Foods 2021

MDPI

Proceeding Paper

Comparative Investigation on Coffee Cascara from Dry and Wet Methods: Chemical and Functional Properties †

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† Presented at the 2nd International Electronic Conference on Foods, 15–30 October 2021; Available online: https://foods2021.sciforum.net/.

Abstract: Coffee processing involves the separation of its different structures considered by-products. There is a current interest in adding new value to coffee by-products through their conversion into food ingredients. Following a biorefinery approach, coffee by-products can produce phenolic and caffeine-rich extracts and water-insoluble residues (WIRs) rich in dietary fiber. Consequently, this work aimed to study the flour and the WIR (obtained through a sustainable and optimized aqueous extraction of phenolic compounds) from the main by-product of coffee processing, the coffee cascara, investigating the chemical and functional differences among ingredients obtained from the dry and wet processing methods. Both dry and wet cascaras (in flour and WIR) presented a high dietary fiber content (46–97%), especially outstanding in the WIRs. Soluble dietary fiber was 2.2 to 3.6-fold higher ($p < 0.05$) in flours than in WIRs. The wet coffee cascara flour exhibited a remarkable antioxidant capacity (2.4 to 4.2-fold higher than the other products), as well as adequate technofunctional and physicochemical properties. All by-products inhibited α-amylase (62–96%) and reduced starch hydrolysis (52–97%), which was associated ($r = 0.965$, $p < 0.05$) with the differential total phenolic content found in samples (6.1–40.4 mg gallic acid equivalents per gram). Similarly, coffee cascara-based ingredients showed pancreatic lipase inhibitory properties (54–65%) and reduced the intestinal absorption of cholesterol (50–88%) and bile salts (81–90%) in vitro. In conclusion, both dry and wet coffee cascara exhibit a similar chemical composition and functional properties and could be revalued as new sustainable ingredients (flours and WIRs, and phenolic-rich extracts) following a biorefinery approach. These coffee cascara-based ingredients may exhibit beneficial health properties reducing oxidative stress and glucose and lipid absorption.

Keywords: coffee by-products; coffee cascara; coffee husk; coffee pulp; dietary fiber; phenolic compounds; antioxidant capacity; hypoglycemic; hypolipidemic

Citation: Cano-Muñoz, P.; Rebollo-Hernanz, M.; Braojos, C.; Cañas, S.; Gil-Ramirez, A.; Aguilera, Y.; Martin-Cabrejas, M.A.; Benitez, V. Comparative Investigation on Coffee Cascara from Dry and Wet Methods: Chemical and Functional Properties. *Biol. Life Sci. Forum* **2021**, *68*, x. https://doi.org/10.3390/xxxxx

Academic Editor(s):

Published: date

1. Introduction

The International Coffee Organization reported a worldwide consumption in 2020/21 of 166,346 in thousand 60 kg bags, mainly consumed in the European continent (54,065 in thousand 60 kg bags) [1]. Since obtaining coffee beans requires separating their outer layers, the high coffee consumption implies generating a significant amount of by-products. However, the by-products generated vary depending on the coffee cherry processing method. The most commonly used processes are the dry and the wet methods [2,3]. Regarding dry processing, the by-products generated are the husk or dry cascara (compris-

ing the skin, pulp, mucilage, parchment) and silverskin (during roasting). In contrast, during the wet coffee cherry processing, the by-products relieved are pulp or wet cascara, parchment, and silverskin [4]. Dry and wet coffee cascaras have been demonstrated to exhibit an adequate nutritional composition. The dry coffee cascara has a high dietary fiber content (26–32 g/100 g) and low lipid content (0.5–3.0 g/100 g). Comparably, dietary fiber (16–24 g/100 g) stands out over the lipid content (1.3–2.5 g/100 g) in the wet coffee cascara [5]. The significant amount of dietary fiber in these by-products generates attention since an appropriate dietary fiber intake provides numerous health benefits. Following a biorefinery approach, coffee by-products can produce phenolic and caffeine-rich extracts and water-insoluble residues (WIRs) rich in dietary fiber [6]. The bioactivity of coffee cascara extracts has already been investigated. Caffeine and phenolic compounds from this by-product can reduce adipogenesis, oxidative stress, and inflammation in the adipose tissue and regulate lipid and glucose metabolism in the liver, thereby preventing obesity and related comorbidities such as non-alcoholic fatty liver disease [7,8]. However, dietary fiber-rich fractions have received less attention and require further research to be fully characterized.

The benefits of dietary fiber include reduced risk of intestinal diseases such as diverticulitis, constipation, inflammatory bowel disease, and colon cancer [9,10]. The fermentation of dietary fiber leads to short-chain fatty acids production, which positively affects mineral utilization and regulates the microbiota [11]. Furthermore, its hypolipidemic and hypoglycemic properties also stand out, which leads to benefits at the level of prevalent diseases such as diabetes, obesity, or coronary heart disease [12]. Nevertheless, globally, a large part of the population does not achieve dietary fiber requirements since its consumption does not reach 15 g/day when it is estimated that the intake should be between 19–28 g/day, being the optimum 24 g/day [13].

Hence, the present work studied the valorization of dry and wet coffee cascaras as sustainable ingredients rich in dietary fiber. For this purpose, the content of total, insoluble and soluble fiber fractions and their chemical characterization, the techno-functional properties, in vitro antioxidant capacity, and hypoglycemic and hypolipidemic properties were evaluated. This study could contribute to the valorization of these by-products as sustainable ingredients rich in dietary fiber.

2. Materials and Methods

2.1. Materials

Supracafé S.A supplied dry and wet coffee cascaras belonging to the Coffea arabica species. Raw samples were ground using a laboratory grinder to obtain the flour. WIRs were obtained by extracting aqueous soluble phytochemicals (0.02 g flour/mL water) for 90 min at 100 °C using an optimized green extraction method [6]. Thus, four samples were considered: flours and WIRs from dry and wet coffee cascaras.

2.2. Dietary Fiber Extraction and Characterization

Total dietary fiber (TDF), insoluble dietary (IDF), and soluble dietary fiber (SDF) were determined by enzymatic–gravimetric assay according to AOAC-991.4 and AACC-32.07.01 methods [14,15]. Dietary fiber (DF) fractions were obtained as indigestible residues after enzymatic digestion of non-dietary fiber components; the insoluble residues were isolated by filtration, and soluble fiber was precipitated with ethanol. Dried residues correspond to IDF and SDF, respectively. The residual ashes and proteins were determined according to standard procedures in the residues for corresponding corrections. TDF was calculated as the sum of IDF and SDF.

2.3. Phenolic Compounds Extraction and In Vitro Antioxidant Capacity Determination

Free (FPC) and bound (BPC) phenolic compounds were extracted in cascara samples according to a previously published procedure [16]. FPC and BPC extracts were analyzed

by the Folin-Ciocalteu method [17]. Total phenolic compounds (TPC) were obtained as the sum of FPC and BPC. The direct and indirect in vitro antioxidant capacity was estimated by $ABTS^{+}$ assay [18].

2.4. Physicochemical and Techno-Functional Properties

Bulk density (BD), water holding capacity (WHC), oil holding capacity (OHC), water absorption capacity (WAC), swelling capacity (SWC), emulsifying activity (EA), foaming capacity (FC), and gelation capacity (LGC, last gelation concentration), were measured according to published protocols [19].

2.5. Evaluation of the In Vitro Hypoglycaemic Properties

2.5.1. Glucose Adsorption Capacity

WIRs and flours (0.25 g) and 25 mL of glucose solution (10, 50, 100, 200 mM) were mixed [19], incubated at 37 °C for 6 h, and centrifuged (3500× *g*, 15 min). The glucose kit (Megazyme KGLUC, Wicklow, Ireland) was used to determine the glucose adsorbed.

2.5.2. In Vitro α-Amylase Inhibition

WIRs and flours (0.25 g) were mixed with 1 mg of α-amylase (Sigma-Aldrich, Saint Louis, MO, USA) and a solution of potato starch (4% *p*/*v*, 10 mL) and incubated at 37 °C for 60 min. The samples were centrifuged (3500× *g*, 15 min) and the supernatant's glucose content was determined using the glucose assay kit (KGLUC, Megazyme, Wicklow, Ireland). Residual amylase activity was determined in comparison with the control without sample [19].

2.5.3. In Vitro Glucose Dialysis Retardation Capacity

In vitro glucose dialysis retardation capacity was estimated by evaluating the glucose dialysis retardation index (GDRI) [19]. The solution was made with 0.5 g of WIRs or flours and 25 mL of glucose solution (50 mM) and was dialyzed against distilled water (80 mL) at 37 °C for 0–150 min. Finally, the glucose content in the dialysate was measured using the assay kit KGLUC (Megazyme, Wicklow, Ireland).

2.5.4. In Vitro Starch Digestibility Retardation

As previously described [19], a solution was formulated adding 0.2 g of flour, 4 mg of α-amylase (Sigma Aldrich, MO, USA), and 10 mL of potato starch solution (4% *p*/*v*) dialyzed against distilled water at 37 °C for 0–150 min. The glucose assay kit KGLUC (Megazyme, Wicklow, Ireland) was used to evaluate the glucose content in the dialysate.

2.6. Evaluation of the In Vitro Hypolipidemic Properties

2.6.1. Cholesterol Binding Capacity

Fresh egg yolks were diluted (1:10) using Milli-Q water and emulsified [19]. WIRs and flours (1 g) were mixed with diluted egg yolk (25 mL), and the pH was adjusted to 2.0 and 7.0. The flasks were incubated (37 °C, 2 h) with oscillation and centrifuged at 4000× *g* for 15 min. The supernatant was diluted with acetic acid (90% *v*/*v*), mixed with *o*-phthalaldehyde and 96% H_2SO_4, and incubated under continuous stirring (60 °C, 30 min). The absorbance was measured at 550 nm. Pure cholesterol was used as a standard.

2.6.2. Bile Salts Binding Capacity

WIRs and flours (0.25 g) were combined with 50 mg of sodium cholate (Sigma-Aldrich, MO, USA) and 25 mL of NaCl solution (0.15 M, pH 7.0) [19]. The final solution was held on the shaker for 1–3 h, all at 37 °C, and centrifuged at 4000× *g* for 20 min. The absorption capacity was calculated as the percentage of absorbed sodium cholate. Samples

were mixed with H_2SO_4 (45% *v*/*v*) and furfural (0.3% *v*/*v*) and incubated at 65 °C for 30 min. Absorbance was measured at 620 nm. Sodium cholate was used as a standard.

2.6.3. In Vitro Pancreatic Lipase Inhibition

A mixture solution was formulated, adding 0.1 g of flours or WIRs, 10 mL of sodium phosphate buffer (0.1 M, pH 7.2), 2 mL of olive oil, and 2 mL of pancreatic lipase solution (0.75 mg pancreatic lipase/mL phosphate buffer). Samples were incubated at 37 °C for 1 h [19]. The content of free fatty acid released was determined by titrating with NaOH (0.05 M). A similar method was carried out by adding bile salts (156.5 mg/mL) to evaluate the bile salts-binding effect on lipase activity. Lipase inhibitory activity (%) was defined as the percentage decrease in the free fatty acid production rate over the control.

2.7. Statistical Analysis

Each sample was analyzed in triplicate ($n = 3$). Data were reported as mean ± standard deviation (SD). The data were analyzed using the *t*-test or one-way analysis of variance (ANOVA) and post hoc Tukey test. Relationships between the analyzed parameters were evaluated by computing Pearson linear correlation coefficients setting the level of significance at $p < 0.05$, $p < 0.01$, and $p < 0.001$ [20]. The statistical analysis was performed by SPSS 24.0. Multivariate analyses were performed with XLSTAT 2020 for Microsoft Excel 2016.

3. Results and Discussion

3.1. Coffee Cascaras Are a Source of Insoluble Dietary Fiber and Phenolic Compounds

IDF proved to be the main fraction in all the samples studied, the WIRs of both dry and wet cascara showed a significantly higher IDF content ($p < 0.05$) than their corresponding flours. The samples presented SDF to a lower extent. Soluble dietary fiber was 2.2 to 3.6-fold higher ($p < 0.05$) in flours than in WIRs. The content of phenolic compounds ranged from 6.1 to 40.4 mg gallic acid equivalents per gram. The antioxidant capacity showed a similar trend to the phenolic content, being higher in the dry cascaras, and lower in WIRs with regard to the corresponding flours. The wet coffee cascara flour exhibited a remarkable antioxidant capacity (2.4 to 4.2-fold higher than the other products). The higher antioxidant capacity and the higher content of phenolic compounds in the flours were expected since WIRs are the result of the aqueous extraction carried out to obtain an extract concentrated in hydrophilic phenolic compounds [6].

3.2. Techno-Functional and Physicochemical Properties

Regarding the techno-functional and physicochemical properties, the dry cascara showed significantly ($p < 0.05$) higher pH values than the wet one. The lower pH found in WIRs may be due to the solubilization of numerous compounds such as alkaloids and phenolic compounds acids during the extraction [21]. Furthermore, the WIRs showed a significantly ($p < 0.05$) lower BD than flours. On the other hand, the WAC of the WIRs was significantly higher ($p < 0.05$) than the flours, highlighting the properties of the wet cascara WIR (8.3 mL/g). Similarly, WHC, SWC and OHC were significantly higher ($p < 0.05$) in the WIRs than flours. The higher dietary fiber content, especially IDF, present in the WIRs could explain the differences observed between these technofunctional properties of the WIRs and flours [22]. However, WIRs showed significantly lower gelation capacity than flours ($p < 0.05$).

3.3. Hypoglycemic and Hypolipidemic Properties

All samples exhibited a decrease in glucose diffusion with respect to the positive control (no sample) over time. The wet cascara samples showed significantly ($p < 0.05$) higher α-amylase inhibition (91.9–96.5%) than that the dry cascara (62.9–73.2%), which was significantly correlated ($r = 0.965$, $p < 0.05$) with the different total phenolic content found in wet and dry samples. Antioxidant compounds such as polyphenols can inhibit enzymes

such as α-amylase due to the interactions of these compounds at the active sites of the enzymes through hydrophobic forces and hydrogen bridges [23]. Likewise, dietary fiber can inhibit α-amylase, the ability of IDF being greater than SDF. The mechanism by which dietary fiber inhibits the enzyme could be different depending on the type of dietary fiber since IDF seems to be a mixed type inhibitor, while SDF would be a non-competitive inhibitor [24,25]. All samples revealed starch digestibility retardation ability. Flours exhibited significantly ($p < 0.05$) higher retardation capacity compared to WIRs. α-amylase inhibition by dietary fiber could be the primary mechanism involved in the ability of the samples to decrease glucose production from starch, although dietary fiber might also trap starch and decrease starch digestibility [26]. Similarly, coffee cascara samples showed pancreatic lipase inhibitory properties (54–65%) and reduced the intestinal absorption of bile salts (81–90%) and cholesterol (50–88%) in vitro. At pH 2, the dry coffee cascara flour showed a significantly higher cholesterol-binding capacity ($p < 0.01$) with respect to its corresponding WIR, as opposed to the wet cascara flour. These properties have been mainly associated with the content of SDF, able to form viscous emulsions and block the absorption of lipids in the gut, but also with lignin, which may interact with bile salts [4].

4. Conclusions

In conclusion, both dry and wet coffee cascara exhibit a similar chemical composition and functional properties and could be revalued as new sustainable ingredients (flours and WIRs, and phenolic-rich extracts) following a biorefinery approach. These coffee cascara-based ingredients may exhibit beneficial health properties displaying antioxidant capacity and capacity to retard glucose and lipid absorption. The results from this work exhibit the great potential of these by-products to be used as dietary fiber-rich ingredients due to their high fiber content with antioxidant, hypoglycemic, and hypolipidemic properties, as well as suitable techno-functional properties. To date, we have validated the safe use of wet coffee cascara as a food ingredient [27]. Nonetheless, future in vitro, *in vivo*, and clinical studies are necessary to confirm the studies carried out and their possible toxicity and safety in humans.

Author Contributions: Conceptualization, M.R.-H., M.A.M.-C. and V.B.; methodology, M.R.-H., C.B. and S.C.; formal analysis, P.C.-M., M.R.-H. and V.B.; investigation, P.C.-M., M.R.-H., C.B. and S.C.; writing—original draft preparation, P.C.-M., M.R.-H., C.B. and S.C.; writing—review and editing, M.R.-H., C.B. and S.C.; visualization, A.G.-R. and Y.A.; supervision, M.R.-H., M.A.M.-C. and V.B.; funding acquisition, M.A.M.-C. All authors have read and agreed to the published version of the manuscript.

Funding: Please add: This research was funded by the COCARDIOLAC project from the Spanish Ministry of Science and Innovation (RTI 2018-097504-B-I00). M. Rebollo-Hernanz received funding from the FPU program of the Ministry of Universities for his predoctoral fellowship (FPU15/04238).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author on reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

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