

## A NATURAL ALTERNATIVE TO CARAMEL COLOR IV IN FOOD AND BEVERAGES WITH THE COFFEE SILVER SKIN OF ARABICA BEANS

UMA ALTERNATIVA NATURAL AO CORANTE CARAMELO IV EM ALIMENTOS E BEBIDAS COM A PELÍCULA PRATEADA DE GRÃOS DE CAFÉ DA CULTIVAR ARÁBICA

UNA ALTERNATIVA NATURAL AL COLORANTE CARAMELO IV EN ALIMENTOS Y BEBIDAS CON LA PELÍCULA PLATEADA DE LOS GRANOS DE CAFÉ DE LA VARIEDAD ARÁBICA

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DOI: 10.54899/dcs.v23i91.5856

Recibido: 12/06/2026 | Aceptado: 15/06/2026 | Publicación en línea: 30/06/2026.

### ABSTRACT

This study aimed to develop a natural alternative to brown-colored dye additives, especially caramel dye IV (INS 150d), by preparing/obtaining an extract from coffee silver skin, which is a byproduct of grain processing, for use in food and beverages. Its application as an alternative to iron oxide (INS 172) in pet food should also be considered, since this alternative does not have an abrasive or corrosive effect on equipment. The methodology was organized through the development of an aqueous extract of a sample of coffee silver skin and physical-chemical characterization. In this sense, excellent results were found in relation to °Brix (53.4°), hue (8940 EBC) and turbidity (1.3 NTU). However, as this study was a first trial, it is still necessary to test samples of the extract of skin obtained with different roasting temperatures and/or from different

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varieties. Still, it is possible to conclude that this dye can be a great natural contribution to new products, such as yogurts, soft drinks, ice cream, jelly candies and muffins.

**Keywords:** Innovation. Technology. New Products. Waste. Natural Dye.

### RESUMO

Esse estudo teve como objetivo desenvolver uma alternativa natural aos aditivos de corantes com tonalidade marrom, em especial ao corante caramelo IV (INS 150d), sendo elaborado/obtido um extrato a partir da película prateada do café, que é um subproduto do beneficiamento dos grãos, para aplicação em alimentos e bebidas. Também deve-se levar em consideração sua aplicação como alternativa ao óxido de ferro (INS 172) em alimentos para animais de estimação, uma vez que esta alternativa não possui efeito abrasivo ou corrosivo em equipamentos. A metodologia foi organizada através do desenvolvimento de um extrato aquoso de uma amostra de película prateada de café e caracterização físico-química. Neste sentido, foram encontrados resultados ótimos em relação ao °Brix (53,4°), tonalidade (8940 EBC) e turbidez (1,3 NTU). Todavia, como este estudo foi um primeiro ensaio, ainda se torna necessário testar amostras do extrato de película obtida com diferentes temperaturas de torra e/ou oriunda de variedades. Ainda assim, é possível concluir que esse corante pode ser uma grande contribuição natural para novos produtos, como: iogurtes, refrigerantes, sorvetes, balas de gelatina e muffin.

**Palavras-chave:** Inovação. Tecnologia. Novos Produtos. Resíduos. Corante Natural.

### RESUMEN

Este estudio tuvo como objetivo desarrollar una alternativa natural a los aditivos colorantes marrones, especialmente colorante caramelo IV (INS 150d), con un extracto preparado/obtenido de la película plateada del café, que es un subproducto del procesamiento del grano, para su aplicación en alimentos y bebidas. También se debe considerar su aplicación como alternativa al óxido de hierro (INS 172) en alimentos para mascotas, ya que esta alternativa no tiene efecto abrasivo ni corrosivo en los equipos. La metodología se organizó mediante el desarrollo de un extracto acuoso a partir de una muestra de película de plata de café y su caracterización físicoquímica. En este sentido, se encontraron excelentes resultados en relación con °Brix (53,4°), tonalidad (8940 EBC) y turbidez (1,3 NTU). Sin embargo, como este estudio fue una primera prueba, aún es necesario analizar muestras de extracto de piel obtenidas a diferentes temperaturas de tostado y/o de diferentes variedades. Aun así, es posible concluir que este colorante puede ser un gran aporte natural para nuevos productos, tales como: yogures, refrescos, helados, caramelos de gelatina y muffins.

**Palabras clave:** Innovación. Tecnología. Nuevos Productos. Desperdiciar. Tinte Natural.



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## INTRODUCTION

Throughout its production chain, until it reaches the consumer, by-products from the coffee industry are produced in large quantities each year. Among other residues, the Coffee Silver Skin (CSS) represents the largest volume of by-product generated during the coffee roasting stage (Gottstein *et al.*, 2021).

In the research by Martuscelli *et al.* (2021), it was possible to characterize the composition of the coffee husk/coffee silver skin and highlight its potential for use as a safe food ingredient in new formulations. The results showed an average total dietary fiber content of 50%, but with a higher proportion of insoluble than soluble fiber. A high content of total phenolic compounds, chlorogenic acid, caffeine, and acid was found and correlated with the high measured antioxidant capacity. In conclusion, the film may be a potential safe food ingredient.

Galvão *et al.* (2024), found several uses for coffee silver skin (CSS), such as: phenolic compounds, functional cookies, sound absorption and other applications in this type of industry, electrodes, dietary products, fertilizers and covering film for agriculture, and biofuel. The authors also suggest that CSS is an excellent source of dietary fiber and demonstrates the possibility of its application in baking and other food products, in addition to protein extraction, and can be an excellent base for new products.

In addition to these applications, the coffee silver skin can also be used as a colorant, as it has an intense dark hue. Caramel color has become one of the most widely used colorants in various foods and beverages due to its complete miscibility with water and excellent coloring properties (Li *et al.*, 2021). Class III and IV caramel color is widely present in soft drinks (non-alcoholic beverages such as Coca-Cola®), a market that is growing annually (Takizawa *et al.*, 2021).

The most used food additive to obtain the brown hue is caramel coloring (INS 150 a, b, c, d), according to Anvisa (2015). Others, such as mixtures of natural or artificial red and green food colorings, iron oxide (INS 172), or artificial brown HT coloring (INS 155), are also widely used. However, all these options are increasingly accepted by consumers, have limited technological applicability, or are only approved for certain food categories.

The food industry chooses to use artificial colors because they are cheaper, but their use can be harmful to health and can cause everything from allergic reactions to the development of some types of cancer (Oliveira; Gherardi; Almeida, 2024). Therefore, the industry has been

researching alternatives to replace these artificial colors, which offer high stability during processing, storage and final product, and which are also produced sustainably and allow a clean label declaration.

In this sense, the objective of this study was to develop a natural alternative to brown color additives, especially caramel IV colorants (INS 150d), obtained from an aqueous extract using the coffee silver skin, which is a byproduct of the processing of beans, for application in food and beverages. Its application as an alternative to iron oxide (INS 172) in pet food should also be taken into consideration, since this alternative does not have an abrasive or corrosive effect on equipment.

## **TECHNIQUES APPLIED TO OBTAIN PRODUCT AND QUANTITATIVE RESULTS**

Since it is an alternative to dyes, the method chosen to measure the hue of the product obtained was the one widely applied to malt extracts, brewing worts, beers and liquid malt substitutes. There are two methods for determining the color of beers: the visual method and the spectrophotometric method. The spectrophotometric method was designed to eliminate subjective effects attributable to the human eye, as well as differences in color impression when comparing beer samples with the color comparator disc (visual method).

Thus, the Analytica-EBC method was used; Section 9 Beer. Method 9.6 Colour of Beer: Spectrophotometric Method (Analytica-EBC, 2010). The sample of the aqueous extract of coffee silver skin developed was diluted with deionized water, so that the reading was within the measuring range of the spectrophotometer (samples before concentration: 0.5% solution / sample after concentration: 0.1% solution). The spectrophotometer was zeroed against a white cuvette, which consists of a cuvette containing distilled water. The hue of the sample was measured in a cuvette at 430 nm.

The samples, as well as the deionized water, were filtered through a 0.45 µm membrane filter in order to remove particles that contribute to turbidity. This filtration is not necessary if the turbidity of the (diluted) sample is less than 1 EBC turbidity unit, which is a measure of the amount of suspended particles in a liquid, especially used in the brewing industry to assess the clarity of beer (European Brewery Convention, 2025).

In the case of samples that present reading results outside the spectrophotometer range, it is necessary to dilute them so that their color is within the measurement range. The corresponding

dilution factor was used to calculate the result (measurement result x dilution factor). The EBC color was calculated according to the following equation:

$$\text{Color (EBC Unit)} = A_{430} \times 25 \times F \quad (1)$$

where,

“A430” is the absorbance of the sample measured at 430nm, “25” refers to the conversion factor and “F” is the dilution factor of the sample used. The EBC system uses a spectrophotometer to measure the absorption of light at different wavelengths, allowing the determination of color in EBC units.

The proposed process steps were carried out according to the sequence explained below, in the laboratories of the State University of Rio Grande do Sul – Uergs / RS / Brazil.

### Infusion

The coffee silver skin of arabica beans was ground to obtain flour. After grinding, the infusion was prepared by adding 10 g of flour to 200 mL of water at 60 °C. The solution was kept stirring at 60 °C for 180 minutes (3 hours), as can be seen in Figure 01.

Figure 1 Infusion process.





Source: Authors (2025).

## Separation of Solids

After the infusion time, the solution was centrifuged at 4000 rpm for 5 minutes (simulation of industrial operation using a “centrifugal decanter”). The solids had a “compacted” appearance (Figure 2). The supernatant had 1.1 °Brix and a hue of 107 EBC.

Figure 2 Separation of solids.



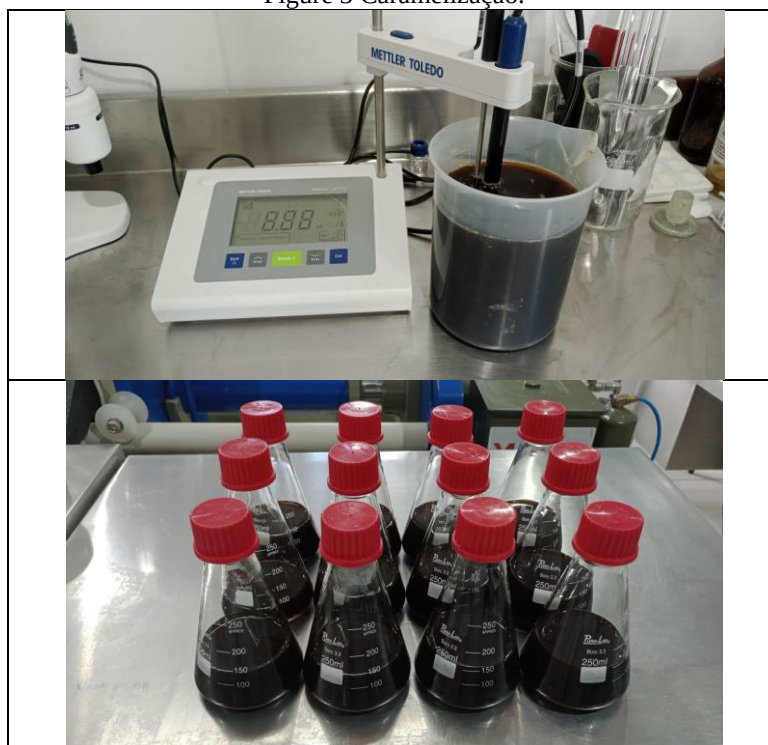
Source: Authors (2025).

## Caramelization

The pH of the supernatant obtained in the solids separation step was adjusted to 9.0, with the addition of calcium hydroxide. Subsequently, the solution was kept at 90°C, under stirring, for 12 hours (Figure 03).

After the first hour, the solution presented a hue of 137 EBC. After the proposed 12 hours, the characteristics obtained were: 1.2 °Brix, pH 6.46, turbidity 204 NTU and hue 175 EBC.

Figure 3 Caramelização.



Source: Authors (2025).

## Ultrafiltration (Simulation = Vacuum System + Pressure System)

To simulate the vacuum filtration system, a mixture composed of 75% Perlite AF 272P (Permeability:  $\approx 2.5$  Darcy) and 25% Perlite AF 332 (Permeability:  $\approx 1.8$  Darcy) was used. The bottom of the Buchner funnel was lined with black band quantitative filter paper (7.5 microns). The perlite mixture was added to the solution, homogenized and subsequently filtered (Figure 04).

Then, 3 g/L of PVPP (polyvinylpolypyrrolidone) was added to the solution, before simulating the pressure filtration system. In the system simulation, the Buchner funnel was also

coated with black stripe quantitative filter paper (7.5 microns), with the addition of layers of diatomaceous earth according to the following sequence: first layer with diatomaceous earth of permeability:  $\approx 1.10$  Darcy, second layer with diatomaceous earth of permeability:  $\approx 0.26$  Darcy) and third layer with diatomaceous earth of permeability:  $\approx 1.04$  Darcy. Before filtration, small doses of diatomaceous earth were added to the solution, and then homogenized.

Figure 4 Ultrafiltration process.



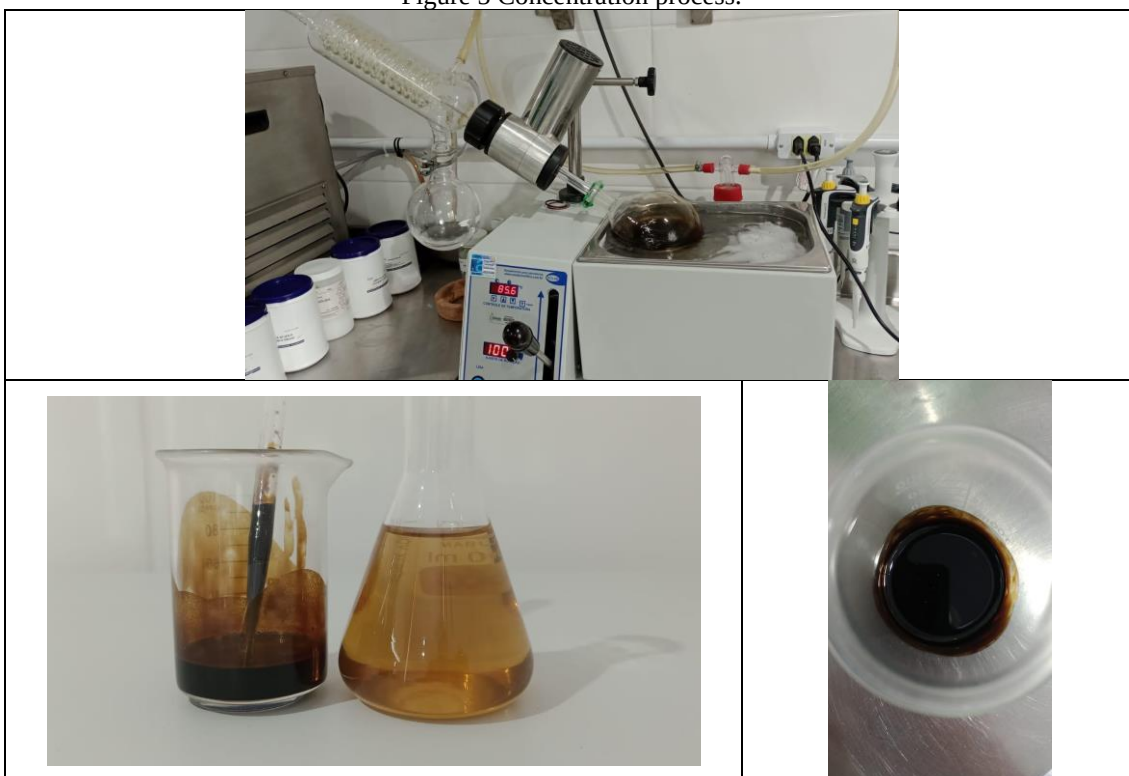
Source: Authors (2025).

After ultrafiltration, the characteristics obtained were:  $1.1^{\circ}\text{Brix}$ , pH 6.46, turbidity 1.3 NTU and hue 105 EBC.

### Concentration

The solution was concentrated using a vacuum rotary evaporator until  $53.4^{\circ}\text{Brix}$  was obtained and the shade obtained was 8940 EBC. Figure 05 shows the comparison of the product obtained (concentrated) and the initial sample, in the 0.1% solution in water.

Figure 5 Concentration process.



Source: Authors (2025).

## RESULT AND DISCUSSION

The study was carried out only once, with the intention of verifying the possibility of developing a natural alternative to caramel color IV (INS 150d), well pigmented, so that low percentages of use could be capable of coloring food and beverages, without leaving a residual flavor and/or contributing to turbidity in food and beverage applications.

An important technological advantage of this process using a residue from the coffee industry is the fact that the extract obtained is not considered a food additive or coloring. It is a natural ingredient, which can be declared as such, without the need for an INS number (International Numbering System, that is, the International Numbering System for food additives).

Some parameters were monitored during the extract preparation process, such as: °Brix, hue, pH and turbidity. In this sense, Table 01 shows the results obtained in the process monitoring. The greatest difference occurred in the °Brix value, turbidity and hue. In the color measurement test, the beer industry usually uses the factor “30.8”, instead of “25” as indicated by the EBC Methodology. This is a “market standard” for evaluating the hue of dyes.

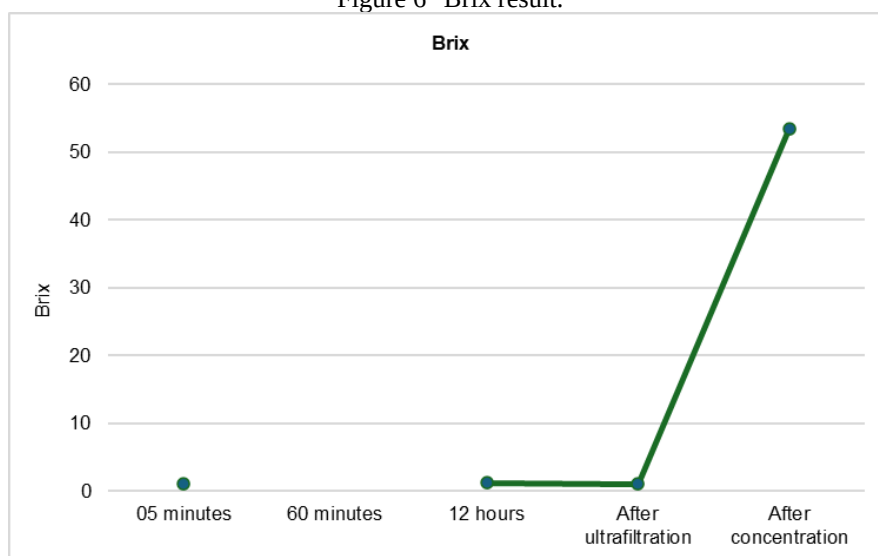
Table 1 Results of the applied process.

| Process time          | Brix  | Hue - EBC | pH   | Turbidity - NTU |
|-----------------------|-------|-----------|------|-----------------|
| 05 minutes            | 1,1°  | 107       | -    | -               |
| 60 minutes            | -     | 137       | -    | -               |
| 12 hours              | 1,2°  | 175       | 6,46 | 204             |
| After ultrafiltration | 1,1°  | 105       | 6,46 | 1,3             |
| After concentration   | 53,4° | 8940      | -    | -               |

Source: Authors (2025).

Regarding °Brix, this is a scale widely used in the food and beverage industry to control the quality of fruit juices, wines, soft drinks, jams and other products by measuring the number of soluble solids in a sucrose solution. The last value found in the analysis was 53.4 °Brix (Figure 06). Thus, it proves to be a powerful natural base for other products, since the higher the °Brix value, the sweeter the products will be.

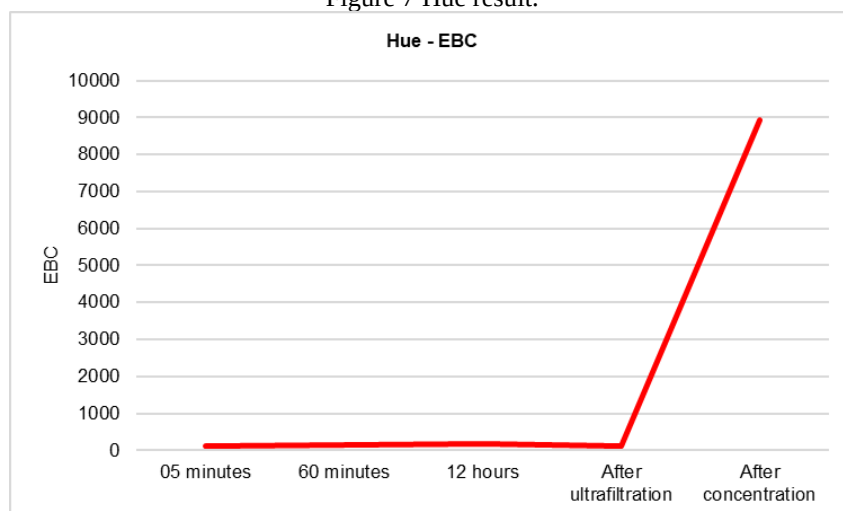
Figure 6 °Brix result.



Source: Authors (2025).

Dyes are used to add or intensify color in foods, beverages, cosmetics and other products. In this sense, the value found in the study gradually increased with the processes that were applied, reaching the value of 8940 EBC, after the concentration stage (Figure 07). This result was above expectations and can be applied in several industries.

Figure 7 Hue result.



Source: Authors (2025).

Regarding turbidity (Nephelometric Turbidity Unit – NTU), which refers to the presence of suspended or colloidal particles in the water that scatter light, making it cloudy or opaque, it was found that the values decreased from 204 to 1.3, which is an excellent result for the first test carried out.

El-Anany *et al.* (2024) evaluated the nutrient content and acceptable qualities of vegetable burgers formulated with chickpea flour, defatted sesame cake flour, coffee husks and pomegranate juice as a colorant. The authors concluded that the proposed product was favorably received.

Borromei *et al.* (2024) found that coffee husks are rich in substances of interest to various sectors, such as antioxidants, chlorogenic acids, and dietary fiber. Thus, the authors organized a project to develop extraction routes for coffee husks, aiming to increase the yield and quality of the extracted compounds, avoiding their degradation. They concluded that the approach is promising for exploring natural resources in a sustainable and efficient way.

In the two works cited above, it was possible to analyze the entire coffee husk, and not just the coffee silver skin, in this sense, this research appears promising for future applications of this residue.

However, since this study was a first trial, it is still necessary to test the application of the coffee silver skin with different roasting temperatures and/or from different varieties, since during the roasting process pigmented compounds are formed due to the Maillard reaction, altering the characteristics of the desired aqueous extract. This is a chemical reaction responsible for the formation of flavors and colors in foods during cooking, and is influenced by the pH of the

sample, with a more alkaline pH tending to accelerate it, resulting in more intense coloration and more complex flavors.

In view of this, it is also necessary to evaluate the need for alkalization of the solution, since there are soluble sugars and proteins in sufficient quantities to promote pigmented compounds during the caramelization stage suggested in this process flowchart, since during the test the solution was alkalized to accelerate the Maillard reaction, increasing the final hue of the extract.

If the processing maintains the alkalization stage, with the use of hydroxide, it is necessary to evaluate the shade obtained at shorter intervals (for example: every 15 minutes). It is also important to determine the ideal time for greater development of pigmented compounds, since keeping the solution agitated at a temperature of 90°C for 12 hours would increase the costs of the process on an industrial scale. On the other hand, it is important to consider that hydroxides are considered food additives and, according to current Brazilian regulations, must be declared in the list of ingredients. Including this additive in the list of ingredients could cause the extract to lose its “natural” claim.

It is also necessary to evaluate the performance of the raw material in applications with different percentages of the aqueous extract of coffee silver skin in products such as: YOGURT (in concentrations of 0.25%, 0.50% and 0.75%); SLUSH ICE or ICE CREAM (proposed dosages: 0.10% and 0.25%); GELATIN CANDY (proposed dosages: 0.05% and 0.10%) and MUFFIN (proposed dosage: 0.55%), as well as the pigmentation power in the applications, its stability and possible residual flavors in the products.

The solids resulting from the centrifugation stage can be reused through dehydration, since they contain insoluble fibers and proteins, but their characterization is necessary. Much of the bitterness, which was pointed out as a challenge in some bibliographic references on the application of coffee silver skin in food products, comes from phenolic compounds that are generally soluble. In other words, it can be inferred that most of the phenolic compounds migrated to the solution and were retained with the help of PVPP in the pressure ultrafiltration stage.

Therefore, it is also necessary to evaluate the dosage of PVPP used during the preparation of the extract, to ensure its complete removal, since if the polyphenols are not completely removed, during application in beverages containing proteins, there may be formation of a protein-polyphenol complex, which generates cloudiness during the shelf-life.

It is also possible to test the ultrafiltration stage using a membrane system, in order to

avoid the generation of solid waste from vacuum and pressure systems: Diatomaceous earth, like perlite, in addition to being expensive, are difficult to treat if sent to Effluent Treatment Plants, since some of the particles float and others settle. It is necessary to test which porosity is most suitable for this type of application in an ultrafiltration system (membrane suggestion: 100 kDa and 200 kDa). Ultrafiltration is recommended for applications of this alternative to caramel color IV (INS 150d) in beverages, such as soft drinks, so that the color/aqueous extract does not alter the turbidity of the product.

It is extremely important to consult ANVISA to validate the legal framework that this alternative use of the coffee silver skin is not a “new ingredient”, since there is a history of safe consumption of coffee in a form similar to that used in regular food. Coffee is already part of the general population's diet, being consumed in the form of infusion.

In relation to legislation, article 13 of Decree Law No. 986/69 (Brasil, 1969) is considered, which only requires that foods containing artificial colors display the statement "Artificially Colored" on the label.

Current Brazilian health legislation, in article 12 of Resolution RDC No. 727/2022 (Anvisa, 2022), determines that this information must appear at the end of the list of ingredients, by indicating the function that the additive performs in the food and its full name and/or INS number (International Numbering System, Codex Alimentarius FAO/WHO).

Color additives that are exempt from FDA certification, the United States government agency that regulates the safety of foods, drugs, cosmetics, and other products, generally include dyes and pigments derived from natural sources, such as vegetables, minerals, or animals, such as: annatto extract (yellow), dehydrated beetroot (blue-red to brown), caramel (yellow to brown), beta-carotene (yellow to orange), and grape skin extract (red or purple).

The United States Federal Food, Drug, and Cosmetic Act (FD&C Act, 2025) provides that a substance capable of imparting color is a color additive and is subject to premarket approval requirements unless the substance is used in a product solely for a purpose other than coloring and is clearly unimportant to the marketing of the product.

## CONCLUSION

Since caramel colorants are widely used food additives in the food industry to impart brown coloration to a broad range of products, concerns regarding their potential health effects

have encouraged the search for natural and sustainable alternatives. In this context, coffee silver skin, a major by-product of the coffee industry, emerged as a promising raw material due to its availability, valorization potential, and reported bioactive properties.

The results obtained in this study demonstrated the feasibility of producing a natural colorant from coffee silver skin aqueous extract, showing satisfactory physicochemical characteristics, including soluble solids content (53.4 °Brix), color intensity (8940 EBC), and low turbidity (1.3 NTU). These findings indicate that the extract has considerable potential for application as a natural brown colorant in food systems. In addition to contributing to the replacement of synthetic or highly processed colorants, the use of coffee silver skin aligns with circular economy principles by adding value to an agro-industrial residue that is often underutilized.

As this research represents an initial investigation, further studies are recommended to optimize extraction conditions and evaluate the influence of different roasting levels, coffee varieties, and processing methods on color development, stability, and sensory characteristics. Future research should also assess the colorant's shelf life, technological performance in different food matrices, consumer acceptance, and possible nutritional and functional benefits associated with its bioactive compounds.

Overall, the results suggest that coffee silver skin extract is a promising natural alternative to conventional caramel colorants and may contribute to the development of cleaner-label food products. Potential applications include dairy products such as yogurt and ice creams, confectionery products such as jelly candies, and baked goods such as muffins, among other food formulations requiring brown coloration. The adoption of this ingredient could simultaneously promote innovation, sustainability, and the valorization of coffee-processing by-products within the food industry.

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