Revista de Agricultura Neotropical

PARACAMPO, N. E. N. P.; ABREU, L. F.; LEMOS, O. F.; BOTH, J. P. C. L. Quality of black pepper produced in northeastern Pará. **Revista de Agricultura Neotropical**, Cassilândia-MS, v. 9, n. 3, e7020, jul./sep. 2022. ISSN 2358-6303. DOI: https://doi.org/10.32404/rean.v9i3.7020.

Quality of black pepper produced in northeastern Pará

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Received: 08/06/2022; Accepted: 26/08/2022

ABSTRACT

Black pepper (*Piper nigrum* L.) is the most widely used spice in the world. This crop has great economic importance in Brazil, especially in the North and Southeast regions. However, the variability in quality between cultivars is high. The objective of this study was to characterize black pepper from northeastern Pará available on the market and obtain an overview of its quality parameters. A total of 23 samples from black pepper cultivars/clones were evaluated for impurities and foreign matter, empty grains, density, moisture and ether extract, as established by Brazilian legislation. Additionally, the contents of total ash, protein, crude fiber and piperine were determined. Among the samples, one was outside the standard for density. The moisture contents were below the maximum limit required by the legislation, demonstrating that the drying and storage of the grains were adequate. Regarding the ether extract, 52% of the samples did not meet the commercialization standards. Concerning piperine, the Equador and Panny clones had levels greater than 5%. It was found that the nutritional composition of black pepper is influenced by the technological level of the production system. In summary, black pepper produced in northeastern Pará has variable quality.

Keywords: *Piper nigrum* L., Physical characterization, Chemical characterization, Piperine.

Qualidade da pimenta-do-reino produzida no nordeste do Pará

RESUMO

A pimenta-do-reino (*Piper nigrum* L.) é a especiaria mais utilizada no mundo. No Brasil, essa cultura tem grande importância econômica, em especial às regiões Norte e Sudeste. Entretanto, a variabilidade na qualidade entre as cultivares é alta. O objetivo deste trabalho foi caracterizar a pimenta preta do nordeste do Pará disponibilizada no mercado, obtendo-se um panorama quanto aos parâmetros de qualidade. Foram avaliadas 23 amostras de pimenta preta de cultivares/clones quanto às impurezas e matérias estranhas, grãos chochos, densidade, umidade e extrato etéreo, conforme estabelecido pela a legislação nacional. Também, foram determinados os teores de cinzas totais, proteínas, fibra bruta e piperina. Dentre as amostras, uma estava fora do padrão para densidade. Os teores de umidade encontravam-se abaixo do limite máximo exigido pela legislação, demonstrando que a secagem e o armazenamento dos grãos foram adequados. Em relação ao extrato etéreo, 52% das amostras não atenderam aos padrões de comercialização. Quanto à piperina, os clones Equador e Panny destacaram-se com teores superiores a 5%. Verificou-se que a composição nutricional da pimenta preta sofre influência do nível tecnológico do sistema de produção. Portanto, a pimenta preta produzida no nordeste do Pará tem qualidade variável.

Palavras-chave: *Piper nigrum* L., Caracterização física, Caracterização química, Piperina.

1. Introduction

Black pepper (*Piper nigrum* L.) is a member of Piperaceae of Asian origin that is grown in the state of Pará, which has climate, humidity and soil conditions favorable for the establishment of commercial cultivation, especially in northeastern Pará; for a long time, this state was the largest national producer and exporter of black pepper. Currently, Pará is surpassed by the State of Espírito Santo in production (Rodrigues et al., 2019; Barata et al., 2021).

Black pepper is the most important and most widely used spice in the world, representing 34% of the total market (Mayr et al., 2021). According to the law, black pepper consists of grains including their peel that are wrinkled due to natural or artificial dehydration and are black in color (MAPA, 2006). Today, the global black pepper market is estimated to have a compound annual growth rate (CAGR) of approximately 5.37% for the period from 2022 to 2027. This market is continuously expanding and is strongly driven by the pharmacological properties of this species (Mayr et al., 2021).

There are more than 100 varieties of black pepper known worldwide, although few are used commercially. The variability in yield and quality between cultivars is high (Ravindran and Kallupurackal, 2012). For decades, Embrapa Eastern Amazon has been conducting research on the black pepper cultivars adopted in the main producing areas in the state of Pará; this research covers the entire production system, with remarkable attention to high productivity and biotic resistance (Lemos et al., 2014; Rodrigues et al., 2017).

Plants contain numerous organic compounds; these compounds can be divided into primary metabolites, which are produced in metabolic processes that perform essential plant functions, such as photosynthesis, and secondary metabolites, which are produced from primary metabolism products and are not considered essential for plant development, but play an important role in defense. The production of these metabolites, especially secondary metabolites, is influenced by genetics, as well as changes in ecosystem balance, such as seasonality and attack by pests and diseases. Thus, biochemical pathways that alter plant metabolism are activated to adapt to such changes (Luz et al., 2017; Erb and Kliebenstein, 2020).

The Technical Standard for integrated black pepper production mandates the use of cultivars or clones adapted to the edaphoclimatic characteristics of a given property or region (MAPA, 2021). In view of this requirement and given the importance of pepper cultivation to the Brazilian economy, especially in the North Region, as the source of a product with high global consumption, black pepper breeding programs focus on the production of crops with good quality parameters. These parameters are based on physicochemical characteristics and the content of piperine, which is the major bioactive compound of black pepper and has been identified as one of the main alkaloids responsible for pungency (Ravindran and Kallupurackal, 2012; Iqbal et al., 2016). Therefore, the objective of this study was to characterize the black pepper cultivated in northeastern Pará available in the market and obtain an overview of its quality parameters.

2. Material and Methods

A total of 23 black pepper samples (approximately 1 kg each) were collected from farms and experimental areas of Embrapa Eastern Amazon in municipalities in northeastern Pará: Baião (2°47'12.6" S 49°39'19.6" W), Castanhal (1°10'57" S 47°53'44" W) and Tomé-Açu (2°27'26.6" S 48°16'49.4" W). Sample collection occurred in 2018 during the following fruit maturation cycles: early (from July to September); intermediate (from August to October); and late (from September to November) (Lemos et al., 2014). The climate is of the “Am” or “Af” type, according to the Köppen classification, with an average annual rainfall ranging from 2,250 to 3,000 mm (Andrade et al., 2017). The cultivated soil has a medium, sandy-clayey, deep and flat texture. The spacing between plants varies from 2.0 m x 2.0 m (dead supports — wooden posts) to 2.25 m x 2.25 m x 4.0 m (live supports — gliricidia). Each black pepper sample analyzed in the laboratory was from a standardized lot and was representative of the plant population that was collected in the field from the following clones: Alencar, Apra, Bento, Bragantina, Singapore, Cleo, Clonada, Equador, Guajarina, Iaçará, Kottanadan, Kuthiravally, Panny, Panama, and Uthirankotta. These clones are cultivars (Apra, Bragantina, Cingapura, Guajarina, Iaçará, Kottanadan, Kuthiravally and Uthirankotta) or genetic materials in use by producers (Alencar, Cleo, Clonada, Equador, Guajarina-Bento, Panny and Panama) that have not yet been recorded and released as cultivars. Fruits in the mature stage were collected from plants from the third year of cultivation, threshed and dried on tarpaulins under full sun for two or three days, and taken to Embrapa Eastern Amazon for laboratory analysis.

The Technical Regulation of the Identity and Quality of Black Pepper recommends starting the classification process by quartering a 1 kg sample. Thus, the samples of black pepper clones were quartered for determination of impurities and foreign matter, empty grains and density in duplicate, according to Normative Instruction 10/2006 of the Ministry of Agriculture, Livestock and Food Supply (MAPA, 2006).

The determined nutritional composition parameters included the moisture, ash, lipids (or ether extract), protein and crude fiber contents. The black pepper samples were analyzed in triplicate for moisture (gravimetric method with desiccation at 105 °C) and ether extract (direct Soxhlet extraction with ethyl ether) according to the procedures established in Normative Instruction 10/2006 of the Ministry of Agriculture, Livestock and Food Supply (MAPA, 2006). The determination of ash (gravimetric method, from the residue of the moisture determination step), protein (Kjeldahl method, where the factor 6.25 was used to convert nitrogen into protein) and crude fiber (gravimetric method, from the residue of the determination of ether extract) was performed in triplicate, according to official methods described in the Official Methods of Analysis (AOAC, 2011).

The quantification of piperine was performed in triplicate by ultraviolet-visible spectrophotometry according to method 987.07 of the Official Methods of Analysis (AOAC, 2011). The results for impurities and foreign matter, empty grains, density, moisture and ether extract were subjected to descriptive statistical analysis to evaluate the tolerance limits of the quality factors of black pepper destined for internal marketing, import and export, according to Normative Instruction 10/2006 (MAPA, 2006).

The results for nutritional composition (ash, protein and crude fiber) and piperine content were initially subjected to the Grubbs test to evaluate the presence of extreme values in the dataset, followed by the Kolmogorov–Smirnov test to examine the normality of the values themselves. To verify the differences between the datasets, one-way analysis of variance (ANOVA) was used, and the Levene test was applied to determine the homogeneity of variances. Then, the Scott–Knott test was used to identify differences between the analyzed data. The descriptive level (p-value) adopted in all tests was $p \leq 0.05$. The calculations were performed using the software Excel® 2010 (Microsoft, WA, USA), Minitab® 19 (Minitab Statistical Software, PA, USA) and Sisvar version 5.6.

In addition, the ether extract, ash, protein, crude fiber and piperine data were organized in an Excel® spreadsheet in matrix format (I, J) with I = 23 samples and J = 5 study variables, and the data were imported into MATLAB® R2016a (MathWorks®). These data were self-scaled and subjected to unsupervised classification by principal component analysis (PCA) to identify clusters and trends using the software PLS_Toolbox 7.3 (Eigenvector).

3. Results and Discussion

According to the Technical Regulation of Identity and Quality, for the internal marketing and commercialization of black pepper, the percentage of occurrence of quality factors should be within the tolerance limits established by three standards: the American Spice Trade Association (ASTA), Brazil 1 (B1) and Brazil 2 (B2). Samples that do not meet the limit for at least one quality factor included in the regulation, including the dispersion of the dataset, are considered “Out of Type” (MAPA, 2006).

The physicochemical characteristics of the black pepper samples are presented in the form of descriptive statistics as the mean ± standard deviation. The percentage of impurities and foreign matter in the evaluated samples ranged from 0.13% to 1.89%; empty grains ranged from 0.03% to 10.91%; and density ranged from 497.22 g/L to 680.60 g/L (Table 1). The tolerance limits defined by the regulation for sample classification according to type are as follows: impurities and foreign matter: maximum of 1% for ASTA, 2% for B1 and 5% for B2; empty grains: maximum of 2% for ASTA, 5% for B1 and 25% for B2; and density: minimum limit of 560 g/L for ASTA, 540 g/L for B1 and 500 g/L for B2 (MAPA, 2006). Only sample 4 (Bragantina) was classified as “Out of Type” because the density (497.22 g/L) did not meet the minimum tolerance limits.

Regarding ether extract, at least 52% of the studied samples were classified as “Out of Type” since the tolerance limit for black pepper according to the three standards (ASTA, B1 and B2) is a minimum content of 6.75% (MAPA, 2006). The variation in the content of ether extract was 4.73% to 9.89% (dry basis, db). Ravindran and Kallupurackal (2012) described lower values for black pepper, oscillating between 0.3% and 4.2% db, while Sruthi et al. (2013) evaluated samples of the Panniyur-1 variety collected in 11 locations in India and found results ranging from 6.16% to 10.34%.

The moisture content ranged from 10.03% to 12.79%, which was within the maximum allowed moisture limit of 14% of the Ministry of Agriculture, Livestock and Food Supply (MAPA, 2006). Therefore, it was inferred that the drying and storage process of these grains was adequate and met the commercialization standards. Machado et al. (2021) obtained values between 9.85% and 14.04% when evaluating 16 samples from the rural area of Espírito Santo.

Thus, based on the results for impurities and foreign matter, empty grains, density, moisture and ether extract - factors that are included in the Technical Regulation - only the Alencar clone met the ASTA international standard. The other data, i.e., nutritional composition and piperine (db), were subjected to a hypothesis test to determine the difference in means. A normal distribution of the data and equivalence of variances were obtained by the Kolmogorov–Smirnov and Levene tests, respectively.

Table 1. Physicochemical characterization of black pepper (*Piper nigrum* L.) cultivars/clones from producers in northeastern Pará cultivated on dead (posts) or live (gliricidia) supports at different maturation cycles of the fruit (early, intermediate or late) after three years of cultivation.

	Cultivar	Producer	Town Municipality	Support	Maturation cycle	Impurities and foreign matter (%)	Empty grains (%)	Density (g/L)	Humidity (%)	Ether extract[1] (%)	Ash[1,2] (%)	Protein [1,2] (%)	Crude fiber[1,2] (%)	Piperine [1,2] (%)
1	Alencar	P1	Tomé-Açu	Dead	Intermediate	0.44±0.11	0.74±0.34	597.36±1.53	12.17±0.12	9.64±0.49	5.13±0.07b	11.98±0.11c	21.11±0.27e	6.27±0.15d
2		P1	Tomé-Açu	Alive	Intermediate	0.37±0.12	0.71±0.12	610.04±0.45	12.28±0.08	9.10±1.78	5.33±0.12a	12.94±0.12a	24.16±0.38b	6.64±0.14c
3	Apra	P2	Baião	Dead	Intermediate	0.48±0.00	4.00±0.01	513.86±0.20	10.03±0.17	7.38±0.28	4.57±0.13d	13.09±0.32a	20.90±0.07e	5.19±0.03g
4	Bragantin	P2	Baião	Dead	Early	0.27±0.04	3.78±0.35	497.22±0.82	10.90±0.16	9.50±0.52	4.85±0.05c	12.07±0.14c	22.29±0.17d	6.20±0.09d
5		P3	Baião	Dead	Early	0.36±0.09	3.29±0.31	530.30±0.48	11.87±0.04	9.20±0.13	4.15±0.12e	9.83±0.25f	23.49±0.30c	5.56±0.05e
6		P4	Castanhal	Dead	Early	1.06±0.01	0.13±0.10	612.82±0.65	10.44±0.13	4.81±0.29	4.44±0.16d	10.19±0.08e	20.54±0.21e	4.46±0.02j
7	Cingapur	P2	Baião	Dead	Early	0.29±0.03	2.32±0.27	547.94±0.54	11.90±0.09	6.81±0.29	4.28±0.08e	13.05±0.21a	19.08±0.45f	5.37±0.08f
8		P3	Baião	Dead	Early	0.66±0.58	4.10±1.09	559.28±0.57	12.24±0.12	6.74±1.86	4.28±0.16e	11.19±0.29d	22.05±0.45d	5.34±0.07f
9		P5	Castanhal	Dead	Early	1.15±0.32	1.72±0.58	608.60±1.41	10.06±0.22	5.08±0.26	4.19±0.07e	9.02±0.21g	20.53±0.63e	4.98±0.03h
10	Cleo	P6	Castanhal	Dead	Early	0.28±0.23	0.16±0.09	614.10±0.57	11.35±0.37	5.49±0.72	4.29±0.07e	12.40±0.27b	16.65±0.1h	4.20±0.07l
11	Clonada	P2	Baião	Dead	Early	0.13±0.04	0.76±0.11	548.94±0.31	10.98±0.02	6.03±0.22	4.35±0.03e	12.51±0.19b	21.14±0.25e	5.40±0.08f
12		P3	Baião	Dead	Early	0.30±0.06	2.45±0.04	556.50±0.59	12.37±0.09	7.41±0.48	4.48±0.14d	11.30±0.64d	26.09±0.66a	5.11±0.01g
13		P3	Baião	Alive	Early	0.20±0.16	1.08±0.00	624.28±0.23	12.03±0.15	6.93±0.36	3.65±0.15f	12.06±0.03c	19.58±0.33f	4.72±0.08i
14	Equador	P1	Tomé-Açu	Alive	Intermediate	0.23±0.04	2.60±1.06	591.26±0.82	12.27±0.09	9.89±1.01	4.53±0.16d	12.58±0.02b	19.55±0.25f	7.18±0.03a
15	Guajarina	P2	Baião	Dead	Intermediate	0.29±0.02	4.46±0.47	519.36±1.07	11.98±0.17	8.73±0.38	4.61±0.02d	12.47±0.32b	22.87±0.35b	6.33±0.08d
16		P3	Baião	Dead	Intermediate	0.41±0.06	3.05±1.31	548.32±0.11	12.79±0.11	7.43±0.35	5.29±0.03a	11.73±0.04c	24.56±0.18b	5.39±0.08f
17	Guajarina	P7	Castanhal	Dead	Intermediate	1.03±0.54	0.44±0.13	584.95±0.78	10.45±0.06	6.94±0.28	4.47±0.16d	10.34±0.25e	20.63±0.12e	4.31±0.02k
18	Iaçará	P4	Castanhal	Dead	Early	1.71±0.58	0.10±0.01	680.60±0.57	10.54±0.05	4.73±0.18	4.74±0.17c	9.57±0.12f	18.78±0.23f	4.50±0.06j
19	Kottanad	P7	Castanhal	Dead	Late	1.89±0.70	0.03±0.01	612.10±0.57	10.98±0.26	6.30±0.92	5.10±0.15b	8.63±0.05h	18.03±0.50g	4.12±0.04l
20	Kuthirava	P3	Baião	Dead	Intermediate	0.57±0.20	6.86±0.82	511.96±0.17	12.62±0.09	8.25±0.06	4.76±0.04c	12.86±0.09a	21.73±0.74d	6.40±0.24d
21	Panakota	P4	Castanhal	Dead	Intermediate	0.69±0.21	0.51±0.08	638.95±0.35	10.43±0.14	6.12±0.14	4.72±0.08c	9.56±0.20f	19.05±0.51f	3.71±0.02m
22	Panny	P2	Baião	Dead	Intermediate	0.13±0.03	1.16±0.42	551.48±0.96	10.89±0.18	8.65±0.26	4.66±0.01c	11.22±0.11d	20.58±0.25e	6.95±0.02b
23	Uthirank	P3	Baião	Dead	Late	0.89±0.21	10.91±1.40	500.66±0.45	12.18±0.41	9.31±0.09	4.31±0.14e	12.68±0.09b	23.19±0.35c	6.54±0.14c

[1] Results expressed on a dry basis. [2] Means followed by the same letter in the columns for each variable do not differ statistically by the Scott–Knott test at 5% probability.

Therefore, the Scott–Knott test was adequate to evaluate the differences between the data at 5% probability. In addition, there were no extreme values according to the Grubbs test, which showed that the data exhibit random variability. The total ash and fixed mineral residue ranged from 3.65% to 5.33% db, within the range of values reported in the literature, and from 2.75 to 6.30% db (Ravindran and Kallupurackal, 2012; Abukawsar et al., 2018; Machado et al., 2021), with values between 3.57% and 12.49% (wet basis, wb) (Al-Jasass and Al-Jasser, 2012; Hossain et al., 2014). These results verified that the evaluated samples are sources of minerals. The protein contents ranged from 8.63% to 13.09% bs and were within the range found by Sruthi et al. (2013) and Abukawsar et al. (2018) for Indian samples, which ranged from 3.27% to 16.86% (db). However, the obtained values are below the content of 25.45% wb found by Al-Jasass and Al-Jasser (2012). The levels for the samples from northeastern Pará are close to the nutritional composition value for ASTA black pepper (10.0%), revealing the nutritional quality of the evaluated pepper samples.

The crude fiber contents varied between 16.65% and 26.09% db, among which sample 12 (Clonada) with the highest concentration. Values between 8.7% and 23.60% for black pepper have been reported in other studies (Al-Jasass and Al-Jasser, 2012; Ravindran and Kallupurackal, 2012; Sruthi et al., 2013; Abukawsar et al., 2018). Therefore, the current samples accumulated more fiber during cultivation, which indicates that they may have a positive effect on the human diet. In general, the nutritional compositions of the black pepper samples are similar to those found in other studies, especially the high fiber content. The piperine content is an indicator the total pungency of black pepper, since piperine constitutes approximately 98% of the total alkaloids (Gorgani et al., 2017). According to Krishnamurthy et al. (2010), the ideal black pepper variety should contain at least 5% piperine.

The piperine contents ranged from 3.71% to 7.18% db. In particular, sample 21 (Panayana) had the lowest concentration, and samples 14 (Equador) and 22 (Panny) were the most promising and spicy samples, with the highest piperine content, higher than the ideal minimum defined for an ideal variety (5%). Bermawie et al. (2019) compiled results from previous studies on the piperine content of Indian black pepper cultivars and found values ranging from 1.6% to 12%, which also included values higher than that expected for an ideal variety (5%). The observed differences may be attributed to the genotype (Zacharias and Parthasarathy, 2008) or to environmental factors (Ahmad et al., 2020).

The PCA (Figure 1) showed that the first three components represented 88.02% of the total data variance (PC1 53.80%, PC2 20.45% and PC3 13.77%). In the 3D score plot of PCA, despite the small sample size, clusters were formed in PC1: one group on the left primarily contained samples from Castanhal, and other clusters on the right contained the other samples. In addition, the samples from Producer 2 in Baião tended to be located in the negative PC3 region, while those from Producer 3 were grouped in the positive PC3 region. The samples from Tomé-Açu were also in the negative PC3 region. Concerning the use of dead (posts) or live (gliricidia) supports, no trend could be observed based on the small sample size.

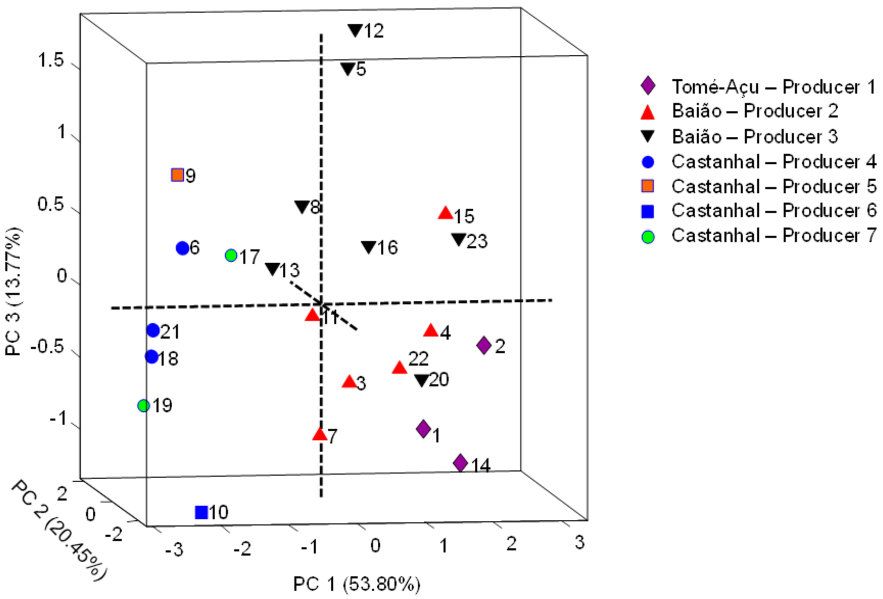

Figure 1. Score plot of principal component analysis (PCA): displays the distribution and grouping of the physicochemical characterization data of 23 samples of black pepper (Piper nigrum L.) cultivars/clones in the scatter 3D plot (PC1 x PC2 x PC3) containing 88.02% of the total data variance.

The loading plot for PC1 is demonstrated in Figure 2 and the most important variables are those with the highest loadings, i.e., ash, ether extract and piperine. The piperine content is related to the ether extract, since piperine is contained in this liposoluble fraction. Thus, samples located on the left of the score graph tend to have a higher ash content and lower ether extract and piperine contents. This is the case for the samples from Castanhal.

These results suggest possible effects of the agricultural practices adopted by producers and the technological level of the production system on the chemical composition of black pepper. In the loading plot for PC3 (Figure 3), the variables with the highest positive and negative loadings are crude fiber, ash and protein. Crude fiber is the most relevant variable for the discrimination of samples from Producer 3 in Baião because these samples have the highest fiber contents.

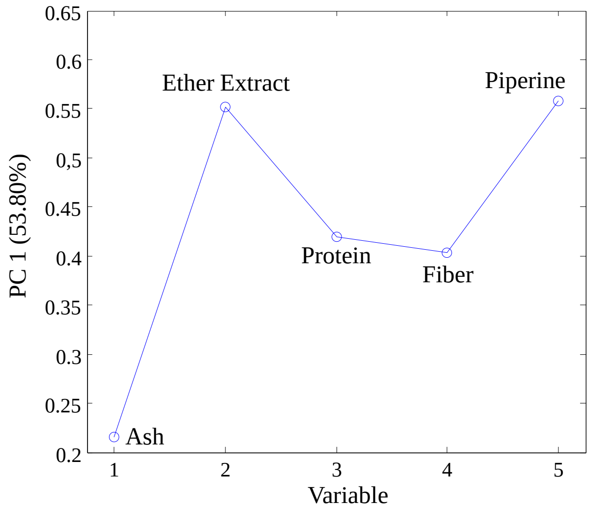

Figure 2. Loading plot of principal component analysis (PCA): shows the variables responsible for the separation of the groups in PC1.

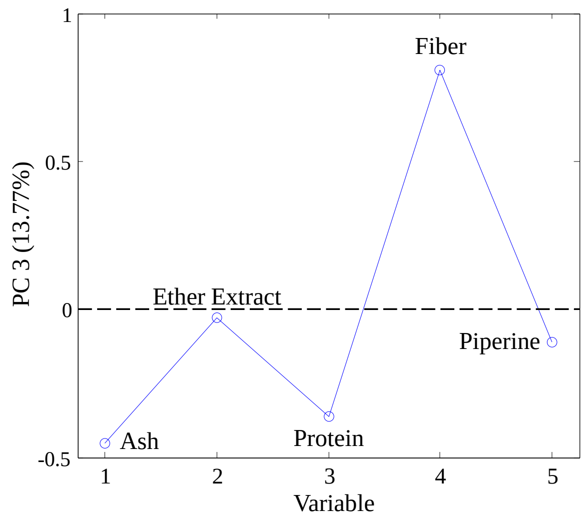

Figure 3. Loading plot of principal component analysis (PCA): displays the variables responsible for the separation of the groups in PC3.

In addition, the score plot shows that the clones Bragantina (samples 4 and 5), Singapore (samples 7 and 8) and Clonada (samples 11 and 12) are positioned on opposite sides of PC3, indicating possible differences between crops of the same genotype, with greater fiber synthesis in the plants cultivated by Producer 3 in Baião.

4. Conclusions

Black pepper produced in northeastern Pará has variable quality because more than half of the evaluated samples had values outside the standards for identity and quality established by Brazilian legislation. However, the samples of the Alencar clone, an unregistered genetic material in use by producers, met the international standard in all parameters analyzed. Regarding piperine, the Equador and Panny clones notably had levels greater than 5%, which is recommended for an ideal variety.

Authors' Contribution

Each named author has substantially contributed to conducting the underlying research and drafting this manuscript, especially Dr Paracampo: analysis, interpretation of data, and writing original draft; Dr Abreu: analysis and revising this manuscript critically; Dr Lemos: resources and revising this manuscript critically; MSc Both: ensuring the integrity of the invetigation, and revising this manuscript critically.

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