule of white pepper was polygonal and polyhedral with an irregular shape. The shape of pepper starch granules had a degree of similarity to that of rice starch (Zhu, Mojel, and Li, 2017). Nonetheless, the length of boiling time affected the starch form; it was shown that the starch started to clot due to the length of boiling time.

4. Conclusions

The yield of white pepper was given from the 6 days soaking time compared with 4 soaking days. The characteristic of white pepper from 6 days soaking was almost met in requirement quality I. However, from 4 days soaking days, it was only for boiling time 15 minutes required in quality I. The piperine and essential oil contents were given ranged from 2.53 to 5.63 and essential content from 1.8 to 2.0 %, respectively. TPC levels tend to decrease slightly with the length of boiling time. The off-flavor substances found in 4 days of soaking were propanoic, butanoic, hexanoic acids, 4-methylindole, and p-cresol. However, 6 days of soaking only found butanoic, hexanoic acids, and p-cresol (4-methyl phenol). The morphology of white pepper showed the clot of starch starting at a 10 minutes boiling time.

Acknowledgments

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