

Original Research Article

## Effects of Flowing Water on Soaking Water Quality During the Retting Process of Pepper Berries (*Piper Nigrum L.*)

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**Abstract:** This study assesses the effects of flowing water on soaking water quality during the retting process of pepper berries. Generally, organic matter and the bioactive compounds that naturally exist in pepper may have leached out into the soaking water because of the prolonged retting process. Daily samplings were carried out by triplicate water samples for seven consecutive days. The soaking test was carried out by having 5 kg of pepper berries under the flowing water with a flow rate of 70 L/min. Six parameters were measured based on standard methods: pH, turbidity, dissolved oxygen, chemical oxygen demand, total dissolved solids, and colour. The results showed the changes of soaking water in turbidity (from  $13.73 \pm 0.85$  NTU to  $33.53 \pm 0.71$  NTU), pH (from  $6.95 \pm 0.02$  to  $6.23 \pm 0.02$ ), dissolved oxygen (from  $7.75 \pm 0.09$  g/ml to  $6.15 \pm 0.02$  g/ml), chemical oxygen demand (from  $24.33 \pm 1.53$  g/ml to  $27.67 \pm 1.53$  g/ml), total dissolved solids (from  $39.53 \pm 2.36$  g/ml to  $58.50 \pm 0.65$  g/ml) and total colour change (from  $0.53 \pm 0.09$  to  $0.87 \pm 0.03$ ) during the retting process were corresponding to the soaking time. As a result, this study reveals that the use of flow water for the retting process tends to avoid sedimentation. Also, it ensures the quality of the white pepper.

**Keywords:** pepper berries; retting process; soaking water; flow; water quality

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## 1. Introduction

White pepper is one of the black pepper's value-added forms. It is highly valued due to its attractive creamy white colour and freedom from microbial contamination, making it ideal for many food preparations. Pepper is one of the world's most popular food flavouring ingredients, and it shares a presence with salt on most dinner tables. The state of Sarawak supplies more than 95% of the total production of white pepper in Malaysia.

The retting method is defined as the fibre extraction that involves decomposition by the microbiological method of the cementing material or dissolution by chemical method in which the fibre bundles are loosened from the adhering tissues and extracted by washing (Bhattacharyya, 1974; Gupta *et al.*, 1976; Ahmed & Akhter, 2001). Retting is best done in slowly in flowing clear water (canal, river, etc.) with a low content of materials such as salts, iron, and calcium content is preferable for successful retting (Kundu *et al.*, 1959) (Ahmed & Akhter, 2001). It is preferable to keep the water at a pH of 7 and a temperature of 35 °C. White pepper is derived from pepper berries by cutting the outer pericarp of the berries (Ramlan, 2010). Soaking is essential during the retting process of white pepper to soften the pericarp of mature pepper berries. In Sarawak, the current method used in white pepper production is to soak the fresh pepper berries under slow running water for 12–14 days, such as rivers after harvesting and threshing (Shamsudin & Chan, 2014; Megat Ahmad Azman *et al.*, 2020).

According to Aziz *et al.* (2018) and Mazaheri and Mozaffari (2019), there are possibilities that the organic matters and the bioactive compounds naturally present in pepper may have been leached out into the soaking water due to prolong retting process. At the same time, inorganic matters and microorganisms are also found in soaking water (Mazaheri & Mozaffari, 2019; Megat Ahmad Azman *et al.*, 2020).

In other studies, many valuable studies were related to the water quality, including soaking water of soy-bulgur (Bayram *et al.*, 2004), integrated urban drainage systems (Vezzaro *et al.*, 2014), drinking water sources (Manickum *et al.*, 2014), dam water (Nthunya *et al.*, 2019), and groundwater (Jose *et al.*, 2020). Therefore, many studies are related to the quality of groundwater, wastewater, and others. However, Shamsudin and Chan (2014), Aziz *et al.* (2018), Megat Ahmad Azman *et al.* (2020), and Megat Ahmad Azman *et al.* (2020) have documented the limited studies related to the white pepper retting process. To the best of the authors' knowledge, there is a lack of information on the soaking water quality during the retting process of pepper berries using flowing water. The characteristics or water quality of the soaking water should be monitored and evaluated to ensure that the soaking process is carried out effectively. Thus, the study aims to determine the effects of flowing water on soaking water quality during the retting process of pepper berries.

## 2. Materials and Methods

### 2.1. Sample Preparations

Fresh mature pepper berries from a variety of Kuching were selected. The ripe pepper berries were removed from the spikes and threshed using a local thresher. Then, they were transferred from a farm to the laboratory at room temperature. The mature pepper berries were sorted according to the colour (visual parameter), dark green and yellowish-green, which will be used for the white pepper retting process.

### 2.2. Soaking Test

A rectangle tank with the dimensions of 107 cm (length) × 73 cm (width) × 30 cm (height) was used as a soaking tank for the soaking test. The size of the tank was based on the number of pepper berries that would be soaked and the amount of water needed for the retting process. Commonly, the ratio of pepper berries and water needed for the retting process is 1:6. However, the amount of water required for this study could be higher than the ratio as it uses the flowing water. For this soaking test, the sorted mature pepper berries of 5 kg were put into a jute sack and then soaked under flowing water with a flow rate of 70 L/min. The soaking test was monitored, and the sample of soaking water was taken for seven consecutive days. This study's seven-day retting process includes five sunny days (days 1, 2, 3, 6, and 7) and two rainy days (days 4 and 5). The retting process in this study was carried out for seven days in the running water state, and the use of a submerged pump (70 L/min) is a reflection of the conventional retting process that soaked pepper berries in the river in a state of slow-flowing water for 1214 days. Therefore, parameters of soaking water quality such as turbidity, pH, dissolved oxygen, chemical oxygen demand, total dissolved solids, and colour for seven days were determined.

### 2.3. Soaking Water Quality Analysis

#### 2.3.1. Turbidity measurement

Turbidity is a water clarity that expresses the amount of light dispersed by any matter in the water when light passes through the sample. An approximate 10 mL of soaking water was used. A turbidity meter (2100 Q, HACH, USA) was used to measure the turbidity of the soaking water.

#### 2.3.2. pH measurement

pH is a parameter to determine whether the water sample is an acid, neutral, or alkali. At room temperature, the pH of soaking water was measured using a pH meter (Spear pH Tester, China).

### 2.3.3. Dissolved oxygen measurement

Dissolved oxygen is a parameter to determine the level of oxygen dissolved in the water. Equipment of YSI Professional Plus Multimeter (Xylem, USA) was used to measure the dissolved oxygen concentration of soaking water.

### 2.3.4. Chemical oxygen demand measurement

Chemical oxygen demand is a parameter to determine the total pollutant that cannot be oxidized biologically in the water. Soaking water's chemical oxygen demand was measured using a spectrophotometer (DR/4000U, HACH, USA).

### 2.3.5. Total dissolved solids measurement

Total dissolved solids are a parameter to determine the inorganic salts and small amount of organic matter found in water. The total dissolved solid of soaking water was measured using YSI Professional Plus Multimeter (Xylem, USA).

### 2.3.6. Colour measurement

Colour is a parameter to determine the changes in water that are influenced by the retting process of pepper berries. A colour meter (UltraScan Pro, HunterLab, USA) was used to determine the colour values, including  $L^*$  (whiteness or darkness),  $a^*$  (greenness or redness), and  $b^*$  (yellowness or blueness). Furthermore, the other colour value, such as total colour change ( $\Delta E$ ) of soaking water, was calculated by using the following equations:

$$\Delta E = \sqrt{\left((L^*_f - L^*_0)^2 + (a^*_f - a^*_0)^2 + (b^*_f - b^*_0)^2\right)} \quad (1)$$

Where  $L^*_f$  = the final measured value for whiteness or darkness;  $L^*_0$  = the initial measured value for whiteness or darkness;  $a^*_f$  = the final measured value for greenness or redness;  $a^*_0$  = the initial measured value for greenness or redness;  $b^*_f$  = the final measured value for yellowness or blueness;  $b^*_0$  = the initial measured value for yellowness or blueness.

## 2.4. Statistical Analysis

All the collected data were analysed by using Minitab Statistic 16 Edition. For all data, a one-way analysis of the variance was performed as a function of time to determine the significant differences between mean values ( $p < 0.05$ ) at 95% of the confidence level. Tukey tests were carried out to assess the influence of ripe pepper berries and the soaking time on the soaking quality of the water during the soaking process by using flowing water and to predict homogeneous groups. For each study, the triplicate was done, and the average of three independent experiments was provided as the details.

### 3. Results and Discussions

Soaking water quality is one of the essential factors in ensuring the quality of white pepper produced. Table 1 shows the parameters for soaking water quality, including turbidity, pH, dissolved oxygen, chemical oxygen demand, colour, and total dissolved solid during the white pepper retting process by using flowing water.

**Table 1.** Mean values for soaking water quality during seven days of the retting process.

Day	Parameters of soaking water quality				
	Turbidity (NTU)	pH	Dissolved oxygen (mg/L)	Chemical oxygen demand (mg/L)	Total dissolved solid (mg/L)
0	13.73 ± 0.85 <sup>d</sup>	6.95 ± 0.02 <sup>a</sup>	7.75 ± 0.09 <sup>a</sup>	24.33 ± 1.53 <sup>d</sup>	39.53 ± 2.36 <sup>c</sup>
1	13.40 ± 0.85 <sup>d</sup>	6.76 ± 0.02 <sup>ab</sup>	7.21 ± 0.15 <sup>b</sup>	18.33 ± 1.53 <sup>e</sup>	39.65 ± 0.65 <sup>c</sup>
2	17.00 ± 1.15 <sup>cd</sup>	6.74 ± 0.01 <sup>ab</sup>	6.66 ± 0.21 <sup>cd</sup>	19.33 ± 0.58 <sup>e</sup>	40.52 ± 1.99 <sup>c</sup>
3	18.10 ± 0.53 <sup>c</sup>	6.73 ± 0.02 <sup>ab</sup>	6.26 ± 0.05 <sup>e</sup>	20.33 ± 1.53 <sup>e</sup>	41.75 ± 1.13 <sup>c</sup>
4	66.10 ± 3.21 <sup>a</sup>	6.12 ± 0.41 <sup>c</sup>	6.95 ± 0.05 <sup>bc</sup>	67.33 ± 1.53 <sup>a</sup>	63.27 ± 0.38 <sup>a</sup>
5	65.67 ± 1.36 <sup>a</sup>	6.54 ± 0.10 <sup>abc</sup>	6.64 ± 0.21 <sup>cd</sup>	39.67 ± 0.58 <sup>b</sup>	62.62 ± 0.38 <sup>a</sup>
6	34.27 ± 1.55 <sup>b</sup>	6.44 ± 0.01 <sup>bc</sup>	6.45 ± 0.12 <sup>de</sup>	29.00 ± 1.00 <sup>c</sup>	57.63 ± 0.99 <sup>b</sup>
7	33.53 ± 0.71 <sup>b</sup>	6.23 ± 0.02 <sup>c</sup>	6.15 ± 0.02 <sup>e</sup>	27.67 ± 1.53 <sup>cd</sup>	58.50 ± 0.65 <sup>b</sup>

Data are expressed as mean ±SD. Different letters indicate statistically significant differences exist  $p < 0.05$  for each column. Means do not share a letter are significantly different. Tukey's test was applied with 95% simultaneous confidence intervals.

#### 3.1. Measurement of Turbidity

As shown in Table 1, the initial value of soaking water turbidity was  $13.73 \pm 0.85$  NTU. The turbidity of soaking water was slightly decreased with the value of  $13.40 \pm 0.85$  NTU on the first day by 2.40%, a reduction of the initial value. Then, the turbidity value determined for the subsequent days increased from  $17.00 \pm 1.15$  NTU (day 2) to  $66.10 \pm 3.21$  NTU (day 4). However, the soaking water turbidity was decreased by 49.27% from day four until day 7 with a value of  $33.53 \pm 0.71$  NTU. Among the seven days of the retting process, soaking water's turbidity on days 4 and 5 had the highest importance. This occurs due to the water pond's matter or biological composition during the rainy period. According to Megat Ahmad Azman *et al.* (2020), the turbidity of stagnant water was  $1103.30 \pm 23.10$  NTU for 3 kg of soaked pepper berries. Therefore, the difference in turbidity values between the result obtained in this study and Megat Ahmad Azman *et al.* (2020) indicates that the use of flowing water in the retting process may reduce the tendency of higher turbidity of the soaking water. Overall, the soaking water's turbidity was changed over time during the retting process. By

using flowing water, organic matter such as hydrolyzable tannin, which forms a dark tannin, can be significantly reduced.

### 3.2. Measurement of pH

The pH of soaking water was measured from day 0 until day 7 of the retting process. The initial pH of soaking water was  $6.95 \pm 0.02$ . Based on Table 1, the pH of soaking water on the first day was  $6.76 \pm 0.02$ , and it decreased steadily after a few days of soaking with the value of  $6.74 \pm 0.01$  on day two until it reached  $6.12 \pm 0.41$  on day 4. However, the pH of soaking water increased on day 5 with a value of  $6.54 \pm 0.10$ . After that, it decreased again on day 6 ( $6.44 \pm 0.01$ ) and day 7 ( $6.23 \pm 0.02$ ). Overall, the pH of soaking water was gradually reduced from day 0 until day seven, as much as 10.36%. Based on the result, the pH change remained neutral when the acid leached content of the pepper berries in the soaking water was lower. The reduction in the pH value of this result was a small percentage compared to the previous study using stagnant water with a value of  $3.67 \pm 0.02$  (Megat Ahmad Azman *et al.*, 2020) during the retting process. Thus, the result showed that the retting process using flowing water would maintain the pH of the soaking water and helps to prevent the anaerobic reaction from occurring.

### 3.3. Measurement of Dissolved Oxygen

As indicated in Table 1, the initial value of dissolved oxygen was  $7.75 \pm 0.09$  g/ml. It decreased steadily after a few days of soaking with  $7.21 \pm 0.15$  g/ml and  $6.66 \pm 0.21$  g/ml on day one and day 2, respectively. Then, the dissolved oxygen value determined on day 3 ( $6.26 \pm 0.05$  g/ml) was also decreased. However, the dissolved oxygen value of soaking water was increased on day four at  $6.95 \pm 0.05$  g/ml. This occurs due to the water pond's matter or biological composition during the rainy period. The dissolved oxygen of soaking water was decreased once more on day 5 ( $6.64 \pm 0.21$  g/ml) until day 7 ( $6.15 \pm 0.02$  g/ml). Due to the prolonged retting process of pepper berries, the bacteria naturally break down organic matter, which consumes some oxygen. However, the flow of water during the retting process may prevent and replace the loss of oxygen during the breakdown of organic matter. Overall, the dissolved oxygen of soaking water was changed from day 0 until day 7 with a reduction of 20.65%. While the dissolved oxygen decreased, it remained within acceptable water quality standards (5–7 g/ml).

### 3.4. Measurement of Chemical Oxygen Demand

The chemical oxygen demand of soaking water was measured from day 0 until day 7 of the retting process. Soaking water's initial chemical oxygen demand value was  $24.33 \pm 1.53$  g/ml. Based on Table 1, the chemical oxygen demand of soaking water on the first day was  $18.33 \pm 1.53$  g/ml, and it was increased steadily after a few days of soaking with the value of  $19.33 \pm 0.58$  g/ml on day two until it reached to  $20.33 \pm 1.53$  g/ml on day 3. However, the chemical oxygen demand values of soaking water from day one until day 3

were insignificant ( $p < 0.05$ ). The soaking water on day 4 of the retting process had a higher chemical oxygen demand with a value of  $67.33 \pm 1.53$  g/ml. This is due to the occurrence of rain on the fourth day. Then, the chemical oxygen demand of soaking water decreased on day 5 with a value of  $39.67 \pm 0.58$  g/ml. After that, it was again reduced on day 6 ( $29.00 \pm 1.00$  g/ml) and day 7 ( $27.67 \pm 1.53$  g/ml). Overall, the chemical oxygen demand for soaking water was increased gradually from day 0 until day seven, as much as 13.73%. Thus, less amount of organic and inorganic oxydizable compounds in soaking water had occurred due to water flow, which has driven the compounds out of the soaking tank.

### 3.5. Measurement of Total Dissolved Solids

As shown in Table 1, the initial total dissolved solid value of soaking water was  $39.53 \pm 2.36$  g/ml. The total dissolved solid of soaking water was not significantly increased from day 1 ( $39.65 \pm 0.65$  g/ml) until day 3 ( $41.75 \pm 1.13$  g/ml). However, the total dissolved solid was increased sharply on day four and day 5 with the value of  $63.27 \pm 0.38$  g/ml and  $62.62 \pm 0.38$  g/ml, respectively. Then, the total dissolved solid determined for the next day was decreased once more with the values of  $57.63 \pm 0.99$  g/ml (day 6) and  $58.50 \pm 0.65$  g/ml (day 7). Among the seven days of the retting process, the total dissolved solid of soaking water on days 4 and 5 had the highest values. Overall, the change in total dissolved solid was due to the content of inorganic salts and some small amount of organic matter dissolved in soaking water.

### 3.6. Measurement of Colour

After undergoing some process, including the soaking process, the colour may change. The colour of soaked pepper berries is, therefore, the most critical parameter for the acceptability of the white pepper final product (Bayram *et al.*, 2004; Megat Ahmad Azman *et al.*, 2020).

**Table 2.** Mean values for colour of soaking water during seven days of retting process.

Day	Colour			
	$L^*$	$a^*$	$b^*$	$\Delta E$
0	$32.17 \pm 0.02^c$	$-0.02 \pm 0.13^a$	$0.62 \pm 0.04^{bc}$	-
1	$32.60 \pm 0.03^a$	$-0.29 \pm 0.08^a$	$0.70 \pm 0.01^{ab}$	$0.53 \pm 0.09^d$
2	$31.98 \pm 0.03^d$	$-0.27 \pm 0.06^a$	$0.43 \pm 0.08^{cd}$	$0.38 \pm 0.08^d$
3	$32.35 \pm 0.01^b$	$-0.01 \pm 0.47^a$	$0.34 \pm 0.19^{de}$	$0.47 \pm 0.07^d$
4	$30.50 \pm 0.04^g$	$-0.18 \pm 0.06^a$	$0.92 \pm 0.11^a$	$1.71 \pm 0.03^b$
5	$31.48 \pm 0.02^e$	$-0.08 \pm 0.11^a$	$0.44 \pm 0.11^{bcd}$	$0.74 \pm 0.03^c$
6	$30.26 \pm 0.03^h$	$-0.20 \pm 0.03^a$	$0.11 \pm 0.03^e$	$1.99 \pm 0.03^a$
7	$31.32 \pm 0.01^f$	$-0.16 \pm 0.05^a$	$0.53 \pm 0.08^{bcd}$	$0.87 \pm 0.03^c$

Data are expressed as mean  $\pm$ SD;  $L^*$ , whiteness or darkness;  $a^*$ , greenness or redness;  $b^*$ , yellowness or blueness;  $\Delta E$ , total colour change. Different letters indicate statistically significant differences exist  $p < 0.05$  for each column. Means do not share a letter are significantly different. Tukey's test was applied with 95% simultaneous confidence intervals.

The colour of soaking water was measured from day 0 until day 7 of the retting process. The initial colour values of soaking water were  $32.17 \pm 0.02$  ( $L^*$ ),  $-0.02 \pm 0.13$  ( $a^*$ ) and  $0.62 \pm 0.04$  ( $b^*$ ). Based on Table 2, the  $L^*$  value of soaking water on the first day was  $32.60 \pm 0.03$ , slightly decreasing on day 2 with the value of  $31.98 \pm 0.03$ . After a few days, the values of  $L^*$  fluctuated due to the water flow into the soaking tank. Therefore, the values of  $L^*$  showed that the soaking water was apparent, yet they were still coloured. Next,  $a^*$  value is negative, which indicates that greenness was present in the soaking water. The value of  $a^*$  was decreased on day 1 with the value of  $-0.29 \pm 0.08$ . It was increased steadily after a few days of soaking with the value of  $-0.27 \pm 0.06$  (day 2) until it reached  $-0.16 \pm 0.05$  (day 3). Although the values of  $a^*$  fluctuated, they were not significantly affected ( $p < 0.05$ ) during the retting process. Based on Table 2, the  $b^*$  values were slightly decreased from  $0.70 \pm 0.01$  (day 1) until reached to  $0.53 \pm 0.08$  (day 7). Therefore, these  $b^*$  values showed the yellowness in the soaking water. Overall, the value of  $L^*$ ,  $a^*$ , and  $b^*$  of the soaking water during the retting process using the flowing water within a range of clear water and still coloured due to the water flow into the soaking tank. These results showed that the soaking water's color does not affect the colour of soaked pepper berries.

The total colour change was defined as a colour change over time. The value of  $\Delta E$  on the first day was  $0.53 \pm 0.09$ . The  $\Delta E$  of soaking water was increased gradually from day 2 ( $0.38 \pm 0.09$ ) until day 6 ( $1.99 \pm 0.03$ ). The value of  $\Delta E$  on the seventh day was  $0.87 \pm 0.03$ , incrementing by 64.15%. Thus, the higher value of the total colour change of soaking water was  $1.99 \pm 0.03$  on the sixth day of the retting process.

#### 4. Conclusions

This research aimed to determine the effects of flowing water on soaking water quality during the retting process of pepper berries. This research shows that the quality of soaking water, such as turbidity, pH, dissolved oxygen, chemical oxygen demand, total dissolved solids, and colour, slightly changed steadily during the retting process. The changes of soaking water in turbidity (from  $13.73 \pm 0.85$  NTU to  $33.53 \pm 0.71$  NTU), pH (from  $6.95 \pm 0.02$  to  $6.23$ ), dissolved oxygen (from  $7.75 \pm 0.09$  g/ml to  $6.15 \pm 0.02$  g/ml), chemical oxygen demand (from  $24.33 \pm 1.53$  g/ml to  $27.67 \pm 1.53$  g/ml), total dissolved solids (from  $39.53 \pm 2.36$  g/ml to  $58.50 \pm 0.65$  g/ml) and total colour change (from  $0.53 \pm 0.09$  to  $0.87 \pm 0.03$ ) during the retting process were corresponding to the soaking time. Prolonged soaking time and flowing water have influenced the quality of soaking water. The fluctuation of the results of the soaking water quality may have occurred due to organic matters and bioactive compounds found in the pepper berries that have been leached into the soaking water, as well as the biological components present in the water pond. However, the water quality was also in the standard range; the soaking water was clear yet still coloured. Based on the results, the quality of the soaking water may ensure that the pepper berries are of good quality. Understanding the quality of the water quality during the retting process of



pepper berries is of practical importance as it controls the process and hence, the quality of the final product. Thus, the retting process of pepper berries using the flowing water was the best method with good soaking water quality conditions compared to stagnant water.

**Author Contributions:** P.N.M.A.A. conducted the experiments, collected and analysed the data of results, and wrote the manuscript. R.S. supervised the research and revised the manuscript. H.C.M. and M.E.Y. supervised the experiments. All authors have read and agreed to the published version of the manuscript.

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