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Evaluation of Some Physicochemical Indicators of Black Pepper Purchased from Main Growing Areas in Vietnam

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Abstract

This study aims to evaluate several physicochemical indicators of black pepper samples purchased from three growing regions: Quang Tri, Ba Ria-Vung Tau, and Dak Lak. The results of ANOVA analysis at α equal 0.05 reveals significant differences in moisture content, total ash content, piperine content, and essential oil content among the pepper samples from these three regions. The results obtained by Principal Component Analysis (PCA) identify the characteristic physicochemical indicators for Dak Lak pepper samples as moisture content, piperine content, and essential oil content. In contrast, Quang Tri pepper samples are characterized by moisture, total ash, piperine, and essential oil content. These findings not only demonstrate a correlation between the physicochemical index of black pepper and the factors associated with its growing regions, but also highlight their practical implications for the cultivation and quality control of black pepper across different regions.

Keywords: Black pepper, Quang Tri, Dak Lak, Ba Ria – Vung Tau.

1. Introduction

Black pepper (*Piper nigrum* L.) is a perennial industrial crop with significant economic and export value. It is widely used as a spice globally, renowned for its distinctive hot and spicy flavor. According to statistical data over several years, Vietnam has played a crucial role in the global pepper market and is one of the world's leading exporters. It is the largest supplier of pepper to markets such as the European Union and the United States (USITC, 2023) [1].

In Vietnam, black pepper is primarily cultivated in the Central region (Phu Yen, Quang Tri), the Central Highlands (Gia Lai, Dak Lak, Dak Nong), and the Southern region (Binh Phuoc, Dong Nai, Ba Ria - Vung Tau). However, due to the diversity in geographical conditions and cultivation methods, its quality and physicochemical composition vary across different growing regions. The composition of black pepper includes water, minerals, piperine, essential oil, fats, and several other nutrients. Piperine is the primary compound responsible for the characteristic pungency of pepper and has various health benefits. The distinctive aroma is produced by the essential oil in the pepper, consisting of approximately 40 compounds [2]. Moisture content represents the free water present in pepper, accurately controlling moisture levels helps protect the product from mold and bacterial growth, ensuring that the pepper remains aromatic, intact, and of high quality. Ash is the residue remaining after the combustion of organic matter in the food. Determining the total ash content in food is an analytical method used to assess the nutritional value of various food ingredients. According to TCVN, IPC, ASTA and ISO standards [3-6], the following physicochemical parameters are specified: moisture content should not exceed 13% of the dry weight (12.5% according to TCVN); total ash content should not exceed 7% of the dry weight; essential oil content should be at least 2 ml/100g of dry weight; and piperine content should be a minimum of 4% of the dry weight (3% according to IPC).

This study aims to analyze and evaluate the quality of black pepper samples sourced from three distinct growing regions: Dak Lak, Ba Ria - Vung Tau, and Quang Tri. Indicators considered important and highly influential in research results include moisture, total ash, piperine, and essential oil content.

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The goal is to identify correlations between the physicochemical properties that determine pepper quality, and the factors associated with the growing regions, thereby highlighting the specific physicochemical indicators characteristic of each growing area.

2. Materials and Methods

2.1. Materials

The unprocessed black pepper samples of Vinh Linh were grown and harvested in three provinces: Quang Tri (40 samples), Ba Ria - Vung Tau (35 samples), and Dak Lak (20 samples).

2.2. Methods

2.2.1. Sampling method

The samples sent to the laboratory were representative and did not deteriorate or change during storage and transportation. The raw material samples collected according to TCVN 4889:1989 [7] were stored in zipper bags for preservation (Fig. 1). Before analysis, the samples were cleaned to remove any contaminants and ground to a particle size of approximately 1 mm (Fig. 2).

Fig. 1. The pepper samples were coded and stored in zipper bags for preservation.

Fig. 2. The ground pepper samples are used for physicochemical analysis.

All experiments were conducted from June 2023 to June 2024 at Hanoi University of Science and Technology, Vietnam.

2.2.2. Methods for determining the physicochemical properties of pepper

Moisture content determination (TCVN 7035:2002 [8]): The samples were dried at 105 °C ± 1 °C for 4 hours under atmospheric pressure until it reaches a constant weight.

Determination of total ash content (TCVN 7038:2002 [9]): The samples were heated in a furnace until converted to ash. Subsequently, the crucible containing the samples was placed in a muffle furnace at 550 °C for 4 hours ± 0.1 hours until a constant weight is reached.

Piperine content determination (TCVN 9683:2013 [10]): Piperine was extracted using ethanol and quantified by spectroscopy at a wavelength of 343 nm.

Essential oil content determination (TCVN 7039:2013 [11]): Essential oil was obtained by using hydrodistillation over a period of approximately 6 hours.

2.2.3. Data processing method

All data were processed and analyzed using Excel software, with one-way ANOVA and Tukey's test employed to compare the physicochemical parameters across three cultivation regions at a significance level of α equal 5%. Subsequently, principal component analysis (PCA) was applied using XLSTAT software to determine the correlation between physicochemical properties and cultivation regions.

One-way ANOVA was used to assess the effect of a causal factor (qualitative data) on an outcome factor (quantitative data) under investigation. Specifically, it examined the influence of the regional factor on the physicochemical properties of pepper.

PCA was used to reduce a set of dependent variables (attributes) to a smaller set of underlying variables (factors) based on a model of correlations among the original variables. Data reduction was achieved by analyzing the data, selecting independent and dependent variables, and generating a two-dimensional plot of the samples.

3. Results and Discussion

3.1. Quality Parameters of Black Pepper Samples from Different Regions

All moisture and total ash content data were measured in triplicate, while piperine and essential oil content were measured in duplicate for each sample. The results in Table 1 show the mean values of the replicates plus/minus standard deviation.

Table 1. Physicochemical parameters of black pepper in Quang Tri, Dak Lak and Ba Ria - Vung Tau

Regional pepper sample	Average moisture (%)	Average total ash (%)	Average piperine (%)	Average essential oil (ml/100g)
Quang Tri	12.43±1.05[a]	4.17±0.08[a]	4.79±0.24[a]	3.18±0.23[a]
Ba Ria – Vung Tau	10.89±1.32[b]	3.80±0.19[b]	4.27±0.52[b]	2.86±0.15[b]
Dak Lak	13.58±1.07[c]	3.95±0.15[bc]	5.22±0.40[c]	3.47±0.29[ac]

Note: Within the same column, values sharing the same letter are not significantly different at the 5% level (*) according to Tukey's test.

3.1.1. Evaluation of the compliance level of pepper samples from 3 cultivation regions

a) Quang Tri

The pepper samples have an average moisture of 12.43% (< 12.5%), an average total ash of 4.17% (< 7%), an average piperine of 4.79% (> 4%), and an average essential oil content of 3.18 ml (> 2 ml/100 g). Therefore, the pepper from Quang Tri meets the quality standards according to TCVN, IPC, and ASTA [3-6].

b) Ba Ria - Vung Tau

The pepper samples have an average moisture of 10.89% (< 12.5%), an average total ash of 3.8% (< 7%), an average piperine of 4.27% (> 4%), and an average essential oil content of 2.86 ml (> 2 ml/100 g). Therefore, the pepper from Ba Ria – Vung Tau meets the quality standards according to TCVN, IPC, and ASTA [3-6].

c) Dak Lak

The pepper samples from Dak Lak have an average moisture content of 13.58% (> 13%), which does not meet the moisture quality standards set by TCVN, IPC, and ASTA. However, the average total ash is 3.95% (< 7%), the average piperine is 5.22% (> 4%), and the average essential oil content is 3.47 ml (> 2 ml/100 g). Therefore, the pepper from Dak Lak meets the quality standards for total ash, piperine, and essential oil according to TCVN, IPC, and ASTA [3-6].

3.1.2. Comparison of physicochemical properties among pepper growing regions

a) Moisture

The analysis (Table 1) reveals that the average moisture content of the pepper samples studied ranges from 10.89% (for samples from Ba Ria - Vung Tau) to 13.58% (for samples from Dak Lak). It is observed that the moisture of pepper samples from Dak Lak tends to be higher compared to those from Quang Tri and Ba Ria - Vung Tau. The ANOVA analysis results indicate a P-value is 5.9×10^{-14}, suggesting a significant difference in moisture among the pepper samples from the three regions. Detailed ANOVA analysis shows the following statistical differences: The moisture content of pepper samples from Quang Tri is significantly different from those from Dak Lak and Ba Ria - Vung Tau, with a significance level of α equal 0.05. In the other hand, the moisture content of pepper samples from Ba Ria - Vung Tau is statistically different from that of samples from Dak Lak, with a significance level of α equal 0.05.

b) Total ash

According to the results in Table 1, the average total ash content of the pepper samples ranges from 3.80% (for samples from Ba Ria - Vung Tau) to 4.17% (for samples from Quang Tri). ANOVA analysis reveals P equal 0.00013, which is less than α equal 0.05, indicating a statistically significant difference in total ash among the regions. Detailed ANOVA analysis identifies the following statistical differences between the regions: The total ash content of pepper samples from Quang Tri is significantly different from those from Dak Lak and Ba Ria - Vung Tau,with a significance level α equal 0.05. On the other hand, the total ash content of pepper samples from Ba Ria - Vung Tau is not statistically different from that of samples from Dak Lak, with a significance level of α equal 0.05.

c) Piperine

The average piperine content in the analyzed samples ranges from 4.27% (for samples from Ba Ria - Vung Tau) to 5.22% (for samples from Dak Lak). ANOVA analysis reveals a *P*-value of 9.21×10^{-7}, which is less than α equal 0.05, indicating a statistically significant difference in piperine content among the pepper samples from the three regions. A detailed ANOVA analysis identifies the following statistical differences between the regions: The piperine content of pepper samples from Quang Tri is statistically different from those from Dak Lak and Ba Ria-Vung Tau, with a significance level of α equal 0.05. The piperine content of pepper samples from Ba Ria-Vung Tau is statistically different from that of samples from Dak Lak, with a significance level of α equal 0.05.

d) Essential oil

The average essential oil content in the analyzed samples ranges from 2.86 ml/100 g (for samples from Ba Ria and Vung Tau) to 3.47 ml/100 g (for samples from Dak Lak). ANOVA analysis shows *P*-value of 2.86×10^{-5}, which is less than α equal 0.05, indicating a statistically significant difference in essential oil content among the pepper samples. Detailed ANOVA analysis reveals the following statistical differences between the regions: The essential oil content of pepper samples from Ba Ria-Vung Tau is statistically different from those from Dak Lak and Quang Tri, with a significance level of α equal 0.05. The essential oil content of pepper samples from Quang Tri and Dak Lak does not differ statistically, with a significance level of α equal 0.05.

3.2. Evaluation of the Correlation between Physicochemical Properties of Black Peppers and Pepper Samples from the Studied Regions

PCA was performed on the data obtained from the physicochemical properties of black pepper, including moisture, total ash, piperine, and essential oil content. To evaluate the concentration levels of black pepper samples from each region, PCA was performed on a dataset comprising 95 samples. The results, as depicted in Fig. 2, demonstrate the clustering of pepper samples from Ba Ria – Vung Tau, Quang Tri, and Dak Lak.

PCA analysis was conducted using the average values of the physicochemical indicators (moisture content, total ash content, piperine content, and essential oil) from the three regions (Table 1) to examine the correlation of these physicochemical indicators and each growing region. The analysis results are shown in Fig. 3. The "scree plot" illustrates the descending order of the eigenvalues and the percentage of variance explained. In principal component analysis, the "scree plot" helps determine the relative importance of the components. In this case, Component 1 (F1) has an eigenvalue greater than one and explains 81.745% of the cumulative variance (Fig. 3).

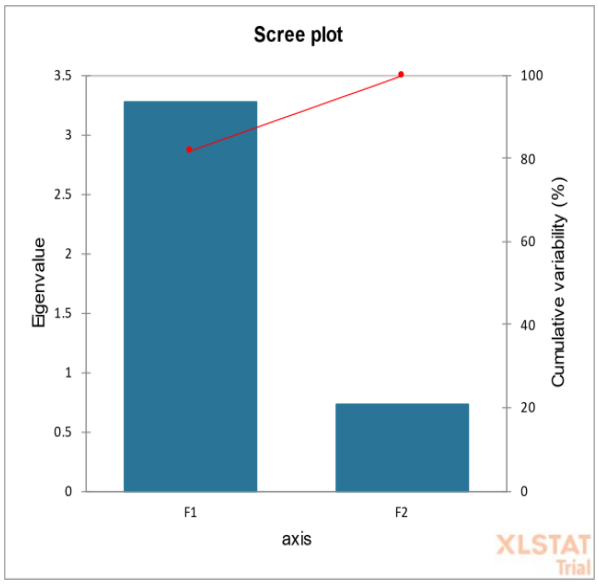

Fig. 3. Eigenvalue and cumulative percentage variance (%) graph of components represented by Screeplot

The factor loading coefficients represent the contribution of each physicochemical parameter to the components (Table 2). Higher factor loadings indicate a stronger correlation. In conjunction with the principal component plane plot between F1 and F2 - the correlation circle of black pepper characteristics (Fig. 4), the parameters can be divided into two distinct regions: Region 1 includes the three parameters - moisture, essential oil, and piperine content; Region 2 comprises the total ash content. The parameters of essential oil, piperine, and moisture content are located close to the axis of the first principal component (F1). Meanwhile, the moisture (0.991) has the most significant influence on the first principal component (F1), followed by piperine (0.987) and essential oil content (0.982), which also have values close to moisture and significantly impact the first principal component (F1). The total ash (0.807) is near the axis of the second principal component (F2) and has the most significant influence on this component.

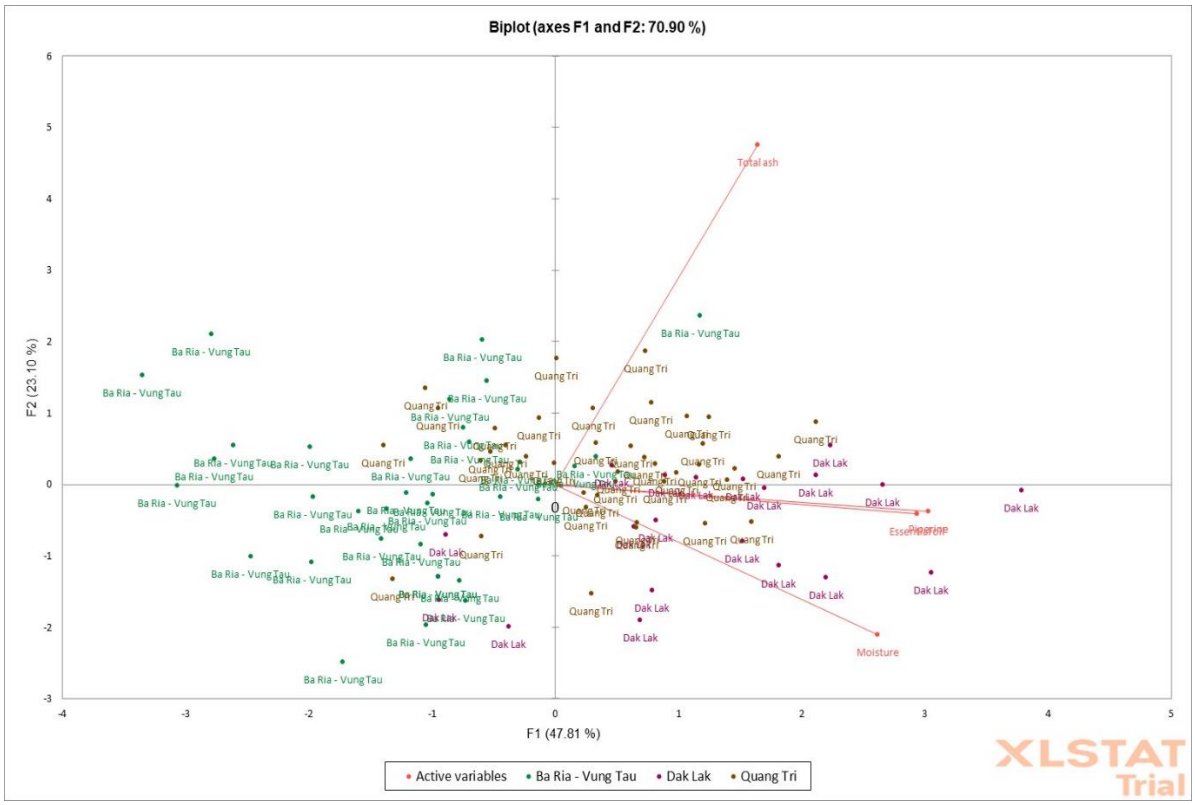

Fig. 4. The distribution of pepper samples from different regions on the PCA composed of F1 and F2

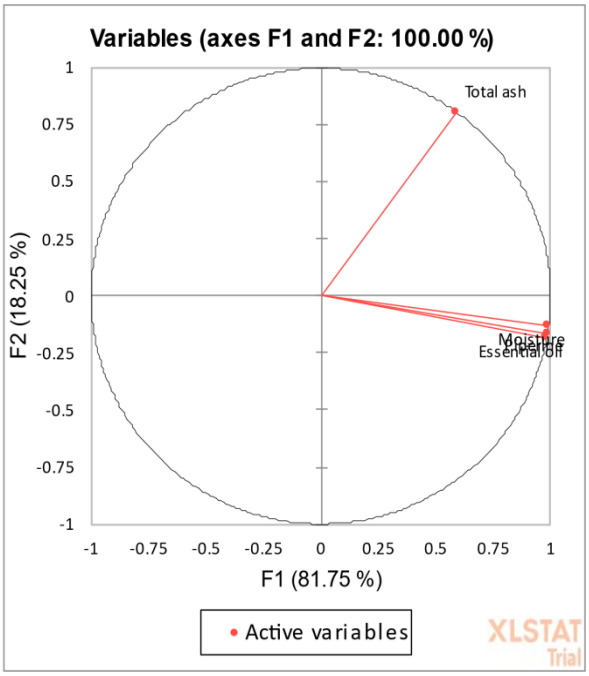

Fig. 5. Principal map plot between principal components F1 and F2 – Correlation circle of black pepper properties

Table 2. Factor loadings

	F1	F2
Moisture	0.991	-0.133
Total ash	0.591	0.807
Piperine	0.987	-0.162
Essential oil	0.982	-0.188

Table 2 and Fig. 4 indicate a strong correlation between essential oil and piperine with F1 & F2 axes.

The PCA results for the average data of black pepper from different regions and their physicochemical parameters are presented on the first principal component plane (explaining 81.75%) and the second principal component plane (18.25%) (Fig. 5). Based on the distribution of the black pepper samples and their physicochemical parameters (Fig. 6), it is observed that the pepper from Quang Tri is characterized by all four parameters: total ash, moisture, piperine, and essential oil content. In contrast, the pepper from Dak Lak is characterized by three parameters: essential oil, moisture, and piperine content. The analysis of black pepper's physicochemical parameters indicates that regional factors and geographical locations can lead to variations in moisture, total ash, and the levels of compounds such as piperine and essential oil. These differences contribute to the variability in the quality of black pepper.

4. Conclusion

The study reveals the impact of regional factors on various physicochemical properties, including moisture, total ash, piperine, and essential oil content. Samples from Dak Lak are characterised by their piperine, essential oil, and moisture. In contrast, samples from Quang Tri are distinguished by moisture, total ash, piperine, and essential oil content. However, no specific characteristics could be identified for samples from Ba Ria – Vung Tau. The data on differences between pepper samples from Quang Tri, Ba Ria – Vung Tau, and Dak Lak can contribute to creating a database on the origins of pepper in Vietnam. This database would support food safety monitoring and rapid traceability of pepper origins, thereby contributing to the sustainable development of the Vietnamese pepper industry and helping meet the increasingly stringent requirements of global markets.

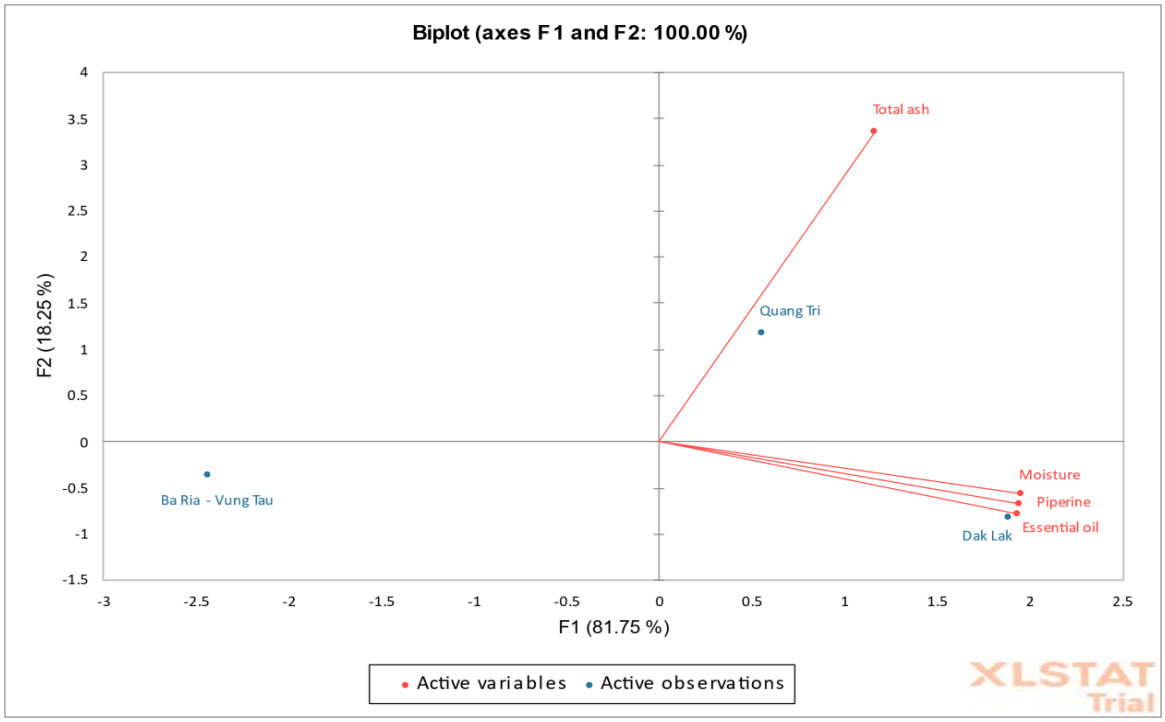

Fig. 6. The distribution of black pepper areas and physicochemical indicators on biplotmap of F1 and F2

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