PRE-TREATMENTS EFFECT ON PEPPER DRYING TIME AND MICROSTRUCTURE

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ABSTRACT: *Traditional pepper drying process takes 4 to 5 days and sometimes longer, especially during rainy days. This paper presented a study on the effects of two pre-treatment methods to the improvement of the drying rate of black pepper production. Two samples of pepper berries were pre-treated by boiling and freezing, respectively. After pre-treatment, the samples must be immediately dried to prevent microbial infections. To achieve this, each sample was then spun in a rotary drum dryer at a controlled temperature. The weight of the sample was recorded at a certain time interval until the sample has achieved a 12% moisture content. Each sample was also tested using Fourier Transform Near-Infrared (FT-NIR) to check the physical and chemical specifications. Scanning Electron Microscopy (SEM) was used to observe the surface morphology of the skin and core of the samples. Pre-treated samples resulted in shorter drying time compared to untreated samples. Observation of the microstructural changes using SEM showed that the dried core of the pretreated samples was less damaged compared to untreated samples. This study demonstrated that pre-treating pepper berries prior to drying improved the drying time from 8 hours (based on an untreated sample) to 2½ hours. Dried pre-treated samples also achieved the standard moisture content. Thus, the application of a rotary drum dryer to dry the pre-treated pepper is the novelty of this research.*

Keywords: Black pepper, Boiling, Freezing, Drying Time, FT-NIR, Moisture Content, Rotary drum dryer, SEM

1 INTRODUCTION

Malaysia is the fifth largest pepper producer in the world after Indonesia, Brazil, Vietnam, and Malabar [1]. Sarawak, a Malaysian state located in Borneo, is the main pepper producer in Malaysia. The pepper plantation in Sarawak alone covers approximately 16,000 hectares. The year 2007 to 2016 observed an increasing trend of pepper planting in Sarawak [2, 3]. The trend highlights that pepper is an important cash crop and source of income to the locals. Furthermore, in alignment with one of the United Nation's sustainable development goals i.e. goal 12 on responsible consumption and production, it is pertinent to investigate aspects that improve management and efficient use of local crops such as pepper.

Regardless of the scale of the pepper farm, drying is an important process in pepper production. Pepper berries have to undergo the drying process to meet the specific level of moisture content. Drying is a process to remove water content by passing hot air through it to hinder any microbial growth in order to improve the shelf life of the food [4, 5]. During the drying process, browning enzyme and phenolic compounds are oxidized by atmospheric oxygen under the catalytic influence of the enzyme phenolase and eventually turn the green colour of matured pepper pigment into the black [6, 7].

Most of the fresh agricultural products have a moisture content of more than 50% and it needs to be dried properly until 5-25% depending on the type of food [8] and 12% for black pepper [9]. Over-drying of peppers results in loss of volatile flavour components and loss of total weight, consequently less return to the farmers. On the other hand, if the peppers are not dried properly, it is exposed to mould and fungus attack.

In Malaysia, many pepper producers use the traditional method of drying which is direct sun drying. Pepper berries are spread on mats in a big open space directly under the sunlight. Drying pepper berries under the sun does not incur any cost to the producers, however, the method cannot produce uniform heat. To ensure that the pepper berries are uniformly dried, it needs to be manually turned from time to time during drying, which can be burdensome [10]. The traditional method of drying peppers usually takes 4 to 5 days during a sunny day and the duration can prolong during rainy days. Also, the quality attributes of peppers may degrade when exposed to solar radiation for too long [11]. For commercialization purposes, the drying process is a critical factor. An optimum drying system that is able to shorten the drying time, minimize damage to the product, and lower operating cost are always the main concern to the pepper producers [12].

Pre-treatment in the food drying process alters the macroscopic properties of the materials due to microstructural changes [13, 14]. It is reported that various methods of pre-treatment affect dried agricultural food materials in terms of nutrients [15, 16, 17]. Macrostructure and microstructure of the products during the drying process changes due to the formation of internal cracks and shrinkage stresses which pull the tissue apart, which consequently affect the food product moisture flow [18]. Previous researches have shown significant improvement in reducing the drying time of pre-treated agricultural food products as compared to untreated ones [19, 20]. In addition, the pre-treatment method of blanching is also advantageous as it removes pesticide residue and reduces microbial load [21].

Observation of the microstructural change can be done using Scanning Electron Microscopy (SEM). The microscopic study of various food products using SEM have been done by a few researchers: red pepper samples [22], sweet potato [14], milk powder [23], Chinese angelica slices and Astragalus slices [24], and banana slices [25].

To be commercially acceptable, peppers must meet the desired specification, mainly measured by its moisture content. The traditional drying process is very conservative, labor-intensive, unhygienic, and weather dependent. These factors may contribute to the inconsistent moisture content in a batch of pepper berries which consequently affects the product's quality. The traditional drying process is not productive and produces inconsistent product quality. Therefore, there is a need to find a method that can expedite the drying process and at the same time meet the desired quality.

This study investigates the improvement of drying rate of black pepper production when two different pretreatment method is used i.e. by freezing and boiling of pepper berries. The drying process of pre-treated samples was done using a rotary drum dryer, as described in section 2. Effects of pre-treatments on drying time and microstructure of the black pepper were studied and discussed in section 3. Section 4 of this paper concludes and highlights significant findings of this study which can be beneficial to small and medium-sized pepper producers.

2 MATERIALS AND METHODS

2.1 Samples

A small number of fresh pepper berries were obtained from a local farmer. The pepper berries were divided into 3 groups of samples, each with the same initial weight of 100g, as depicted in Figure 1. The berries were manually threshed to remove the stalks. One of the samples was marked as an untreated sample, referred to as UNTREATED in the following parts of this paper. The other two samples were pre-treated using two different pre-treatment methods i.e. by freezing and by soaking in boiling water, referred to as FREEZING and BOILING, respectively.

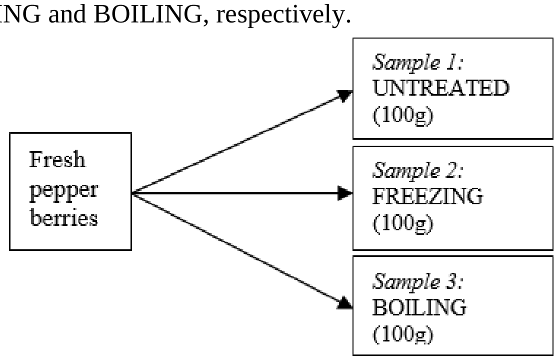

Figure 1. Fresh pepper berries grouped into 3 samples named UNTREATED, FREEZING, and BOILING.

2.2 Experimental Setup

A schematic diagram of the pepper drying experiment is shown in Figure 2. Rotary drum speed was set to 800 revolutions per minute (RPM) throughout the drying process. Charcoal is used as a source of heat in the experiment. As shown in Figure 2, a rectangular wooden box was fabricated to cover the entire body of the drum dryer, with a small hole on top of the box for ventilation. The wooden box is designed to trap heat so that the samples inside the dryer are exposed to the heat from the charcoal. Thermocouple type K was used to measure the temperature inside the wooden box and placed at the same level as the rotating drum on the right side. The range of temperature during the experiment must be kept below 70°C [9]. Regular checking of the thermocouple was done to ensure the temperature was within the range. If the temperature goes beyond 70°C, the samples will be burnt and eventually ruined the chemical properties. When the temperature started to drop, more charcoals were added. Sections 2.2.1 - 2.2.3 explains the processes conducted on each group of samples.

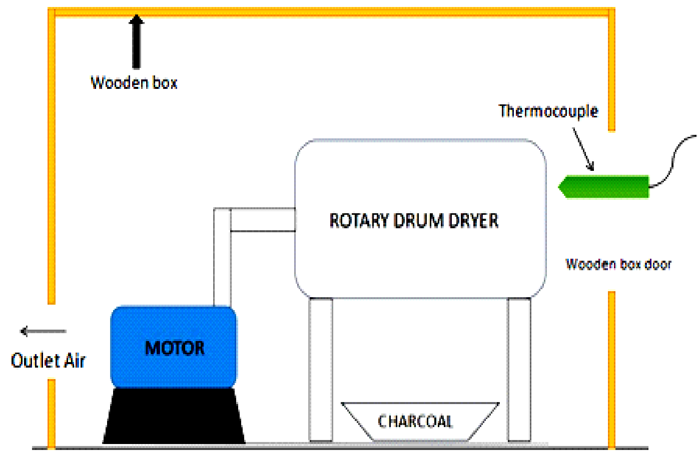

Figure 2. Schematic diagram of the pepper drying experiment

2.2.1 Untreated, spin, and heated (UNTREATED)

A clean and untreated fresh pepper berries sample was placed into the drum dryer. After the drum dryer was secured, burnt charcoal was placed under the dryer, then the drum dryer was switched on. The weight of the UNTREATED sample was recorded for every hour until it reached a constant weight.

2.2.2 Pretreatment by freezing, spin and heated (FREEZING)

Clean and fresh pepper berries sample was placed in the fridge overnight at 3°C. The FREEZING sample was taken out and the frozen pepper berries were weighed and recorded. The weight after being frozen for 12 hours is 98.6 g, a reduction in weight by 1.43%. The same procedures as mentioned in section 2.2.1 were repeated until the desired constant weight was achieved.

2.2.3 Pretreatment by boiling, spin and heated (BOILING)

Fresh pepper berries were soaked in boiled water for 10 minutes. After 10 minutes, water was drained and the boiled pepper berries were weighed and recorded. After being soaked in boiled water for 10 minutes, the BOILING sample weight was 102 g, an increase in weight by 2%. The further drying process was conducted as described in section 2.2.1. The experiment was carried out until the desired constant weight was achieved.

2.3 Fourier Transfrom Near-Infrared (FT-NIR)

All dried samples (UNTREATED, FREEZING, and BOILING) were kept in an air tight container. The dried samples from each batch were examined using Perkin Elmer Spectrum One NTS for the FT-NIR test. FT-NIR is a non-destructive test to measure the moisture content, volatile oil, piperine, non-volatile ether extract, and total ash of the samples for each treatment. Samples from each batch were placed on a petri dish.

2.4 Scanning Electron Microscopy (SEM)

SEM test was conducted to observe the morphological surface of the samples. After the pre-treatment processes were completed, one random pepper berry was selected from each sample. The randomly selected pepper berry was cut and the cross-section yields the core of the pepper berry. A small portion of the pepper's skin and core were observed under SEM. All the specimens were mounted on the specimen stub. After that, the specimens were coated with a thin layer of conductive material using a sputter coater. Then, the specimens were loaded under Tabletop Microscope TM3030. All the samples were analyzed at magnifications of x500 (200µm).

3 RESULTS AND DISCUSSION

3.1 Effect of Pre-Treated Sample on Drying Time

Table 1, Table 2, and Table 3 summarise the parameters collected during the experiment for UNTREATED, FREEZING, and BOILING samples, respectively. For each batch of samples, 3 parameters were collected i.e. drying time, initial weight (W_I), final weight (W_F), weight loss (W_L), and temperature recorded by the thermocouple. From time to time, the sample was taken out and weighed to ensure it meets the desired moisture content. Drying time for minute 400th and 480th shows a very small difference that indicates the sample has reached the optimum moisture loss. As shown in Table 1-3, the temperature inside the wooden box was not consistent, ranging between 47°C to 57°C but was kept lower than 70°C to avoid pepper berries from being burnt. It is difficult to control the temperature inside the box due to the use of charcoal as the source of heat.

Table 1. Parameters collected for UNTREATED samples

Drying time (min)	$W_L = W_F - W_I$ (%)	Temperature (°C)
60	4.25	47
120	12.24	49
180	24.95	54
240	38.63	53
300	63.33	55
340	67.61	54
400	71.35	55
480	71.40	57

Table 2. Parameters collected for a FREEZING sample

Running time (min)	$W_L = W_F - W_I$ (%)	Temperature (°C)
120	68.73	52
150	69.81	57

Table 3. Parameters collected for BOLING sample

Running time (min)	$W_L = W_F - W_I$ (%)	Temperature (°C)
120	65.01	52
150	67.21	57

Sample weight against drying time obtained from all experiments are summarised in Figure 3. In general, the 3 groups of samples show a similar trend in which weight decreases during the drying process. At the end of each curve (Figure 3), the weight becomes constant. As shown in Figure 3, the pre-treated samples had a shorter drying time compared to the UNTREATED sample. The UNTREATED sample took 480 minutes to achieve constant weight while pre-treated samples by FREEZING and BOILING took 150 minutes to reach constant weight. Pre-treated samples reduce nearly 70% of the drying time compared to the UNTREATED sample. According to Tunde-Akitunde [20], the drying rate of the samples can be improved by using some pre-treatment methods since the pre-treatment remove the surface resistance. Thus, the finding in this study showed significant improvement in pre-treated samples drying time which is in agreement with Tunde-Akitunde [20].

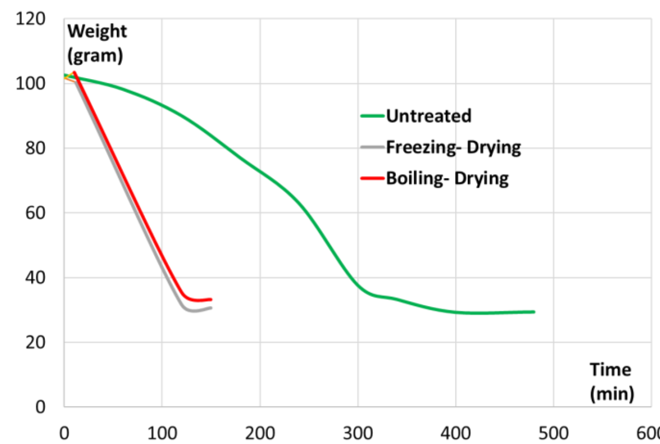

Figure 3. Sample's weight against drying time of UNTREATED, FREEZING, and BOILING samples

During FREEZING pre-treatment, 1.4% of the initial sample weight was lost before the heating and spinning process took place. It is suggested that the dehydration process occurred during the freezing process. In contrast, the BOILING sample showed an increase in weight compared to the initial sample weight. This is due to water absorption during the soaking process in boiling water. Boiled water that soaked the BOILING sample turned the pepper berries tender, thus making it easier to release the pepper berries moisture to the immediate surroundings [26]. Referring to Figure 3, the trend of the curves for FREEZING and BOILING pre-treated samples are similar. However, it is noted that the drying time for the FREEZING sample is shorter than the BOILING sample. This finding suggests that freezing the pepper berries before drying can improve the drying rate. Similar trends were shown by other researches on different foods such as avocado [27]; beans, carrots, and potato [28].

3.2 FT-NIR Spectroscopy

FT-NIR spectrum was used to identify the physical and chemical specifications of the samples. Table 4 summarised the FT-NIR result of the dried samples and compared it to the Malaysian Pepper Board (MPB) standard. Generally, the samples are over-dried as the percentage of moisture content was lower than 12%.

Table 4. FT-NIR results for the dried samples

Sample ID	Moisture (%)	Volatile Oil (%)	Piperine (%)	NVEE (%)	Total Ash (%)
MPB Standard*	< 12.0	>1.0	> 4.0	> 6.0	< 5.0
BOILING	9.46	5.02	7.48	4.63	1.60
FREEZING	8.11	4.55	6.85	4.84	4.84
UNTREATED	6.76	4.23	7.54	4.35	0.85

The standard guideline from MPB is included for comparison. Basically, all the samples have successfully passed the standard guideline except for the non-volatile ether extract (NVEE). The results are slightly lower than the standard requirement. The moisture contents of the UNTREATED, FREEZING, and BOILING samples were 6.76%, 8.11%, and 9.46% respectively. The samples were over-dried which had passed the standard requirement of 12% moisture content. Lower NVEE was due to an over-dried sample. Therefore, the actual drying time for all samples to achieve the standard requirement is supposedly shorter than the results shown in Figure 3.

The BOILING sample shows the highest percentage of volatile oil followed by FREEZING and UNTREATED samples. According to Bartholomew & Sinclair [29], regardless of the thickness of the fruits, the volatile oil can be found in specialized receptacles (glands) in the surface layers of the peel. A similar study was conducted in [30] on the effect of different drying methods on the volatile compounds on Lactarius deliciosus. Thus, the studies confirmed that pre-treated methods affect the volatile oil content of the berries. Meanwhile, BOILING and UNTREATED show quite a similar amount of piperine content. FREEZING showed the lowest percentage of piperine and exceed the standard minimum requirement of 4%. UNTREATED samples show the lowest percentage of total ash compared to BOILING and FREEZING samples.

3.3 SEM Morphology

Figure 4(a)-(c) show the SEM images of the fresh and pre-treated samples of pepper berries skin before the drying process.

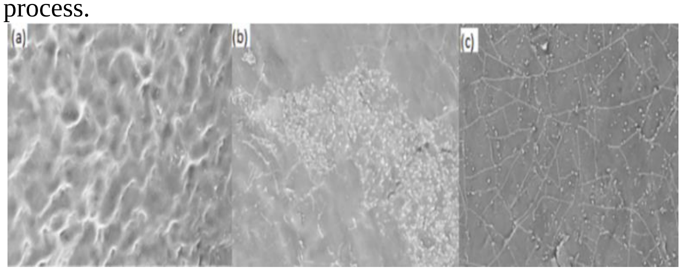

Figure 4. SEM micrographs of pepper berry skin (a) fresh, (b) boiled, (c) frozen.

SEM gives a better image of microstructure changes of pre-treated and untreated samples. Figure 4(a) shows a smooth surface with no pore observed. In Figure 4(b), after being soaked in boiling water, tiny water molecules can be seen on the skin of the pepper berry. In Figure 4(c), a cracked surface is observed on the frozen pepper berry. The surface of the frozen sample cracked as the moisture content in the berry was converted to solid-state. It is also mentioned in the research done by Krokida & Maroulis [31] that the structural damage and remarkable changes to the structural properties can be seen after the diffusion of water during drying forms cracks to the solid structure.

As shown in Figure 4(b) and (c), pre-treatment alters the microstructure of the pepper berry i.e. in the forms of cracks and water molecules on the pepper skin. This finding suggests that the pre-treatment methods actually damage the plant cuticle. The plant cuticle is a protective hydrophobic layer that coats the epidermal surfaces of the fruit so that it can control gas and water exchange with the environment as well as to protect from pathogens [32]. Thus, microstructure changes on the pepper berry skin enable moisture to be released easily and quickly compared to the non-treated sample. However, the drying process should be conducted continuously until the pepper completely dried (12% moisture content) since the damaged skin of the treated pepper can no longer protect the pepper from stringent pathogens.

Figure 5(a)-(c) show the SEM images of the fresh and pre-treated samples of pepper berries core before the drying process.

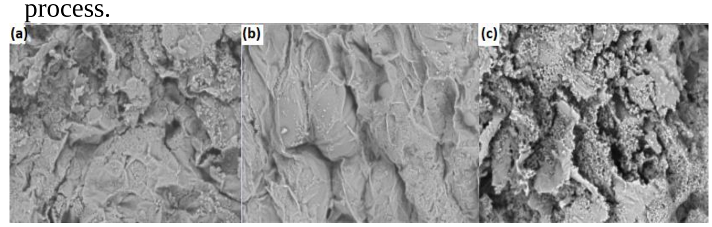

Figure 5. SEM micrographs of pepper berry core (a) fresh, (b) boiled, (c) frozen.

The core of fresh pepper berry showed less porosity compared to frozen berry. The tightly packed structure that has fewer pores in Figure 5(a) caused slower moisture removal during drying [31] thus the time required to dry the untreated sample is longer than the pre-treated one. Figure 5(b) shows swollen-like tissue. This revealed the presence of water absorption in the berries during soaking pre-treatment. In Figure 5(c), porosity is evident in the frozen sample. The porous structure is an advantage quality for the drying process as porous structures expedite the moisture released [31].

Further study has been conducted to observe the dried samples via SEM. SEM images of the dried pepper berries skin for different treatments are shown in Figure 6(a)-(c).

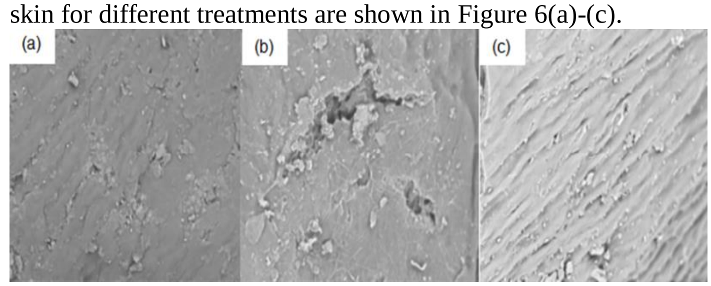

Figure 6. SEM micrographs of dried pepper skin (a) untreated, (b) pre-treatment by boiling, (c) pre-treatment by freezing.

Changes in structural properties can be seen after drying as the pepper moisture was removed. From the micrograph, every drying method affects the structural changes in the samples. Similar results were also observed in other studies: [13, 14, 15, 16, 17, 31]. Figure 6 (b) shows a rough surface of the skin with some burst area detected. Boiled water that was used to soak the sample makes the plasmatic membrane to soften during the pre-treatment. Cracked skin of frozen berries as shown in Figure 4(c) contribute to the observation of images in Figure 6(c).

Figure 7(a)-(c) show the results of the microstructure of the dried core samples. A large number of porous honeycomb-like structures were spotted in the core of dried boiling pepper in Figure 7(b) compared to only a small amount in Figure 7(a). The cell wall of the pre-treated samples shows less damage compared to the untreated one. Similar findings found by Luo, Patterson, & Swanson [33] with the treated apple tissue with papain that showed less severe cell wall breakdown than the one without treatment. Figure 7(c) shows less shrinkage among all the samples.

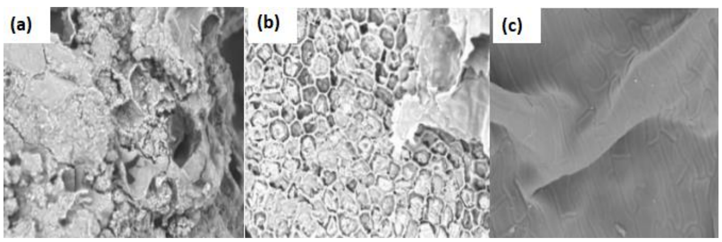

Figure 7. SEM micrographs of dried pepper core (a) untreated, (b) pre-treatment by boiling, (c) pre-treatment by freezing.

4 CONCLUSION

Drying with different pre-treatment methods affect the microstructures of both the outer and inner parts of pepper berry. In this study, it was found that pre-treatment can improve pepper drying time. Prior to drying, microstructure changes were observed on the pre-treated samples. The rate of loss in weight against time for the untreated sample is low, as it took about 8 hours to dry the sample. On the other hand, the pre-treated samples have a higher rate of loss in weight against time as it took about 2½ hours to dry. The pre-treatment methods also improved the appearance of the treated samples which can be observed on the pre-treated dried core of the pepper berry.

Pre-treatment by freezing shows the fastest way to dry the pepper berries. However, in terms of practical and economical point of view, the boiling pre-treatment method is recommended. Freezing treatment requires a large freezer for large-scale production, hence the cost of freezer and electricity must be considered. Meanwhile, boiling pre-treatment only requires soaking pepper berries in boiled water for about 10 minutes, thus is more practical and economical. Furthermore, the overall physical and chemical specifications of the boiling treatment showed better results compared to freezing.

Generally, pretreatment methods are able to improve the current drying time. On top of that, the use of a rotary drum dryer enables the drying process to be done continuously indoors in hygienic conditions. The combination of pre-treatment methods and rotary drum dryer allows for shorter drying time with a minimum number of workers and space. The quality of the product can be improved too, thus enable higher profit gain by the farmers. The findings in this study are expected to assist the black pepper production among small and medium-sized farmers.

5 ACKNOWLEDGEMENTS

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