EXPLORING THE PHYSICOCHEMICAL PROPERTIES OF RECOVERED INDUSTRIAL HEMP SEED (CANNABIS SATIVA L.) CAKE WASTE

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Abstract. *Hemp, scientifically known as Cannabis sativa L., is an annual plant belonging to the Cannabaceae family. Its historical utilisation by people spans various purposes, including textiles and food. Considered one of humanity's earliest cultivated plants, hemp has played a significant role in various civilisations. Traditionally, its fibres have been employed in producing ropes, apparels and fabrics, while its seeds have been valued for their nutritional richness. Hemp's health benefits are also associated with phenolic compounds, which occur in varying amounts in almost all classes of plant foods and agro-industrial residues. Hemp seed (Cannabis sativa L.) cake, derived from the cold-pressing process of hemp seed oil extraction, is abundant in protein, fibre and biologically active compounds. Three types of hemp seeds (Silvana, Armanca, Teodora) grown at the Lovrin Agricultural Research and Development Station were subsequently studied after oil extraction. The recovered hemp seed leftover waste cakes were analysed to compare their physical, chemical, nutritional composition, and total phenolic content. The ash, moisture, lipid, total protein, and crude fibre levels were measured for each variety. The results will enable further research to valorise secondary byproducts generated during the hemp seed oil extraction and contribute to reducing food waste, thereby advancing the sustainability of the agri-food systems.*

Keywords: *Cannabis sativa L, hemp seed cake, total phenolic, sustainability*

INTRODUCTION

Industrial *Cannabis sativa* L. plants with a low level of d-9-tetrahydrocannabinol (THC, <0.2%) are cultivated for extracting fiber, seeds, and their derivatives, including oil [FARINON B. 2022]. This crop holds significant agricultural value in Canada [OOMAH, B.D 2022] the USA [ALDEN, D.M 1998], and China [TANG, C.H 2006]. Hemp fiber is extensively used in contemporary paper and textile manufacturing in these countries. The seeds of Cannabis sativa are the edible part of the plant and are increasingly used to produce hemp seed-based food products, which are now widely available to consumers [STAMATIE, G.D 2022]. Roasted hemp seeds are sold in Chinese markets, while a significant portion of the seeds are exported as bird feed. In Eastern Europe, hemp seed oil is a viable substitute for butter, especially for those with limited access to dairy products [ANWAR, F 2006]. The environmental impact of food processing has been a long-standing concern. Recently, several strategies have emerged to manage organic waste. A common feature among these approaches is the reclassification of organic material from waste to valuable by-products. For example, in the oil production process, press cakes are left behind after oil extraction. These remnants are typically rich in protein, fiber, and bioactive substances [NAKOV, G]. This study aimed to repurpose waste from hemp seed processing following oil extraction. The physical, chemical, and nutritional composition, as well as the total phenolic content, of the residual hemp seed cakes, were analyzed and compared. Measurements were taken for ash, moisture, lipid, total protein, and crude fiber levels for each variety.

MATERIAL AND METHODS

The hemp seed varieties analyzed in this study were Silvana (HCS), Armanca (HCA), and Teodora (HCT). These varieties are authorized for cultivation in Romania and are listed in the EU Common Catalogue of Agricultural Plant Species [FOOD SAFETY] with a THC content of less than 0.2%. They were cultivated at the Lovrin Agricultural Research and Development Station located at 45°57′03″N 20°46′32″E.

PU10 cold-pressed oil press was used to extract oil from hemp seeds. The system is equipped with a special cold pressing mechanism and a single transport auger. The hemp seed cake was ground with a Grindomix GM200 mill and stored in polyethylene bags until used for feed formulations.

Determination of Proximate Composition and Energetical Values of Hemp Seed Cake

The proximate composition analysis was carried out using ISO methods. Moisture was determined using SR EN ISO 712:2010, lipid using SR EN ISO 11085:2016, protein using the Kjeldahl method according to Regulation 152/2009, ash using SR EN ISO 20483:2007, and fiber ISO 6492:1999. The carbohydrate content was calculated by subtracting the sum of moisture, lipids, proteins, and ash from 100%. The energy value was calculated following the method described by DAS P. C. et al. (2019). Equation (1) was used, taking into consideration that 1 gram of carbohydrates provides 4 calories, 1 gram of protein provides 4 calories, and 1 gram of fat provides 9 calories.

Energy value (kcal/100 g) = carbohydrates (%) × 4 + lipids (%) × 9 + proteins (%) × 4 (1)

Phytochemical Characteristics of Hemp Seed Cake

Preparation for the Alcoholic Extract

To weigh each sample, take 1 g and place it in a container with a lid. Add 10 mL of 70% ethanol (Chimreactiv, Bucharest, Romania). After securing the lid, the container was shaken for 30 minutes using a Holt mechanical shaker (Idl, Freising, Germany). The resulting extracts were then filtered through the Whatman filter paper.

Total Phenolic Content (TPC) determination:

The total phenolic content (TPC) was evaluated using the Folin-Ciocâlteu method [COCAN, I. 2022]. Each filtered extract (0.5 mL) was mixed with a Folin-Ciocâlteu reagent (Sigma-Aldrich Chemie GmbH, Munich, Germany) aqueous solution (distilled water) in a 1:10 (v/v) ratio. The mixture was kept at room temperature for 5 minutes before adding a 6% (w/v) aqueous solution of Na_2CO_3 (1 mL) to each sample. The extracts were placed in a thermostat (INB500, Memmert GmbH, Schwabach, Germany) at 50°C for 30 minutes. The absorbance was measured at 750 nm using the Specord 205 spectrophotometer (Analytik Jena Inc., Jena, Germany) against the blank, which was also prepared under similar conditions to the samples. A calibration curve was established by using standard gallic acid solutions (Fluka, Madrid, Spain) with concentrations ranging from 10 to 100 mg GAE/L. Total phenolic content (TPC) was expressed as mg gallic acid equivalents (GAE)/kg. Analyses were performed in triplicate and results were reported as mean value ± standard deviation (SD).

RESULTS AND DISCUSSION

Proximate Composition of hemp seed cake.

The results of moisture, ash, lipid and protein content of hemp seed cakes are presented in figures 1-4. Numerous researches have shown that hemp seed cake contains up to 50% protein substances, 9–20% lipids, 6–7% dietary fiber, significant amounts of minerals and can be used as feed or in food manufacturing [CAPCANARI T, 2023]. The samples' humidity varied between 6.73% in HCT and 7.42% in HCS, ash content ranged from 4.30% HCS, 5.48% HCT, and 5.86% HCA, with an average value of 5.22%, lipid content varied between 9.76% in HCA, 13.87% in HCS, and 14.73% in HCT. The protein content of the analyzed samples ranged from 27.30% HCT to 28.53% HCA.

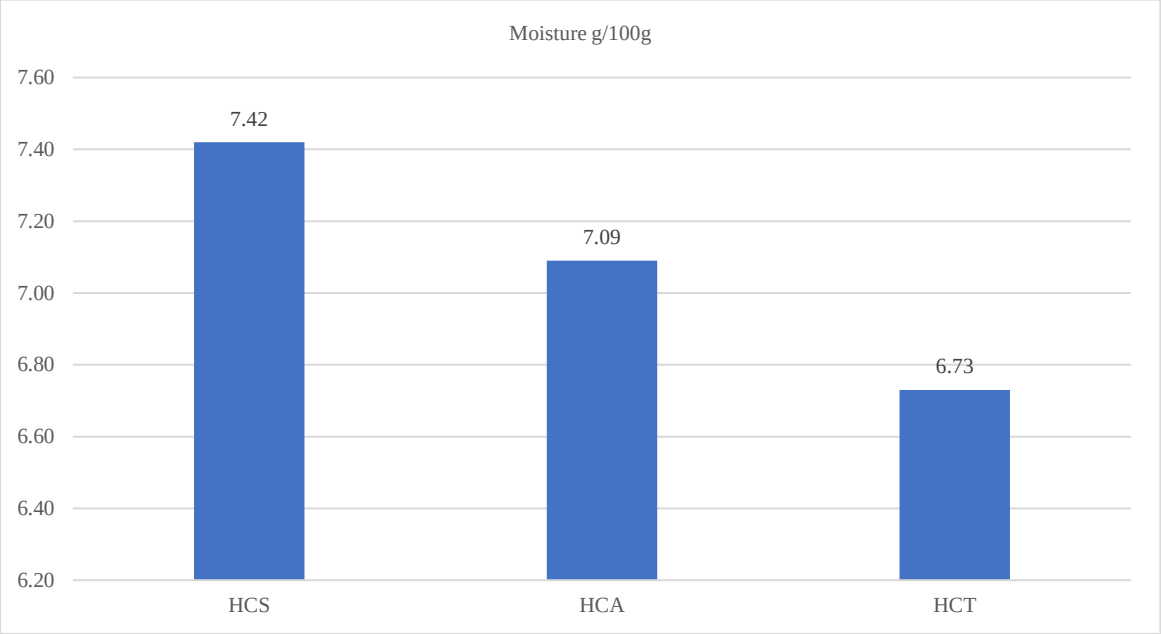

Figure 1. Moisture (g/100g) content for hemp seed cake samples

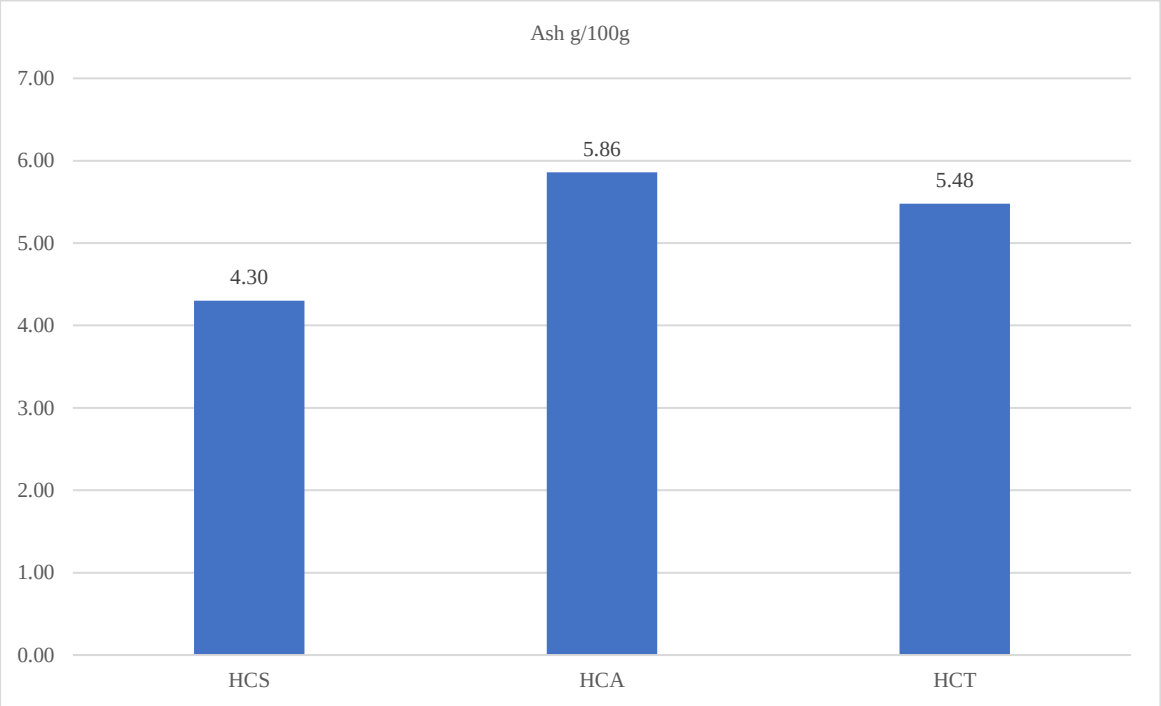

Figure 2. Ash (g/100g) content for hemp seed cake samples

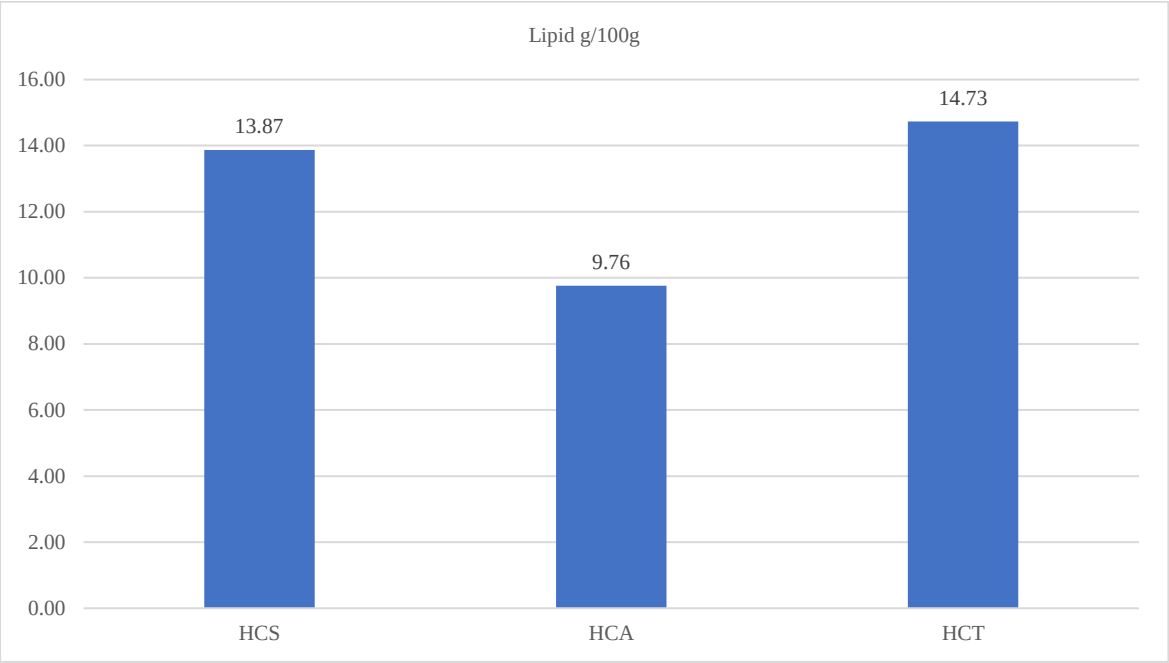

Figure 3. Lipid (g/100g) content for hemp seed cake samples

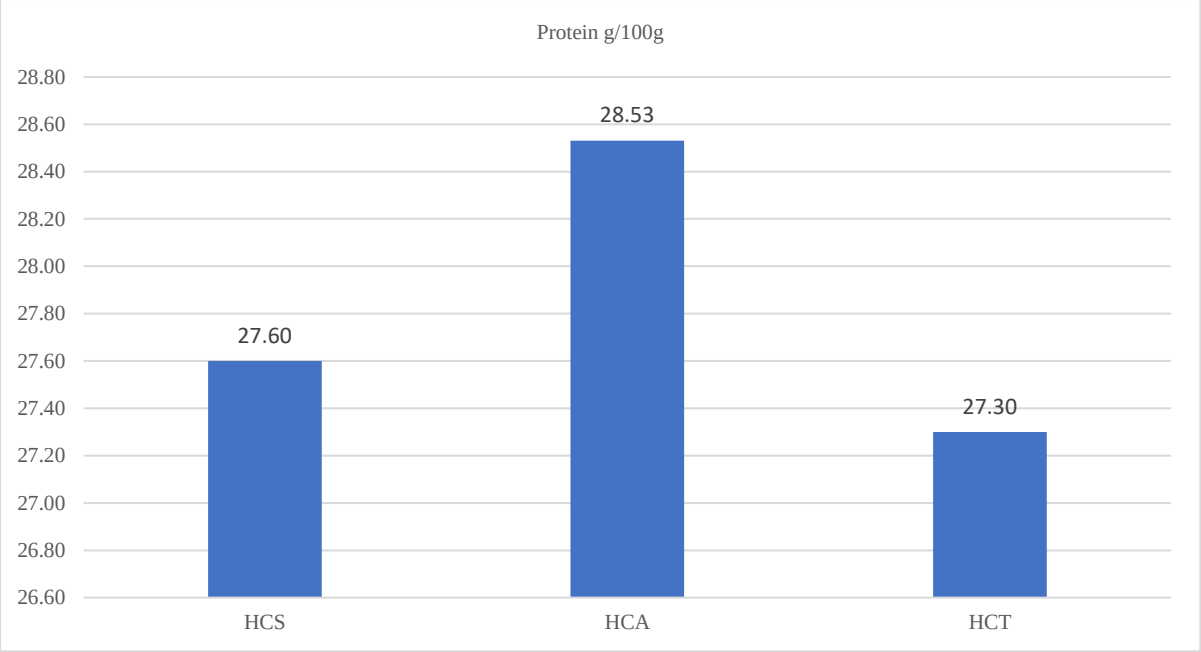

Figure 4. Protein (g/100g) content for hemp seed cake samples

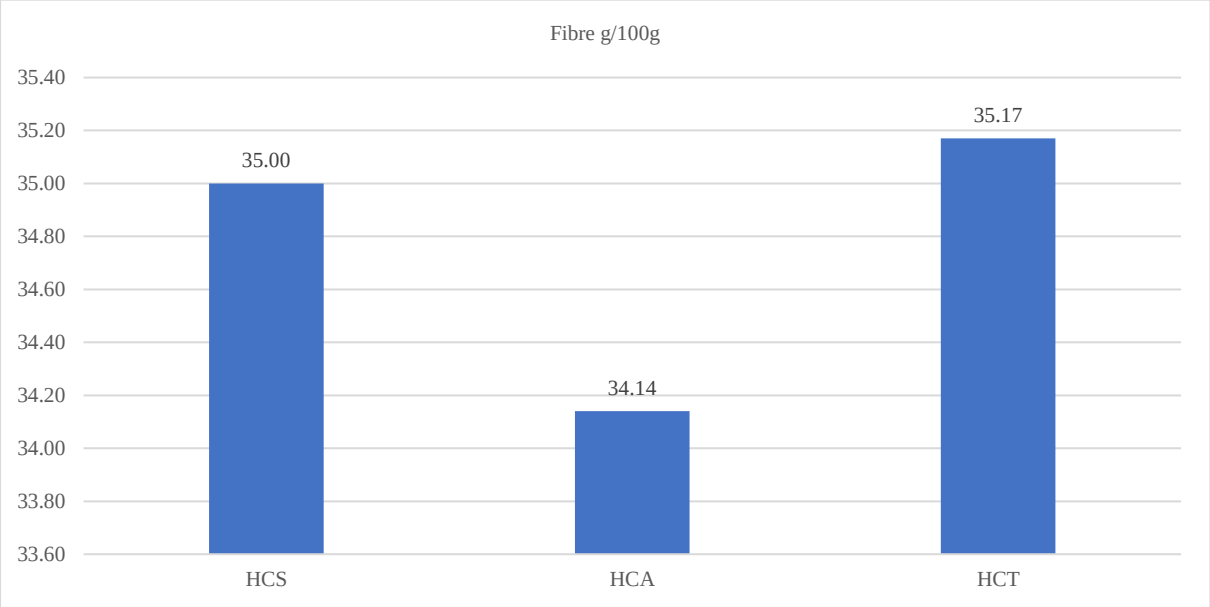

Figure 5. Fibre (g/100g) content for hemp seed cake samples

The sample contains 34.14% HCA, 35.00% HCS, and 35.17% HCT fiber.

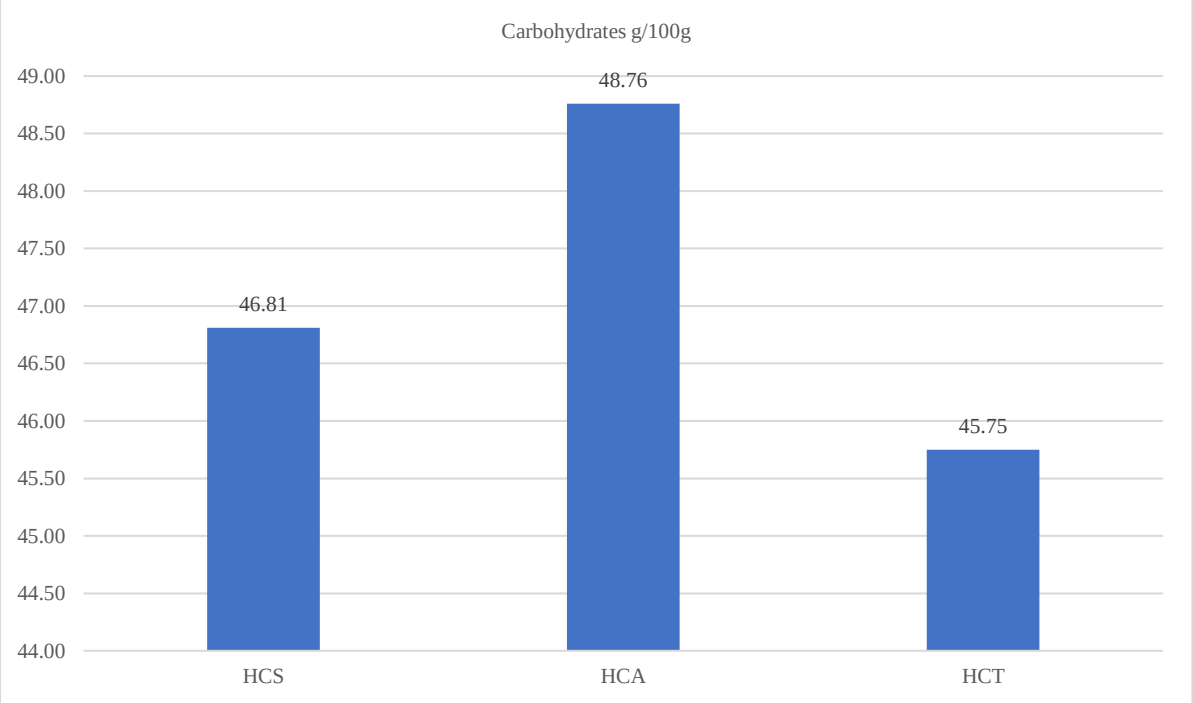

Figure 6. Carbohydrates (g/100g) content for hemp seed cake samples

The carbohydrate content in the analyzed samples varies between 45.75% HCT and 48.76% HCA.

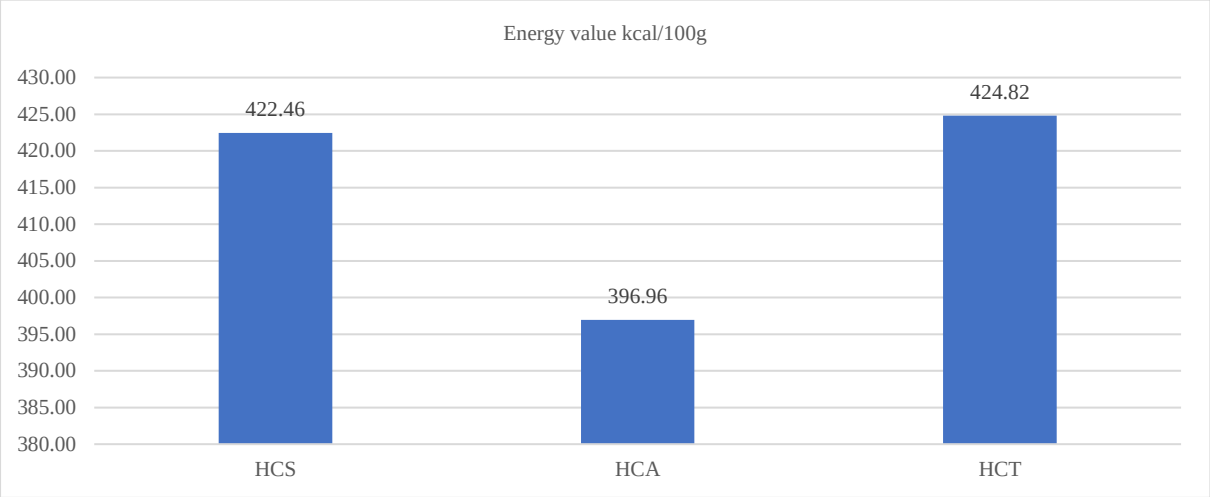

Figure 7. Energy value (kcal/100g) content for hemp seed cake samples

Following the calculations, the highest energy value belongs to probe HCT 424,82 kcal/100g, followed by HCS 422,46 kcal/100g and HCA 396.96 kcal/100g.

The proximate composition of the analyzed hemp seed cake is consistent with data reported in the specialized literature. Differences between the results of this study and the existing literature may be due to variations in geographical location, climate, local agronomic factors, and the analytical methods used. CAPCANARI, T et al. (2023) previously reported similar values. MIERLIȚĂ (2019) reported values of 32.06% and 31.22% for the protein content of hemp seed cake. TUFARELLI, V. et al. (2023) reported similar values for lipids (8.42%), ash (5.36%), and crude fiber (31.5%). ARANGO. S. et al (2022) reported similar values for ash (6.19 g/100g), lipids (8.70 g/100g), and crude protein (28.17 g/100g).

Figure 8 illustrates the total polyphenol content (TPC) in the analyzed hydroalcoholic extracts, measured in milligrams of gallic acid equivalent (GAE) per kg of sample, utilizing the UV spectrophotometric technique. Results indicate the mean value of three independent assays

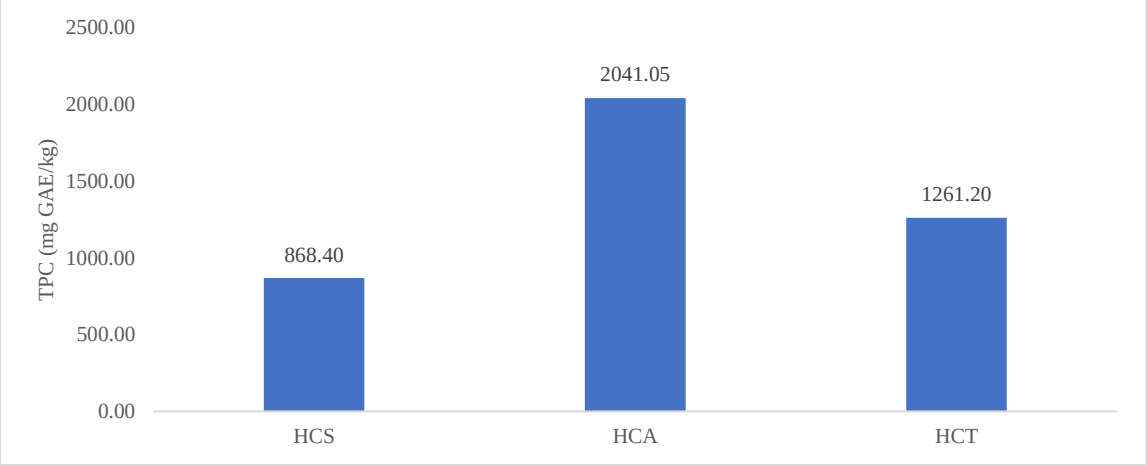

Figure 8. TPC content for hemp seed cake samples extracted

Based on the information provided, it is evident that the HCA hydroalcoholic extract exhibited the highest value at 2041.05 mg GAE/kg, while HCT showed a value of 1261.20 mg GAE/kg, and the lowest value was observed in HCS at 868.40 mg GAE/kg.

Other authors have previously examined the polyphenolic content of hemp seed cake samples, and their findings align with the values obtained in this investigation. OANCEA A.-G. et al (2023) reported a value of total polyphenols 2.874 (mg/g GAE).

CONCLUSIONS

Hemp seed cake is a by-product of oil extraction, commonly used as animal feed. However, its chemical composition and low cost make it an appealing ingredient for developing value-added products. The study's findings will facilitate further research into valorizing secondary by-products generated during hemp seed oil extraction, reducing food waste, and advancing the sustainability of agri-food systems.

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