

### **TROPICAL AGRICULTURAL SCIENCE**

Journal homepage: http://www.pertanika.upm.edu.my/

## Physicochemical Characterisation of White Pepper: A Comparative Study Between Traditional Sun Drying and Convective Rotary Drum Drying Methods

Ana Sakura Zainal Abidin<sup>1\*</sup>, Mohamad Syafiq Iylia Jamadi<sup>1</sup>, Sinin Hamdan<sup>1</sup>, Mohammad Omar Abdullah<sup>2</sup>, Gloria Elaine John<sup>1</sup>, Annisa Jamali<sup>1</sup>, Rasli Muslimen<sup>1</sup> and Zehnder Jarroop Augustine Mercer<sup>3</sup>

<sup>1</sup>Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia <sup>2</sup>Department of Chemical Engineering and Energy Sustainability, Faculty of Engineering, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia <sup>3</sup>Malaysian Pepper Board, Jalan Utama, Tanah Putih, 93916 Kuching, Sarawak, Malaysia

### ABSTRACT

Drying is a crucial process in preserving the physicochemical qualities of white pepper. This study investigates the impact of two drying processes, namely traditional sun drying (TSD) and rotary drum drying (RDD), on the quality of white pepper. TSD requires three consecutive sunny days for drying, whereas RDD achieves the target moisture content of 12% within a rapid drying time of 120 min. The research employs thermogravimetric analysis (TGA), Fourier transform infrared (FTIR) spectroscopy analysis, and scanning electron microscopy (SEM) to analyse the dimensions, thermo-physical profiles, chemical constituents, and microstructure of the pepper samples. RDD, with a drying temperature of 55°C and centrifugation force of 129.7 × g, ensures fast and uniform drying while preserving the physicochemical qualities of white pepper. In terms of physical

#### ARTICLE INFO

Article history: Received: 23 October 2023 Accepted: 05 December 2023 Published: 27 August 2024

DOI: https://doi.org/10.47836/pjtas.47.3.26

E-mail addresses:

zaasakura@unimas.my (Ana Sakura Zainal Abidin) msiylia96@gmail.com (Mohamad Syafiq Iylia Jamadi) hsinin@unimas.my (Sinin Hamdan) amomar@unimas.my (Mohammad Omar Abdullah) gloriajohn97@gmail.com (Gloria Elaine John) jannisa@unimas.my (Annisa Jamali) mrasli@unimas.my (Rasli Muslimen) zehnder@mpb.gov.my (Zehnder Jarroop Augustine Mercer) \* Corresponding author characteristics, RDD results in larger dried pepper dimensions, measuring 4.56 mm on average, compared to TSD, which measures 4.35 mm. SEM observations reveal varying pore sizes and cracks in both drying methods. Additionally, quality validation conducted by the Malaysian Pepper Board demonstrates that RDD exhibits superior quality compared to TSD. The RDD samples show moisture content, piperine, volatile, and ash percentages of 11.83, 8.18, 2.53, and 0.82, respectively, while the TSD samples show 10.37, 7.16, 2.43, and 0.74. All samples complied with Standard Malaysian White Pepper No. 1 and International Pepper Community Grade 1. Future studies should focus on enhancing different drying methods to achieve efficient white pepper drying while preserving its quality.

Keywords: Agrotechnology, FTIR, SEM, TGA

### INTRODUCTION

White pepper has extensive applications in the food, pharmaceutical, perfumery, and cosmetics industries (Megat et al., 2020; Olalere et al., 2017; Tiwari et al., 2020). It is derived from dried, skinless mature pepper berries, scientifically known as Piper nigrum L. (Salehi et al., 2019). There are various types of Piper nigrum, including Semongok Emas, Semongok Aman, Kuching, Semongok Perak, and India (Azman et al., 2020; Chen & Tawan, 2020). The Kuching variety is particularly suitable for white pepper production due to its thinner pericarp, facilitating the extraction of smooth and ivory-coloured white pepper (Azman et al., 2020; Malaysian Pepper Board [MPB], 2017a; Megat et al., 2020).

White pepper is produced from yellowish-green mature berries and red, fully ripe pepper berries (Azman et al., 2020; Singh et al., 2013). The berries undergo a soaking process to remove their pericarp (Azman et al., 2020). The physical structure of pepper berries consists of a fleshy outer part surrounding a single shell of hardened endocarp with a single seed inside. The pericarp comprises three sections: exocarp (outer layer), mesocarp (middle layer flesh), and endocarp (innermost layer). Following soaking, the berries must undergo immediate drying (Aziz et al., 2019).

Pepper drying is a crucial process for moisture removal while preserving the chemical properties, which can easily deteriorate during drying. External factors such as temperature, humidity, airflow, drying volume, and pressure influence the drying process (Abidin et al., 2020; Mühlbauer & Müller, 2020). Increasing air temperature accelerates moisture removal, thereby shortening drying time, but excessively high temperatures can degrade the chemical properties (Abidin et al., 2020; Rigit et al., 2013). Generally, pepper drying methods can be categorized as traditional sun-drying and modern methods such as drum drying, convective drying, infrared drying, and vacuum drying.

Sun drying is widely practised today for its simplicity, convenience, and low cost (Lamidi et al., 2019; Saha et al., 2022). However, it is time-consuming, unhygienic, and reliant on weather conditions (Abidin et al., 2020). Direct exposure to sunlight affects product quality, leading to vitamin loss, aroma degradation, and uneven drying (Lamidi et al., 2019; Saha et al., 2022). Therefore, RDD has been developed to improve pepper drying and energy efficiency (Friso, 2023). RDD is a very suitable and effectively dry grainy biomass material as white pepper berries (Kaveh & Abbaspour-Gilandeh, 2020; Rezaei & Sokhansanj, 2021). RDD operates by tumbling the drying material in a rotating drum with hot air to expedite moisture removal. The horizontal position of the drum allows gravity to assist the material's movement inside (Trojosky, 2019; Wae-hayee et al., 2021). Flights or fins within the drums improve the material contact with hot air, which enhances thermal and moisture diffusivity (Wae-hayee et al., 2021). Air velocity and drum rotation influence the material's drying capacity, drying time, and final moisture content (Kerr, 2019). RDD has significantly improved the drying time of black pepper from 4-7 days of sun drying to a few hours (Abidin et al., 2020). Rotary drum drying offers rapid, hygienic and uniform batch drying.

The quality of white pepper can be assessed through chemical analysis, including moisture content, oleoresin content, volatile oil, piperine, and starch (Aziz et al., 2019; Chithra et al., 2011). Physical quality is determined by surface appearance, colour, bulk density, and extraneous matter in the white pepper sample (Aziz et al., 2019). The MPB and the International Pepper Community (IPC) have established standard specifications for the chemical and physical quality of white pepper products (Tables 1 and 2). Compliance with these standards is crucial for white pepper products to enter the international market. Therefore, ensuring that the proposed drying process can produce white pepper that meets the specified chemical and physical quality standards is essential.

Pepper berries are rich in chemical components such as minerals, lignans, flavonoids, aromatic compounds, amides, and primary alkaloids or lipids. When it comes to white pepper, there are eight important chemical parameters to consider: piperine, volatile oil, oleoresin,  $\beta$ -caryophyllene, sphericity,  $\alpha$ -pinene, limonene, and moisture content (Chithra et al., 2011; Olalere et al., 2018; Salehi et al., 2019). Piperine (C<sub>17</sub>H<sub>19</sub>NO<sub>3</sub>), an amide alkaloid, is a valuable nutrient

#### Table 1

	Grade					
Characteristic	Standard Malaysian White Pepper No. 1	Sarawak Special White	Sarawak FAQ White	Sarawak Field White	Sarawak Coarse Field White	
Moisture, percentage by weight, maximum (%)	12.00	15.00	16.00	16.00	16.00	
Light berries, per cent per weight, maximum (%)	0.20	0.50	1.00	1.50	-	
Extraneous matter, percent by weight, maximum (%)	0.25	0.25	0.50	1.00	3.00	
Amount of black/dark grey berries in white pepper, per cent by weight, maximum (%)	1.00	1.00	3.00	3.00	5.00	

Malaysian Pepper Board (MPB) standard for white pepper (MPB, 2017b)

*Note.* MPB mentions FAQ as the third-class white pepper without any definition of abbreviation (MPB, 2017b)

Table 2

Parameters		Grade			
	Ι	II	III		
Physical properties					
Bulk density (g/L), min	600.0	600.0	550.0		
Light berries/Corn (m/m)%, max	1.0	2.0	2.0		
Extraneous matter (m/m)%, max	0.8	1.5	2.0		
Black coloured berries/Corn (m/m)%, max	2.0	3.0	10.0		
Mouldy berries/Corn (m/m)%, max	1.0	3.0	3.0		
Insect spoiled berries (% by wt.), max		2.0	2.0		
Broken berries (m/m)%, max	2.0	3.0	3.0		
Chemical properties					
Moisture content (m/m)%, max	12.0	13.0	14.0		
Total ash (m/m)% max, on dry basis		4.0	4.0		
Non-volatile matter using ether extract (m/m)% min, on a dry basis	6.0	6.0	6.0		
Volatile matter (oil) (ml/100 g)%, min, on dry basis	1.5	1.5	1.0		
Piperine (m/m)%, min	4.0	3.5	3.0		

International Pepper Community standard specifications for white pepper international market (CAC, 2021)

in pepper but is susceptible to damage during heating processes (Singh et al., 2013). Pepper berries contain piperine in concentrations ranging from 2.4 to 7.4%, co-existing with five other alkaloids and four isomeric forms of piperine (Tiwari et al., 2020). Volatile or essential oils are secondary metabolites in plants widely used in fragrance, cosmetics, medicine, and food for their scent and flavour (Nikolić et al., 2015). In white pepper,  $\beta$ -caryophyllene is the primary component of the essential oils, while piperine is present in the oleoresin (Singh et al., 2013). White pepper contains essential oils (1-2.5%) and alkaloids (5-9%), with the major constituents being piperine (1.7-7.4%), chavicine, and piperidine (Kusumorini et al., 2021).

The structure of piperine ( $C_{17}H_{19}NO_3$ ), also known as 1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl], consists of three

functional groups: aromatic, aliphatic, and amide (Tiwari et al., 2020). Piperine is a weak base compound with a distinct pungent flavour, smell, and hot effect. Its functional groups can be detected using FTIR include C-H (peaks at 2,800-3,000), O=C-N and C=C (diene) (peak at 1,635), C=C (benzene) (peaks at 1,495-1,589), =C-O-C (peaks at 1,030-1,257), and C-O-C (peak at 1,134) (Kusumorini et al., 2021; Mohammed et al., 2016). According to Sarifudin et al. (2021), piperine functional groups such as C-N aliphatic amines, C-O stretch ethers, C-N stretch aromatic amines, C-C stretch in ring aromatics, and C=C stretch alkenes are observed as peaks at wavenumbers of 1,134, 1,193, 1,251, 1,492, and 1,633 per cm, respectively. White pepper also contains minerals and trace elements, including sodium, magnesium, potassium, and calcium, which exhibit wide variability in

white pepper extracts (oleoresins) (Olalere et al., 2019). Thus, the chemical properties of white pepper, such as moisture content, volatile matter, piperine, non-volatile matter, and ash, justify the quality standards used to assess its quality.

TGA provides high sensitivity, reproducibility, and response to even minor mass variations. The sample's temperature range and thermal stability help identify peak profiles (Picolotto et al., 2020). Thermal profiling of white pepper's physical stability using TGA shows weight loss peaks in the temperature range of 270-350°C, indicating the decomposition of crude protein, lipid, and starch (H. Liu et al., 2018). At the same time, FTIR spectroscopy analysis is a reliable, cost-efficient, non-destructive analytical technique for physicochemical study that allows for the accurate, rapid, and direct assessment of various functional group properties (Candoğan et al., 2021; Wang, 2012). Studies have demonstrated that FTIR and TGA can provide valuable data for identifying herbs and spices (L. Liu et al., 2020; Laouni et al., 2022; Wang, 2012). Therefore, FTIR and TGA are convenient techniques for investigating the structural and compositional changes of macromolecules found in spices and herbs, including white pepper.

The drying process involves mass and energy transfer, with moisture vaporized by heat and diffused through pores, affecting the pepper berries' microstructure (Surendhar et al., 2019). The drying method employed significantly influences microstructural changes in the berries, as well as their moisture diffusivity and drying rate. Scanning electron microscopy (SEM) is commonly used to observe microstructural changes in the drying process of various agricultural products, including black pepper, red pepper, sweet potato, Chinese angelica, *Astragalus*, and banana (Abidin et al., 2023).

Sun drying is convenient for Sarawak local producers, although it is ineffective and unhygienic. Extensive research has been conducted on drying agricultural products, but limited studies focus on white pepper drying. Concerns have been raised regarding the physicochemical quality of the white pepper produced using rapid modern drying methods. Therefore, the main objective of this study is to characterize the white pepper produced through two different drying methods, TSD and RDD. The thermophysical profiles and chemical constituents of the results were compared.

## MATERIALS AND METHODS

### White Pepper Sample Preparation

Fresh Kuching variety pepper was obtained from a Simunjan, Sarawak, Malaysia farmer. The pepper berries underwent a traditional soaking process. The soaked berries were then divided into two groups for drying using two different methods: TSD and RDD. Detailed schematic diagrams and drying procedures of RDD have been explained and adapted from Abidin et al. (2020). For the RDD method, a constant drum speed with centrifugation force of 129.7 × g and a chamber temperature of 55°C were maintained. The samples were dried until they reached the desired moisture content. The drying process of 200 g sample for TSD took three consecutive sunny days, and RDD required 120 min of drying time. A portion of the dried pepper berries from both methods was sent to the MPB for quality validation. Another portion of the samples was prepared for further characterization using analytical techniques, namely FTIR spectroscopy, TGA, and SEM. These techniques were employed to analyse the chemical and physical properties of the white pepper samples.

### **Quality Validation**

The quality of the samples obtained from both drying methods was validated at the MPB Central Testing Laboratory. MPB is an accredited institution that follows the MS ISO/IEC 17025:2017 standard. MPB is responsible for conducting analyses to determine the natural chemical content in pepper samples, including piperine, volatile oil, non-volatile ether extract, total ash, acid insoluble ash, and aroma profile. The PerkinElmer Spectrum One Near-infrared Testing System Fourier Transform Near Infrared (NTS FT-NIR) Spectrometer (USA) was employed for the analysis. The FT-NIR machine has been synchronized to the database of laboratory analysis results on the chemical content of white pepper. The sample was analysed in its berry form. The chemical content percentage provided by the FTNIR machine has errors up to 1% (max).

### Sizing and Dimensions

The white pepper berries obtained from RDD and TSD methods were measured

using a vernier calliper. Specifically, the Pro'sKit model PD-153 (Taiwan) standard vernier calliper with a sensitivity of 0.02 mm and made of AISI 430 stainless steel was utilized for the measurements. A total of 100 white pepper berries from each drying method were selected for measurement. For each berry, three readings were taken using the vernier calliper, recording the measurements for the first axis (a), second axis (b), and third axis (c) of the diameter. The average of the three axes was then calculated, and the diameter measurement for the respective white pepper berry sample was considered, as Megat et al. (2020) described.

### **Thermo-Gravimetric Analysis**

TGA was performed using Shimadzu DTG-60H simultaneous DTA-TG (USA) apparatus capable of high-temperature measurements up to 1,500°C and an inert gas atmosphere or air (oxygen). The heating rate, atmosphere, gain or loss of mass, temperature, and heat flow were applied to identify the mass profile for white pepper berries. For the analysis, reference alumina  $(Al_2O_3)$  crucibles were used. The sample was prepared by placing one white pepper berry, weighing approximately 100 mg, into the crucible. The TGA experiment involved heating the sample from room temperature (25°C) to 700°C at 10°C/min while maintaining a streaming nitrogen  $(N_2)$  atmosphere of 100 ml/min. The TGA equipment was calibrated using a calibration set/manual provided by Shimadzu (USA). Each TGA analysis was repeated four times to ensure reliable and consistent results. By monitoring the variations in mass as a function of time and temperature under a dynamic atmosphere, TGA enabled the examination of the thermal behaviour of the white pepper berries.

### Fourier Transforms Infrared Spectroscopy

This study performed infrared-spectra (IR-spectra) analysis to determine the chemical bonds and functional groups in the sample constituents within the 4,000-400 per cm range. The analysis was conducted using attenuated total reflectance Fourier-transform infrared (ATR-FTIR) spectroscopy on a Shimadzu Fourier Transform Infrared Spectrophotometer (Model IRAffinity-1S, USA). The FTIR analysis for both samples was performed according to the method by Chumroenphat et al. (2021) with modification to the standard procedure introduced by MPB that utilized white pepper samples in berry form. The analysis was repeated five times using duplicate samples to ensure reliable results.

# Surface Morphology by Scanning Electron Microscopy

Five white pepper berries were randomly selected from each sample group for surface structure observation. Hitachi scanning electron microscopy TM4000Plus (Japan), equipped with the TM 4000plus program, was employed for this purpose. The selected pepper berry from each sample was mounted on a sample stub and coated with a thin layer of conductive material, gold, using a sputter coater (Abidin et al., 2023). The surface of the sample was then observed under the scanning electron microscope at magnifications of  $150 \times (300 \ \mu\text{m})$  and  $600 \times (50 \ \mu\text{m})$ . It allowed for a detailed examination of the surface characteristics and microstructure of the white pepper berries.

### **Statistical Analysis**

A one-way analysis of variance (ANOVA) analysis was conducted using Microsoft Excel software to assess the significant differences between the mean values of the parameters related to berry size. The data were presented as the mean of 100 determinations with the corresponding standard deviation. A *p*-value of 0.05 or less was considered statistically significant, indicating a meaningful difference between the mean values of the compared parameters.

## **RESULTS AND DISCUSSION**

### **Quality Validation**

Samples from both drying methods were taken to the MPB laboratory for FT-NIR analysis validation. The average percentages of moisture, piperine, volatile oil, and ash content for the samples from both drying methods are presented in Table 3. The analysis was conducted in five duplications. The test was conducted using whole forms of white pepper berries. Based on the MPB and IPC standards (Tables 1 and 2), it can be observed that the samples from both drying methods were below the maximum limit of 12% moisture content,

Table 3

Comparison of chemica	l quality analys	is for white	e pepper	samples from	rotary drum	drying (RI	DD) and
traditional sun drying (T	'SD) methods wi	th the Intern	ational P	Pepper Commi	inity (IPC) and	d Malaysiaı	n Pepper
Board (MPB) standards							

Chemical quality	IPC Grade 1 White pepper	MPB	RDD	TSD
Moisture (%)	≤12.00	≤12.00	11.83	10.37
Piperine (%)	<u>≥</u> 4.00	<u>≥</u> 4.50	8.18	7.16
Volatile (%)	≥1.50	<u>≥</u> 0.80	2.53	2.43
Ash (%)	<u>≤</u> 3.50	<u>≤</u> 2.00	0.82	0.74

which is in line with the quality standards. However, the TSD sample had a moisture content of 10.37%, indicating that it was over-dried compared to the RDD sample, which had a moisture content of 11.83%. Over-drying the berries can reduce the farmer's income, as the price is typically based on the product's weight.

On the other hand, the piperine content in both drying methods exceeded the minimum standard requirement, with the RDD sample containing 4.18% more piperine compared to the standard and the TSD sample containing 3.16% more piperine. Similarly, the volatile oil content for both samples met the standard requirements and exhibited a minimal difference of 0.1% between the two methods.

The ash content in both samples was significantly lower than the standard requirements, with the RDD sample showing a reduction of 2.68% and the TSD sample showing a reduction of 2.76%. It is important to note that the ash content was nearly identical for both samples, as they originated from the same source.

In summary, the results indicate that the RDD method yielded better quality white pepper compared to the TSD method, as it retained higher levels of piperine and volatile oil content, essential factors contributing to the overall quality of white pepper.

### Sizing and Dimensions

The dimensions of 100 white pepper berries dried using the RDD and TSD methods were analysed through ANOVA statistical analysis using Microsoft Excel. The analysis revealed a *p*-value of less than 0.05, indicating significant differences in berry size between the two drying methods. Tables 4 and 5 summarize the dimension data obtained from the analysis. The average berry size for RDD was 4.56±0.29 mm, while for TSD, it was 4.35±0.23 mm. Both average sizes meet the standard specifications for white pepper, with the IPC standard requiring a size range of 2-6 mm (Codex Alimentarius Commission [CAC], 2017) and the MPB specifying creamy white pepper to have a size greater than 4 mm (MPB, 2017b). The results reveal that the RDD sample exhibited a slightly larger size, approximately 0.2 mm larger, compared to the TSD sample. It suggests that the RDD method resulted in minimal shrinkage during the drying process, as the size of the berries remained closer to their original dimensions.

_				
_	Axis dimension	Average (mm)	Variance (mm <sup>2</sup> )	Standard deviation (mm)
	Diameter	4.56	0.086	±0.290
	а	4.53	0.083	±0.290
	b	4.57	0.089	$\pm 0.300$
	с	4.56	0.086	±0.290

Table 4The average size of rotary drum drying white pepper berries for 100 samples

Table 5

The average size of traditional sun drying white pepper berries for 100 samples

Axis dimension	Average (mm)	Variance (mm <sup>2</sup> )	Standard deviation (mm)
Diameter	4.35	0.054	±0.23
а	4.35	0.049	±0.22
b	4.36	0.063	±0.25
с	4.35	0.052	±0.23

### **Thermo-Gravimetric Analysis**

The TGA results for samples obtained from the RDD and TSD methods are depicted in Figure 1. The graphs illustrate a consistent trend in thermal profiling, with a narrowing towards the end. The initial region of the graphs represents the loss of water content from the samples' moisture content, with temperatures ranging from room temperature up to 100-110°C, similar to previous findings by H. Liu et al. (2018) and Simonovska et al. (2016). The weight loss represents the evaporated water and other low molecular weight components. Subsequently, a mass break into volatiles occurs between 100 and 170°C, indicating the thermal decomposition of these components, including moisture. The mass loss until 170°C may not be apparent, but it is aligned with the moisture content measured in the corresponding samples. The RDD samples exhibit slightly higher moisture content compared to TSD samples (Table 3).

Notably, the RDD samples exhibit a significant mass dropped approximately 34-40.4% between the temperature range of 280 to 350°C. In contrast, the TSD samples demonstrate different mass drops (40.71-46.97%) within the temperature range of 270 to 350°C. Similar results were observed by H. Liu et al. (2018) in the temperature range of 270-350°C, with a mass drop of 48.07 to 51.16%, representing the decomposition of crude protein, lipid, and starch in the white pepper samples (H. Liu et al., 2018). According to Simonovska et al. (2016), the degradation of cellulose occurs between 240 to 350°C. It is worth noting that slight differences were observed between the results of this research and the literature, which could be attributed to the different forms of the sample used during the TGA analysis. In this study, whole white pepper berries were utilized, whereas other researchers, such as H. Liu et al. (2018), pulverized the samples before conducting the analysis.



*Figure 1*. Thermo-gravimetric analysis graph for white pepper samples from the rotary drum drying (RDD) and traditional sun drying (TSD) methods, respectively

### Fourier Transform Infrared Spectroscopy

Figure 2 shows FTIR transmittance spectra for white pepper samples dried using RDD and TSD methods. In the spectra, characteristic peaks were observed for both drying methods, exhibiting identical peak patterns. In Figure 2, the peaks appear at approximately 3,340-3,290, 2924, and 1,651-1,620 per cm in both samples. It shows that FTIR detects the same functional groups for both TSD and RDD. The results (3,340-3,290 per cm) indicate O-H vibration, which is harmonised with the findings of Simonovska et al. (2016). The sharp peaks at 2,960-2,920 and 2,880-2,860 per cm (Huynh et al., 2020; Simonovska et al., 2016) were assigned to the asymmetrical and symmetrical stretching vibrations of aliphatic C-H methylene groups and olefinic chains, which are typical bands for alkanes and aligned to the result (2,924 per cm). The peak 1,651-1,620 per cm correspond to alkenyl C=C stretch and

O=C-N symmetrical stretching similar to (Kusumorini et al., 2021; Mohammed et al., 2016; Sarifudin et al., 2021) findings.

In the fingerprint region of both spectra shown in Figure 2, peaks were observed at 1,442-1,438, 1,249-1,238, and 1,053-1,018



*Figure 2.* Fourier transform infrared graph for white pepper samples from the rotary drum drying (RDD) and traditional sun drying (TSD) methods, respectively

per cm, with only the TSD sample displaying an additional peak at 883-879 per cm. A peak at 1,600 per cm indicates the vibration of the aromatic skeletal structure, typically associated with lignin peaks, while a peak at 1,450 per cm represents a less intense deformation vibration of C-H in the aromatic ring of lignin moieties (Simonovska et al., 2016). Intense bands at 1,640, 1,440, and 885 per cm are characteristic of *b*-caryophyllene, d-limonene, 3-carene, a-pinene, b-pinene, and other hydrocarbons found in oils, representing stretching and bending vibrations of the C-C, C=C, and C-H bonds, respectively (Huynh et al., 2020). The peak around 1,251 per cm is associated with the C-O groups, esters of fatty acids or ketones (Huynh et al., 2020). In contrast, the symmetrical stretching of =C-O-C occurs around 1033 cm<sup>-1</sup> (Kusumorini et al., 2021; Mohammed et al., 2016; Sarifudin et al., 2021).

Piperine is the main active compound of interest in this study and plays a crucial role in determining the chemical quality of the white pepper samples. Piperine consists of aromatic, aliphatic, and amide functional groups (Tiwari et al., 2020). These functional groups manifest as distinct peaks in FTIR analysis. Specifically, piperine exhibits functional groups of C-H peaks at 3,000-2,800 per cm, O=C-N and C=C (diene) peaks at 1,635 per cm, C=C (benzene) peaks at 1,589–1,495 per cm, =C-O-C peaks at 1,257-1,030 per cm, and C-O-C peaks at 1,134 per cm (Kusumorini et al., 2021; Mohammed et al., 2016). Additionally, Sarifudin et al. (2021), who used pulverized samples,

claimed that piperine functional groups include C-N aliphatic amines, C-O stretch ethers, C-N stretch aromatic amines, C-C stretch in ring aromatics, and C=C stretch alkenes, which are observed as peaks at specific wavenumbers of 1,134, 1,193, 1,251, 1,492, and 1,633 per cm, respectively. In contrast, RDD and TSD analysis only show three peaks of piperine at wavenumbers 1,251, 1,492, and 1,633 per cm, indicating low piperine intensity. However, as shown in Table 3, the piperine content is considerably higher compared to (1.7 to 7.4%) (Kusumorini et al., 2021). Moreover, the RDD and TSD samples prepared for the analysis were in berry form instead of pulverized.

# Surface Morphology by Scanning Electron Microscopy

Figures 3 and 4 present the microstructure of white pepper samples dried using the RDD and TSD methods, respectively. The micrographs clearly show distinct differences in the microstructure between the two samples. The RDD sample exhibits well-defined pores and cracks, while the TSD sample demonstrates a finer, more organised microstructure.

The soaking process of the pepper berries before drying contributes to morphological changes, similar to pretreatment effects (Abidin et al., 2023). Additionally, pore collapse occurs during drying (Kang et al., 2018). The presence of pores and cracks in the microstructure facilitates moisture diffusion. However, the diffusion of water vapour during the



Figure 3. Microstructure of rotary drum drying white pepper at a magnification of: (a) 150'; and (b) 600'



Figure 4. Microstructure of traditional sun drying white pepper at a magnification: (a) 150'; and (b) 600'

rapid drying process leads to the disruption of the solid structure, resulting in notable changes and the formation of cracks in the microstructure of the RDD sample. This phenomenon is attributed to the rapid release of water vapour and the generation of highwater vapour pressure within the sample due to the supplied heat energy during drying (Surendhar et al., 2019). The formation of pores and cracks in the microstructure results from the heat and moisture gradients that occur during the drying process, causing deterioration, deformation, and folding (Chumroenphat et al., 2021). The SEM images significantly illustrate the impact of the drying method on the microstructure of the white pepper surfaces. These observations provide further insights into how microstructural damage can affect the drying time, as well as the chemical and physical quality of the samples.

### CONCLUSION

This study investigated the physicochemical characterization of white peppers dried using two different methods, RDD and TSD. TGA, FTIR, and SEM analyses were performed to assess the drying method's impact on the samples' thermo-physical profiles and chemical constituents. The results provide valuable insights into the impact of drying methods on white pepper's chemical and physical properties.

RDD and TSD samples met the IPC white pepper standard and MPB creamy white pepper specification, indicating their compliance with quality standards. However, the samples from RDD exhibited better chemical properties, as revealed by TGA and FTIR analysis, compared to those from TSD. It can be attributed to the improved drying time achieved through RDD, which had a significant effect on the microstructure of the samples. SEM observations clearly showed the presence of pores and cracks in the RDD samples. Furthermore, the dimension of white pepper berries from RDD surpassed that of TSD samples. It suggests that the controlled drying method accelerates the drying rate and enhances the thermo-physical and chemical properties of white pepper.

In conclusion, this study's findings demonstrate that employing controlled drying methods can expedite the drying rate and improve the thermo-physical and chemical properties of white pepper. The results from TGA, FTIR, and the validation by the MPB laboratory support these findings. These findings open opportunities for further research to explore different drying methods that not only focus on drying efficiency but also aim to enhance the quality of white pepper.

### ACKNOWLEDGEMENTS

The authors express their gratitude for the funding support from the Ministry of Higher Education, Malaysia, through the Fundamental Research Grant Scheme (FRGS/1/2021/TK0/UNIMAS/02/8). They also acknowledge the research facilities as well as the technical assistance Universiti Malaysia Sarawak and the Malaysian Pepper Board provided.

### REFERENCES

- Abidin, A. S. Z., Kifli, M. Z., Jamali, A., Muslimen, R., & Ahmadi, R. (2020). Development of black pepper rotary drum dryer system. *International Journal of Integrated Engineering*, 12(7), 11–19. https://doi.org/10.30880/ijie.2020.12.07.002
- Abidin, A. S. Z., Rolland, M. M., Hamdan, S., Muslimen, R., Tanjong, S. J., Ahmadi, R., Mohamaddan, S., & Jamali, A. (2023). Pretreatments effect on pepper drying time and microstructure. In *AIP Conference Proceedings:* 8<sup>th</sup> Brunei International Conference on Engineering and Technology 2021 (Vol. 2643, No. 1, p. 050043). AIP Publishing. https://doi. org/10.1063/5.0110455
- Aziz, N. S., Sofian-Seng, N.-S., Mohd Razali, N. S., Lim, S. J., & Mustapha, W. A. W. (2019). A review on conventional and biotechnological approaches in white pepper production. *Journal* of the Science of Food and Agriculture, 99(6), 2665–2676. https://doi.org/10.1002/jsfa.9481
- Azman, P. N. M. A., Shamsudin, R., Man, H. C., & Ya'acob, M. E. (2020). Some physical properties and mass modelling of pepper berries (*Piper nigrum* L.), variety Kuching, at different maturity levels. *Processes*, 8(10), 1314. https://doi. org/10.3390/pr8101314
- Candoğan, K., Altuntas, E. G., & İğci, N. (2021). Authentication and quality assessment of meat

products by Fourier-transform infrared (FTIR) spectroscopy. *Food Engineering Reviews*, *13*, 66–91. https://doi.org/10.1007/s12393-020-09251-y

- Chen, C. Y. S., & Tawan, C. (2020). Botany, diversity, and distribution of black pepper (*Piper nigrum* L.) cultivars in Malaysia. *Borneo Journal of Resource Science and Technology*, 10(1), 10–23. https://doi.org/10.33736/bjrst.1566.2020
- Chithra, G., Mathew, S. M., & Deepthi, C. (2011). Performance evaluation of a power operated decorticator for producing white pepper from black pepper. *Journal of Food Process Engineering*, 34(1), 1–10. https://doi. org/10.1111/j.1745-4530.2008.00333.x
- Chumroenphat, T., Somboonwatthanakul, I., Saensouk, S., & Siriamornpun, S. (2021). Changes in curcuminoids and chemical components of turmeric (*Curcuma longa* L.) under freeze-drying and low-temperature drying methods. *Food Chemistry*, 339, 128121. https:// doi.org/10.1016/j.foodchem.2020.128121
- Codex Alimentarius Commission. (2017). Standard for black, white and green peppers (CXS 326-2017). https://www.fao.org/fao-whocodexalimentarius/sh-proxy/en/?lnk=1&url=htt ps%253A%252F%252Fworkspace.fao.org%25 2Fsites%252Fcodex%252FStandards%252FC XS%2B326-2017%252FCXS\_326e.pdf
- Friso, D. (2023). Mathematical modelling of rotary drum dryers for alfalfa drying process control. *Inventions*, 8(1), 11. https://doi.org/10.3390/ inventions8010011
- Huynh, T. T.-T., Mai, T.-C., Dang, C.-H., Vo, T. T.-T., Nguyen, D.-T., Dang, V.-S., Nguyen, K. D. V., Tran, V.-T., & Nguyen, T.-D. (2020). Influence of extractions on physicochemical characterization and bioactivity of *Piper nigrum* oils: Study on the non-isothermal decomposition kinetic. *Arabian Journal of Chemistry*, *13*(10), 7289–7301. https://doi.org/10.1016/j.arabjc.2020.08.008

- Kang, K.-Y., Hwang, K.-R., Park, J.-Y., Lee, J.-P., Kim, J.-S., & Lee, J.-S. (2018). Critical point drying: An effective drying method for direct measurement of the surface area of a pretreated cellulosic biomass. *Polymers*, 10(6), 676. https:// doi.org/10.3390/polym10060676
- Kaveh, M., & Abbaspour-Gilandeh, Y. (2020). Impacts of hybrid (convective-infrared-rotary drum) drying on the quality attributes of green pea. *Journal of Food Process Engineering*, 43(7), e13424. https://doi.org/10.1111/jfpe.13424
- Kerr, W. L. (2019). Food drying and evaporation processing operations. In M. Kutz (Ed.), *Handbook of farm, dairy, and food machinery engineering* (3<sup>rd</sup> ed., pp. 353–387). Academic Press. https://doi.org/10.1016/B978-0-12-814803-7.00014-2
- Kusumorini, N., Nugroho, A. K., Pramono, S., & Martien, R. (2021). Development of new isolation and quantification method of piperine from white pepper seeds (*Piper nigrum* L.) using a validated HPLC. *Indonesian Journal* of Pharmacy, 32(2), 158–165. https://doi. org/10.22146/ijp.866
- Lamidi, R. O., Jiang, L., Pathare, P. B., Wang, Y. D., & Roskilly, A. P. (2019). Recent advances in sustainable drying of agricultural produce: A review. *Applied Energy*, 233–234, 367–385. https://doi.org/10.1016/j.apenergy.2018.10.044
- Laouni, A., El Orche, A., Elhamdaoui, O., Karrouchi, K., El Karbane, M., & Bouatia, M. (2022). A preliminary study on the potential of FT-IR spectroscopy and chemometrics for tracing the geographical origin of Moroccan virgin olive oils. *Journal of AOAC INTERNATIONAL*, 106(3), 804–812. https://doi.org/10.1093/ jaoacint/qsac136
- Liu, H., Zheng, J., Liu, P., & Zeng, F. (2018). Pulverizing processes affect the chemical quality and thermal property of black, white, and green pepper (*Piper nigrum L.*). Journal of Food

*Science and Technology*, *55*, 2130–2142. https:// doi.org/10.1007/s13197-018-3128-8

- Liu, L., Zuo, Z.-T., Wang, Y.-Z., & Xu, F.-R. (2020). A fast multi-source information fusion strategy based on FTIR spectroscopy for geographical authentication of wild *Gentiana rigescens*. *Microchemical Journal*, 159, 105360. https:// doi.org/10.1016/j.microc.2020.105360
- Malaysian Pepper Board. (2017a). *Manual penanaman lada* [Pepper planting manual]. MPB.
- Malaysian Pepper Board. (2017b). *Sarawak pepper: A hallmark of quality*. MPB.
- Megat, A. A. P. N., Shamsudin, R., Che Man, H., & Ya'acob, M. E. (2020). Effect of soaking process on physical properties of mature pepper berries (*Piper nigrum* L.). *Food Research*, 4(Suppl. 1), 116–123. https://doi.org/10.26656/fr.2017.4(S1). S04
- Mohammed, G. J., Omran, A. M., & Hussein, H. M. (2016). Antibacterial and phytochemical analysis of *Piper nigrum* using gas chromatographymass spectrum and Fourier-transform infrared spectroscopy. *International Journal of Pharmacognosy and Phytochemical Research*, 8(6), 977–996.
- Mühlbauer, W., & Müller, J. (2020). Drying. In Drying atlas: Drying kinetics and quality agricultural products (pp. 9–35). Woodhead Publishing. https://doi.org/10.1016/B978-0-12-818162-1.00003-1
- Nikolić, M., Stojković, D., Glamočlija, J., Ćirić, A., Marković, T., Smiljković, M., & Soković, M. (2015). Could essential oils of green and black pepper be used as food preservatives? *Journal of Food Science and Technology*, *52*, 6565–6573. https://doi.org/10.1007/s13197-015-1792-5
- Olalere, O. A., Abdurahman, H. N., Yunus, R. M., Alara, O. R., Ahmad, M. M., Zaki, Y. H., & Abdlrhman, H. S. M. (2018). Parameter study, antioxidant activities, morphological

and functional characteristics in microwave extraction of medicinal oleoresins from black and white pepper. *Journal of Taibah University for Science*, *12*(6), 730–737. https://doi.org/10. 1080/16583655.2018.1515323

- Olalere, O. A., Abdurahman, N. H., Alara, O. R., & Habeeb, O. A. (2017). Parametric optimization of microwave reflux extraction of spice oleoresin from white pepper (*Piper nigrum* L.). *Journal of Analytical Science and Technology*, 8, 8. https:// doi.org/10.1186/s40543-017-0118-9
- Olalere, O. A., Abdurahman, N. H., Yunus, R. M., Alara, O. R., & Ahmad, M. M. (2019). Mineral element determination and phenolic compounds profiling of oleoresin extracts using an accurate mass LC-MS-QTOF and ICP-MS. *Journal of King Saud University - Science*, 31(4), 859–863. https://doi.org/10.1016/j.jksus.2018.05.018
- Picolotto, A. M., Ariati, A. M., dos Santos Franciscato, L. M. S., Bittencourt, P. R. S., Caramão, E. B., Sakai, O. A., & Moritz, C. M. F. (2020). Chemical and thermoanalytical characterization of the pink pepper (*Schinus terebinthifolius* Raddi) seeds essential oil. *Revista Mundi Engenharia, Tecnologia e Gestão*, 5(7), 1–21. https://doi.org/10.21575/25254782rmetg2020 vol5n71120
- Rezaei, H., & Sokhansanj, S. (2021). A review on determining the residence time of solid particles in rotary drum dryers. *Drying Technology*, 39(11), 1762–1772. https://doi.org/10.1080/073 73937.2021.1912081
- Rigit, A. R. H., Jakhrani, A. Q., Kamboh, S. A., & Kie, P. L. T. (2013). Development of an indirect solar dryer with biomass backup burner for drying pepper berries. *World Applied Sciences Journal*, 22(9), 1241–1251. https://doi.org/10.5829/idosi. wasj.2013.22.09.2724
- Saha, G., Sharangi, A. B., Upadhyay, T. K., Alkeridis, L. A., Alshammari, N., Alabdallah, N. M., & Saeed, M. (2022). Dynamics of drying

turmeric rhizomes (*Curcuma longa* L.) with respect to its moisture, color, texture and quality. *Agronomy*, *12*(6), 1420. https://doi.org/10.3390/ agronomy12061420

- Salehi, B., Zakaria, Z. A., Gyawali, R., Ibrahim, S. A., Rajkovic, J., Shinwari, Z. K., Khan, T., Sharifi-Rad, J., Ozleyen, A., Turkdonmez, E., Valussi, M., Tumer, T. B., Fidalgo, L. M., Martorell, M., & Setzer, W. N. (2019). *Piper* species: A comprehensive review on their phytochemistry, biological activities and applications. *Molecules*, 24(7), 1364. https://doi. org/10.3390/molecules24071364
- Sarifudin, A., Sholichah, E., Setiaboma, W., Ekafitri, R., Afifah, N., Ratnawati, L., Pudiyanto, E. A., & Achyadi, N. S. (2021). Adulterated powdered white pepper products by tapioca flour sold in Indonesian's online market investigated by simple FTIR analytical method. *Journal of Food* and Nutrition Research, 9(6), 297–303. https:// doi.org/10.12691/jfnr-9-6-5
- Simonovska, J., Škerget, M., Knez, Ž., Srbinoska, M., Kavrakovski, Z., Grozdanov, A., & Rafajlovsk, V. (2016). Physicochemical characterization and bioactive compounds of stalk from hot fruits of *Capsicum annuum* L. *Macedonian Journal* of Chemistry and Chemical Engineering, 35(2), 199–208. https://doi.org/10.20450/ mjcce.2016.944
- Singh, S., Kapoor, I. P. S., Singh, G., Schuff, C., De Lampasona, M. P., & Catalan, C. A. N. (2013). Chemistry, antioxidant and antimicrobial

potentials of white pepper (*Piper nigrum* L.) essential oil and oleoresins. *Proceedings of the National Academy of Sciences, India Section B: Biological Sciences, 83*, 357–366. https://doi. org/10.1007/s40011-012-0148-4

- Surendhar, A., Sivasubramanian, V., Vidhyeswari, D., & Deepanraj, B. (2019). Energy and exergy analysis, drying kinetics, modeling and quality parameters of microwave-dried turmeric slices. *Journal of Thermal Analysis and Calorimetry*, 136, 185–197. https://doi.org/10.1007/s10973-018-7791-9
- Tiwari, A., Mahadik, K. R., & Gabhe, S. Y. (2020). Piperine: A comprehensive review of methods of isolation, purification, and biological properties. *Medicine in Drug Discovery*, 7, 100027. https:// doi.org/10.1016/j.medidd.2020.100027
- Trojosky, M. (2019). Rotary drums for efficient drying and cooling. *Drying Technology*, *37*(5), 632–651. https://doi.org/10.1080/07373937.2018.1552597
- Wae-hayee, M., Yeranee, K., Suksuwan, W., Alimalbari, A., Sae-ung, S., & Nuntadusit, C. (2021). Heat transfer enhancement in rotary drum dryer by incorporating jet impingement to accelerate drying rate. *Drying Technology*, 39(10), 1314–1324. https://doi.org/10.1080/07 373937.2020.1742150
- Wang, Y. (2012). Application of Fourier transform infrared microspectroscopy (FTIR) and thermogravimetric analysis (TGA) for quick identification of Chinese herb *Solanum lyratum*. *Plant Omics*, 5(6), 508–513.