USE OF SILVER COFFEE FILM: ALTERNATIVE EXTRACTION OF BIOACTIVE COMPOUNDS AND ANTIOXIDANT POTENTIAL FOR A CIRCULAR ECONOMY

Ana Terra de Medeiros Felipe[1], Ana Cecília de Azevedo Carvalho[2] and Katia Nicolau Matsui[3]

INTRODUCTION

According to the International Coffee Organization (ICO), approximately 4.01 billion kilograms were produced for arabica coffee in the years 2022 to 2023, generating a significant amount of waste along the entire production chain, from harvesting to processing and roasting (Thomlinson, 2018). Among the main by-products are the husk, pulp, mucilage, silver film and coffee grounds, whose improper disposal can cause serious environmental impacts, such as soil and water pollution, in addition to the emission of greenhouse gases. However, these residues contain bioactive compounds, arousing interest for their reuse in various chemical areas and food industries (Costa et al., 2017).

The silver film of coffee, also known as coffee silverskin, one of the main residues generated during the roasting of the beans, is a thin layer that surrounds the coffee and detaches during this process (Lorbeer et al., 2022). This material represents a significant fraction of the waste generated in coffee production and has the potential for applications in several areas due to its fiber content and other nutrients, although its use is still limited on an industrial scale (Gottstein et al., 2024). Although it is often discarded, it has bioactive compounds such as phenolic acids and flavonoids, with antioxidant activity, which can be used in different industrial applications (Hoseini et al., 2021). Recent studies have explored the use of this by-product in areas for the manufacture of biodegradable material, due to its bioactive properties (Castro et al., 2018). Thus, the silver film presents itself as a promising alternative to add value to the coffee chain and minimize the environmental impact of the coffee industry.

[1] Federal University of Rio Grande do Norte – RN
[2] Federal University of Rio Grande do Norte – RN
[3] Federal University of Rio Grande do Norte – RN

Coffee film is an agricultural by-product rich in bioactive compounds. Optimizing the extraction method can increase the yield and effectiveness of these compounds for applications in the food and cosmetics industry. The incorporation of residues, such as the silver film of coffee, is of great industrial interest due to its innovative potential (Costa et al., 2018)

Phenolic compounds are also responsible for the organoleptic characteristics of plant foods, influencing the color and flavor of the food, in addition to being interesting in food areas for their antioxidant and antibacterial properties (Alara et al. 2021). The antioxidant activity of coffee film can be evaluated by methods such as DPPH and ABTS, which measure its ability to donate electrons and neutralize radicals. Therefore, the identification of this activity is important to extend the shelf life of a package (Martinez-Saez et al., 2014).

The use of this biomass contributes to the reduction of solid waste, promoting a circular economy. The use of coffee film in industrial processes adds value to the production cycle, transforming a previously unused material into an ecological solution to problems such as excess waste and dependence on non-degradable plastics. (Hosseini et al., 2021).

OBJECTIVE

This study aims to provide an alternative to the coffee by-product, proposing value-added applications. Therefore, the use of coffee film for the extraction of bioactive compounds and the potential antioxidant activity of this residual biomass presents a sustainable alternative and contributes to a circular economy, creating economic and environmental opportunities, optimizing the use of natural resources.

METHODOLOGY

COLLECTION

The silver film of the coffee was provided by the company 3corações, located in Natal, Rio Grande do Norte, Brazil, (Latitude: 5°74'184"S, Longitude: 35°28'778"W) in October 2024 and taken to the food engineering laboratory (LEA) of the Federal University of Rio Grande do Norte (UFRN) for future analysis.

PRE-PREPARATION AND STANDARDIZATION

The samples were dried in an oven (Lucadema, São Paulo, Brazil) for 6 hours at 50 °C, then ground and sieved in mesh 32 for powder standardization.

HUMIDITY

The gravimetric method was used for moisture analysis. Approximately 3 grams of the sample were weighed on previously dried and tarted weighing porcelain. The samples were placed in an incubator (Lucadema, São Paulo, Brazil) at a temperature of 105°C until they reached a constant weight (AOAC, 2000).

WATER ACTIVITY

The analysis was performed using a direct water activity meter, equipped with a digital sensor (Aqualab S3TE, Decagon, USA). The samples were placed in small sampling capsules provided by the equipment, ensuring that their surface was completely exposed to the sensor for an accurate reading. The capsules were inserted into the equipment's reading chamber, which was closed and allowed to stabilize until it reached equilibrium with the internal environment.

EXTRACTION

The extraction was done using ethanol as a solvent in a combined method, first in an ultrasonic bath (ALTSONIC Clean 3IA, 3 L, São Paulo, Brazil) at a frequency of 40 kHz and 100 W, without agitation, for 15 minutes and then it was maintained under mechanical agitation (SP-10209/A, SP Labor, Brazil) at room temperature for 45 minutes. The proportion between the sample and ethanol was 2 g in 100 mL (1:50 g/mL). They were then vacuum filtered and the extracts were stored under refrigeration and protected from light.

TOTAL PHENOLIC COMPOUNDS (CFT)

Total phenolics were determined with the Folin-Ciocalteu method in 96-well microplates with modifications as proposed by Dung (2024). In 25 µL of the extract diluted 20 times, 50 µL of distilled water, 25 µL of Folin-Ciocalteu reagent (1:10, v/v) and 100 µL of 7.5% sodium carbonate (w/v) were added. The mixture was kept at rest under shelter from light for 90 minutes at room temperature. After the reaction time, the absorbance was measured at 765 nm using a microplate reader (ASYS UVM 340, Biochrom Ltd, England).

Previously, a standard curve was constructed with gallic acid in different concentrations (0 to 250 mg/L), allowing the quantification of total phenolic compounds. The results were expressed in gallic acid equivalents (GAE) per gram of dry sample (mg GAE/g).

ANTIOXIDANT ACTIVITY (DPPH)

To evaluate the antioxidant capacity by the DPPH (2,2-diphenyl-1-picrylhydrazil) method, measuring the reduction of free radicals following the method described by Bobo-García et al. (2014). A 150 µL aliquot of a methanolic solution of DPPH (150 µM) was added to 20 µL of the samples. The mixtures were then incubated at room temperature, in the dark, for 40 minutes, and the absorbance was measured at 515 nm using a microplate reader (ASYS UVM 340, Biochrom Ltd, England).

STATISTICAL ANALYSIS

All analyses were performed in at least triplicate and are presented as mean ± standard deviation. The analyses of bioactive compounds and antioxidant activity were performed in 8 replicates.

DEVELOPMENT

The drying process is essential for the preservation of plant foods, as it reduces the water content, limiting microbial activity and chemical reactions that can compromise the quality of the product. Humidity above 12% in these foods favors the growth of microorganisms, such as fungi, in addition to accelerating spoilage processes and reducing the stability of the product during storage (ANVISA, 2015; Lorbeer et al., 2022).

In the case of the silver film of the coffee, the analysis revealed a moisture content of 7.45%, as shown in Table 1, which is below the critical limit of 12%. This value indicates that the material has low susceptibility to microbiological degradation, favoring its conservation and storage. This characteristic is critical for its use in industrial applications or as a functional ingredient in food (Lorbeer et al., 2022).

Table 1. Physicochemical parameters for dried Clitoria petals.

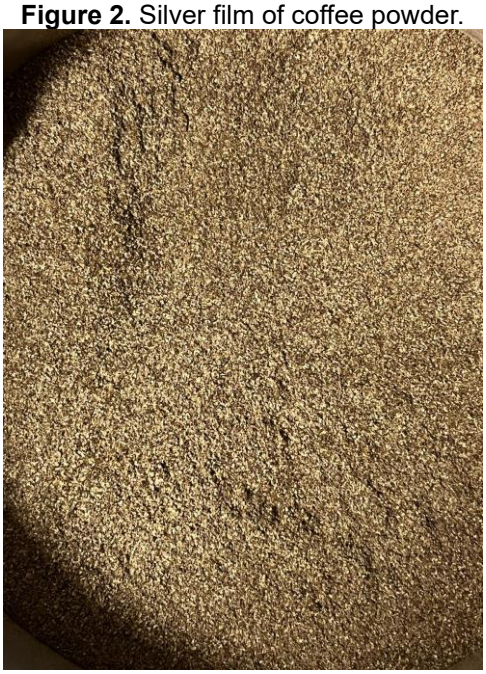
Physicochemical parameters	Average value
Humidity (%)	7.45 ± 1.39
Water Activity (aw)	0.445 ± 0.03

Source: Authors (2024).

Table 1 shows the value obtained for the moisture content of the silver coffee skin, being 7.45 ± 1.39, which is similar to that found by Gottstein et al. (2021) of 7.64% for the skin.

Water activity (Aw) analysis is essential for dry samples, it measures the availability of water for chemical reactions and microbial growth, and is a critical indicator of stability and safety (Gottstein et al., 2021). Aw values lower than 0.6 generally limit the development of microorganisms, while values close to 0.4, such as the one obtained for the silver film of coffee is shown in Table 1, around 0.445, indicating high microbiological stability. This level is sufficient to prevent significant spoilage during storage, highlighting the material's potential as a safe ingredient for food and industrial applications (Gottstein et al., 2021).

Figure 2. Silver film of coffee powder.



Source: Authors (2024).

The results obtained for the content of total phenolic compounds in the silver film of coffee demonstrated a significant concentration, reaffirming the potential of this by-product as a source of bioactive compounds. Compared to previous studies, the values found were similar or lower than those reported in different coffee cultivars and processing methods, indicating that the silver film is rich in polyphenols, especially chlorogenic acid. This variation can be attributed to factors such as the type of grain, the cultivation conditions, the waste generation process, and the extraction method used. In addition, the positive correlation between phenolic concentration and antioxidant capacity suggests that the silver

film can be exploited as a functional ingredient in food formulations, promoting the reuse of waste from the coffee industry and contributing to sustainability (Dong et al., 2024; Esposito et al., 2021).

In this study, the extraction assisted with an unconventional method of ultrasound combined with the conventional extraction of the silver film from the coffee resulted in a concentration of 274.63 mg EAG/100g, as shown in Table 2. It is worth mentioning that several intrinsic factors of the extraction process influence the yield of compounds such as phenolics and antioxidant capacity, such as temperature, extraction time, type of solvent used and the concentration of the solution.

In a recent study, Dong et al. (2024) using petroleum ether, acetone, and methanol as a solvent extractor and ultrasound for 30 minutes resulted in 474.64 mg EAG/g of dry extract. The result can be attributed to sequential extraction for 3 times and the use of solvents with higher polarity that are able to extract more phenolic compounds (Dong et al., 2024). Costa et al. (2018) reported approximate values of 4.3 mg EAG/g for ethanolic extracts.

Table 2. Concentration of total phenolic compounds and antioxidant activity against the DPPH radical of the coffee silver film extract obtained by ultrasound and mechanical agitation.

Analysis	Findings
Phenolic Compounds (mg EAG/100g)	274.63 ± 11.4
DPPH (%)	25.12 ± 0.71

Source: Authors (2024).

The data obtained for the antioxidant activity of the silver film of coffee, evaluated by the DPPH method, indicated a high capacity for free radical scavenging, with values above 25%. These results corroborate previous studies that highlight silver film as a rich source of natural antioxidants, mainly attributed to the presence of phenolic compounds, such as chlorogenic acids and flavonoids. Comparatively, the values obtained are within the range reported for coffee by-products in different studies, depending on the type of solvent and extraction method employed. The high correlation between the concentration of phenolic compounds and the antioxidant activity reinforces the potential of this by-product for applications in food formulations, packaging and cosmetics, contributing to the sustainable reuse of agro-industrial waste and the valorization of the coffee production chain.

The result obtained in the present study is that the extract has 25.12% of DPPH free radical scavenging capacity, as shown in Table 2. These results are in line with data in the

literature, which highlight silver film as a rich source of natural antioxidants, especially phenolic compounds such as chlorogenic acids and flavonoids (Dong et al., 2024). Compared to other coffee by-products, such as pulp and peel, the silver film showed superior antioxidant activity, which can be attributed to its differentiated chemical composition and high concentration of bioactive compounds. The direct relationship between the total phenolic content and the observed antioxidant activity reinforces the potential for using this residue as a functional ingredient in food and cosmetic products, promoting the sustainable use of by-products of the coffee chain (Castaldo et al., 2020).

In a similar study, Dong et al. (2024) reported an antioxidant activity of 50% for the same radical, using a concentration of 20 µg/mL and 448 µg/mL of extract from the isolated phenolic compound fractions. Costa et al. (2018) also found significantly higher values of antioxidant activity in extracts obtained by conventional methods (519 ± 37 mg TE/l for 100% ethanolic extract), indicating the potential for efficient extraction of antioxidant compounds from the silver film of coffee.

FINAL CONSIDERATIONS

The results obtained in this study reinforce the potential of the silver film of coffee as a promising source of bioactive compounds, particularly phenolics, with significant antioxidant properties. The application of optimized extraction methods, such as the combination of ultrasound and mechanical agitation, has proven to be effective in the recovery of compounds of interest, expanding the possibilities of using this by-product in food formulations, cosmetics and even biodegradable packaging. In addition, the low water activity and moisture content highlight the microbiological stability and the potential for prolonged storage, favoring its integration into industrial processes. The valorization of the silver film of coffee contributes to the sustainability of the production chain, promoting the circular economy and the use of agro-industrial waste in an innovative and environmentally responsible way.

REFERENCES

1. ALARA, Oluwaseun Ruth; ABDURAHMAN, Nour Hamid; UKAEGBU, Chinonso Ishamel. Extraction of phenolic compounds: A review. Current Research in Food Science, [S.l.], v. 4, p. 200-214, 2021.

2. AOAC. Official methods of analysis of the Association of Official Analytical Chemists. 15th ed. Arlington: AOAC, 1990.

3. BOBO-GARCÍA, Gloria et al. Intra-laboratory validation of microplate methods for total phenolic content and antioxidant activity on polyphenolic extracts, and comparison with conventional spectrophotometric methods. Journal of the Science of Food and Agriculture, v. 95, n. 1, p. 204-209, 2015. DOI: 10.1002/jsfa.6706.

4. CASTALDO, L. et al. In vitro bioaccessibility and antioxidant activity of coffee silverskin polyphenolic extract. Molecules, v. 25, n. 9, p. 2132, 2020. Disponível em: https://doi.org/10.3390/molecules25092132.

5. CASTRO, A.C.C.M.; ODA, F.B.; ALMEIDA-CINCOTTO, M.G.J.; DAVANÇO, M.G.; CHIARI-ANDRÉO, B.G.; CICARELLI, R.M.B.; PECCININI, R.G.; ZOCOLO, G.J.; RIBEIRO, P.R.V.; CORRÊA, M.A.; ISAAC, V.L.B.; SANTOS, A.G. Green coffee seed residue: A sustainable source of antioxidant compounds. Food Chemistry, v. 246, p. 48–57, 2018.

6. COSTA, Anabela S.G. ALVES, Rita C.; VINHA, Ana F.; COSTA, Elísio; COSTA, Catarina S.G.; NUNES, M. Antónia; ALMEIDA, Agostinho A.; SANTOS-SILVA, Alice; OLIVEIRA, M. Beatriz P.P. (2017). Nutritional, chemical and antioxidant/pro-oxidant profiles of silverskin, a coffee roasting by-product. Food Chemistry 267 (2018) 28–35.

7. COSTA, Anabela S.G. et al. Nutritional, chemical and antioxidant/pro-oxidant profiles of silverskin, a coffee roasting by-product. Food Chemistry, [S.l.], v. 267, p. 28–35, 2018.

8. DONG, S.; DING, L.; ZHENG, X.; WANG, O.; CAI, S. Phenolic Compositions of Different Fractions from Coffee Silver Skin and Their Antioxidant Activities and Inhibition towards Carbohydrate-Digesting Enzymes. Foods, v. 13, n. 19, p. 3083, 2024. Disponível em: https://doi.org/10.3390/foods13193083.

9. ESPOSITO, L.; DI MATTIA, C. D.; RICCI, A.; MASTROCOLA, D. Characterization of Coffee Silver Skin as Potential Food-Safe Ingredient. Foods, v. 10, n. 6, p. 1367, 2021. Disponível em: https://doi.org/10.3390/foods10061367.

10. GOTTSTEIN, V.; BERNHARDT, M.; DILGER, E.; KELLER, J.; BREITLING-UTZMANN, C. M.; SCHWARZ, S.; KUBALLA, T.; LACHENMEIER, D. W.; BUNZEL, M. Coffee Silver Skin: Chemical Characterization with Special Consideration of Dietary Fiber and Heat-Induced Contaminants. Foods, v. 10, n. 8, p. 1705, 2021. Disponível em: https://doi.org/10.3390/foods10081705.

11. HOSEINI, Marziyeh; COCCO, Stefania; CASUCCI, Cristiano; CARDELLI, Valeria; CORTI, Giuseppe. Coffee by-products derived resources. A review. Biomass and Bioenergy, v. 148, p. 106009, 2021.

12. HOSSEINI, Seydeh Negar; PIRSA, Sajad; FARZI, Jafar. Biodegradable nano composite film based on modified starch-albumin/MgO; antibacterial, antioxidant and structural properties. Polymer Testing, v. 97, p. 107182, 2021.

13. INTERNATIONAL COFFEE ORGANIZATION (ICO). Annual Review 2022-2023. Londres: ICO, 2023. Disponível em: https://www.ico.org. Acesso em: 16 out. 2024.

14. MARTINEZ-SAEZ, Nuria; ULLATE, Mónica; MARTIN-CABREJAS, María A.; MARTORELL, Patricia; GENOVÉS, Salvador; RAMON, Daniel; DEL CASTILLO, María Dolores. A novel antioxidant beverage for body weight control based on coffee silverskin. Food Chemistry, v. 150, p. 227–234, 2014.

15. SCHWARZ, S.; FRANKE, H.; LACHENMEIER, D. W. Toxicological Assessment of Roasted Coffee Silver Skin (Testa of Coffea sp.) as Novel Food Ingredient. Molecules, v. 27, n. 20, p. 6839, 2022. Disponível em: https://doi.org/10.3390/molecules27206839.

16. THOMLINSON, Thayer. Coffee & biochar: exploring biochar as a solution for sustainability in the coffee industry. Biochar International, 2018. Disponível em: https://biochar-international.org/wp-content/uploads/2023/01/Coffee_FINAL_Oct-2018.pdf. Acesso em: 02 nov. 2024.