U.P.B. Sci. Bull., Series B, Vol. 88, Iss.2, 2026 ISSN 1454-2331

# EFFECT OF HYDROTHERMAL TREATMENT ON SOME PROPERTIES OF SPENT COFFEE GROUND

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*This study investigates the effect of hydrothermal carbonization (HTC) process on the properties of liquid extracts resulted from spent coffee ground processing. Two samples, coded HTC-LE I and HTC-LE II were obtained by HTC at 220 °C, the ratio between biomass and distilled water of 1:10, generated autogenic pressure, and processing times, 1 h and 12 h, respectively. Physical-chemical properties such as pH, electrical conductivity, total dissolved solids, size dimensions and zeta potential, as well heavy metals and Na, K, Ca, and Mg ion metals were conducted at laboratory scale. Hydrothermal processing time conditions govern a dynamic equilibrium between metal release and immobilization pathways. Processing of spent coffee ground for 1 h favors solubilization and complexation, while 12 h treatment shifts the system toward stabilization and sequestration of metals within the solid hydrochar phase. Also, the antimicrobial property recommends the potential use of HTC liquid extracts in agriculture.*

**Keywords**: spent coffee ground, hydrothermal carbonization, physical-chemical characteristics, metal ions, antimicrobial potential

## 1. Introduction

Environmental pollution is one of the greatest challenges of the 21st century, driven primarily by the intensive use of fossil fuels, increasing waste generation, and excessive industrialization. In this context, renewable resources and plant biomass have emerged as viable solutions for mitigating environmental impact, offering both clean energy alternatives and opportunities for the efficient use of organic waste. Coffee is one of the most traded commodities worldwide, with

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a global annual production exceeding 10 million tons in 2019 [1]. According to the International Coffee Organization, 80% of the world's coffee production originates from ten countries, with Brazil alone producing approximately 2,859,502 tons annually.

The process of producing a cup of coffee is complex and generates several byproducts, including coffee husks, pulp, skin, and spent coffee grounds [2]. The growing amount of food waste contributes significantly to global greenhouse gas emissions, with an estimated 8–10% of total emissions attributed to food waste [3]. After decomposition in soil, spent coffee grounds can serve as an effective fertilizer, providing cellulose, hemicellulose, lignin, fats, ash, protein, and nitrogen to plants [4]. Spent coffee grounds are a valuable byproduct of coffee production generated by the instant coffee industry, households, restaurants, and coffee shops. Approximately 2 kg of coffee grounds are produced for every 1 kg of instant coffee manufactured [5],[6]. Various processing techniques have been investigated for the valorization of coffee grounds, including lipid extraction using green solvents [3], pyrolysis [7], hydrothermal carbonization (HTC) [8],[9], biorefining [10], melt processing [11], and torrefaction [12]. The value-added applications of spent coffee grounds include the production of solid fuel [5,13,14], biocatalysts [15], biopolymers [16], adsorbents for air and water purification [2], pellets [17], and extracts rich in bioactive molecules such as polyphenols and caffeine [1,18,19]. The hydrochar process water can be used in various ways, such as recycling to increase hydrochar yield and concentrate organic compounds for easier downstream processing [8,9]. In this context, the aim of this study is to investigate the liquid extract obtained from the autoclaving of spent coffee ground as a potential biostimulant for plants growth and to evaluate its antimicrobial potential by determining the minimum inhibitory concentrations (MICs) against microbial strains.

## 2. Experimental part

### 2.1. Materials

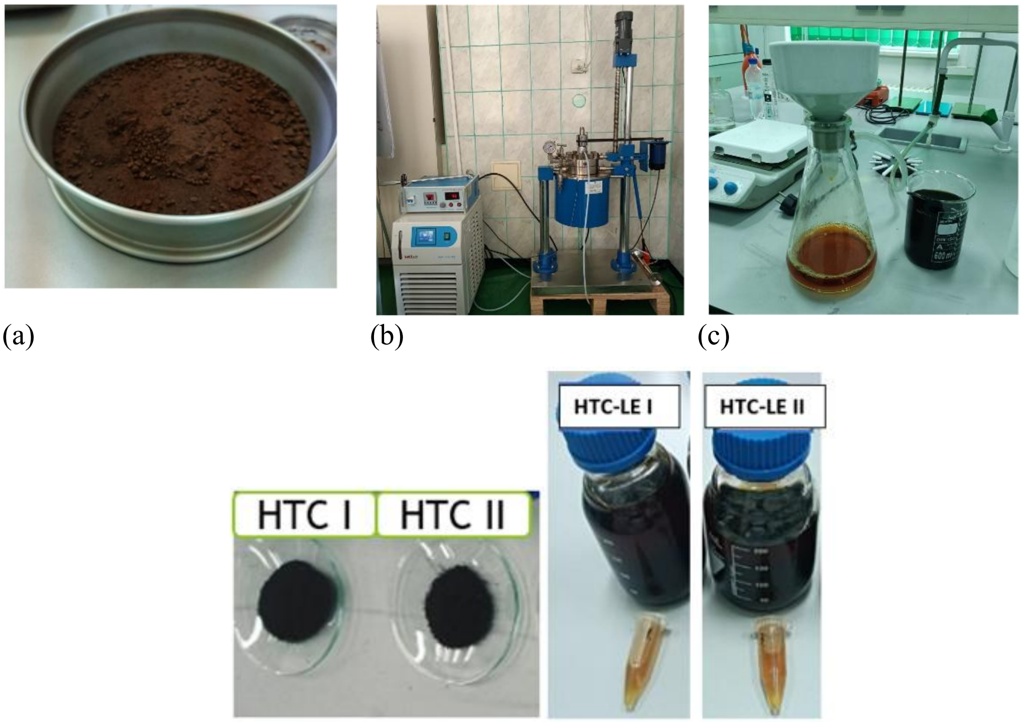
Spent coffee ground was used as received, collected from daily consumption. The sample was dried in an oven at a temperature of approximately 80 °C for 4 hours. The particles size distribution of the coffee ground was determined by sieve analysis using sieves with pore sizes of 1 mm, 500 µm, 90 µm, 45 µm, and 32 µm. The results of the particles size analysis indicate that the predominant fraction, which was selected for thermal autoclaving, corresponds to 90 µm.

A multielement standard solution (ME), with a concentration of 1000 ppm was used for preparing standard solutions for the calibration curve. Nitric acid ($HNO_3$), peroxide of hydrogen ($H_2O_2$) of analytical grade were used for digestion of water samples. An acidic solution (0.5% $HNO_3$ in distilled water) was used as reference and tube washing of spectrometer.

## 2.2. Methods

### *2.2.1. Hydrothermal carbonization (HTC)*

Hydrothermal carbonization of the coffee ground was performed at a temperature of 120 °C, using a coffee grounds-to-solvent ratio of 1:10, contact times of 1 h and 12 h, respectively, and autogenic pressure. The liquid extracts coded as HTC-LE I and HTC-LE II were collected by vacuum filtration (using a 1.2 μm pore size filter) of the hydrothermally treated samples. The resulting biochar was subsequently washed thoroughly with distilled water and stored in sealed glass bottles in the fridge at a temperature of approximately 5 °C for further use. The laboratory processing of spent coffee ground is shown in Fig. 1.



(a) (b) (c)

(d) (e)

Fig. 1. Spent coffee ground (a), Hydrothermal carbonization equipment (b), Washing of the hydrochar achieved (c), Dried hydrochar (d), Liquid extracts resulted from the processing of spent coffee ground at 120 °C and 220 °C, respectively (e)

### *2.2.2. Physical-chemical characterization of liquid extracts (HTC-LE)*

The liquid extracts (HTC-LE) were characterized by measuring the following parameters: electrical conductivity (expressed in mS/cm), salinity, total dissolved solids (TDS) (expressed in g/L) (using a CONSORT C862 multi-parameter analyzer), and pH (using a CONSORT C831 multi-parameter analyzer).

Dimension of particles and zeta potential of liquid extracts were measured by using Zetasizer (Nano-ZSP, Malvern, UK).

### *2.2.3. Quantification of some metals*

The liquid extracts obtained from the hydrothermal autoclaving of spent coffee grounds were analyzed using flame atomic absorption spectrometry (ContraAA 800 D atomic absorption spectrometer, Analytic Jena, Germany) to determine heavy metals ($Cu^{2+}$, $Co^{2+}$, $Cd^{2+}$, total Cr, $Ni^{2+}$, total Fe, $Mn^{2+}$, $Pb^{2+}$, $Zn^{2+}$) and some alkali and alkaline earth metals ($Na^{+}$, $K^{+}$, $Ca^{2+}$, $Mg^{2+}$) (Fig. 2). The samples were digested to remove the organic matter. 30 mL from each liquid extract samples were digested using a BERGHOF Microwave Digestion System with Built-in, Non-Contact Temperature Measurements (Berghof Products + Instruments GmbH Labor-Technik, Eningen, Germany) in a mixture of 5 mL concentrated $HNO_3$ and 2 mL $H_2O_2$ with a concentration of 30%. Similar, a blank sample containing distilled water instead of HTC liquid extract was processed in the same way as analyzed samples.

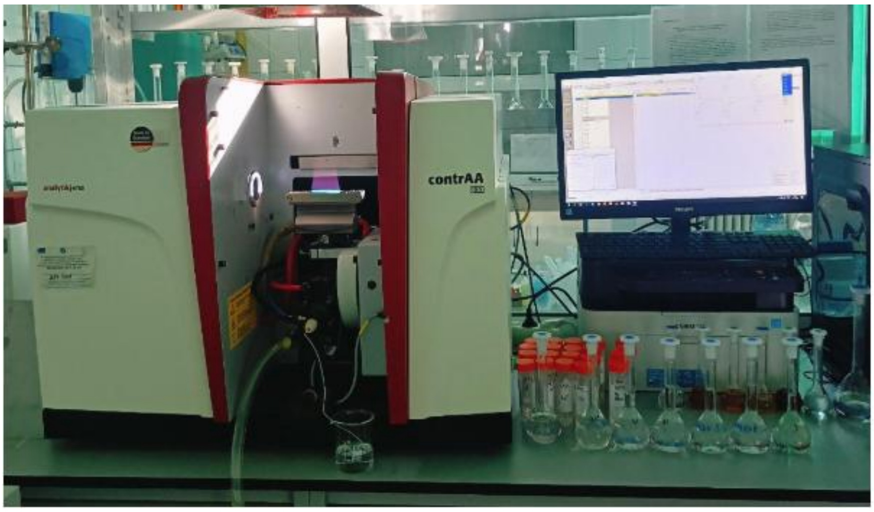


Fig. 2. ContraAA 800 D atomic absorption spectrometer, Analytic Jena

### *2.2.4. Evaluation of the antimicrobial activity of the HTC liquid extracts*

Evaluation of the compounds' antimicrobial activity was performed using standard strains: *Staphylococcus aureus* 25923 ATCC, *Enterococcus faecalis* 29212 ATCC, *Escherichia coli* 8739 ATCC, and *Pseudomonas aeruginosa* 27853 ATCC. In 96-well plates, serial binary dilutions of the test solutions were performed, $10^6$ CFU/mL (colonies forming units) microbial suspension was added and incubated for 24 hours at 37 °C. The Minimal Inhibition Concentration (MIC) was determined as the lowest concentration that inhibit bacterial growth compared to positive control (bacterial strain without test compounds).

## 3. Results

### 3.1. Physical-chemical properties of HTC-liquid extracts

Table 1

**Some physical-chemical parameters of HTC liquid extracts**

| | pH value | Electrical conductivity, mS/cm | TDS, g/L | Salinity |
|---|---|---|---|---|
| HTC-LE I | 4.33 | 2.31 | 1.24 | 1.1 |
| HTC-LE II | 4.62 | 2.40 | 1.28 | 1.1 |

The HTC-LE I and HTC-LE II show relatively similar physical-chemical properties, indicating that the hydrothermal treatment time (1 h vs. 12 h) did not lead to major differences in bulk solution characteristics (Table 1). This suggests that most extractable soluble species are already released within the shorter processing time. Fig. 3 and Table 2 show the size dimensions and the parameters recorded from the zetasizer analysis (the size of maximum peak, its intensity, polydispersion index (Pdi), and zeta potential) of the investigated samples.

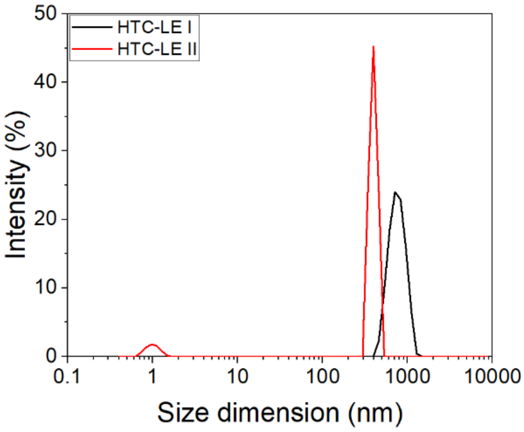


Fig. 3. Size dimension distribution for HTC-LE I and HTC-LE II samples

Table 2

**Zetasizer parameters recorded for HTC liquid extract samples**

| | Peak 1, nm | Intensity, % | Peak 2, nm | Intensity, % | Pdi | Zeta potential, mV |
|---|---|---|---|---|---|---|
| HTC-LE I | 762.8 ± 165.7 | 100 | - | - | 0.7 | −5.40 ± 4.37 |
| HTC-LE II | 397.2 ± 42.02 | 94.1 | 0.9996 ± 0.1739 | 5.9 | 0.951 | −5.49 ± 3.90 |

The HTC-LE I sample shows a single dominant population with a hydrodynamic diameter of 762.8 ± 165.7 nm (100% intensity), suggesting the presence of relatively large and possibly aggregated particles or macromolecular clusters. The polydispersity index (Pdi) of 0.7 indicates a moderately broad to broad size distribution, consistent with a heterogeneous colloidal system. The zeta potential of −5.4 ± 4.37 mV suggests a very low surface charge and therefore poor electrostatic stability, implying a tendency toward aggregation or limited dispersion stability (Table 2).

The HTC-LE II sample shows a bimodal distribution, with a main peak at 397.2 ± 42.0 nm (94.1% intensity) and a second minor population at 0.9996 ± 0.1739 nm (5.9% intensity). The presence of a smaller particle fraction suggests that the longer hydrothermal treatment promotes further breakdown of larger structures into smaller colloidal species or soluble molecular aggregates (Table 2). However, the Pdi of 0.951 indicates a highly polydisperse system, reflecting a very heterogeneous mixture of particle sizes and possibly overlapping populations of nanoparticles, dissolved macromolecules, and small aggregates.

### 3.2. Atomic absorption spectrometry (AAS) analysis

The profile of heavy metals presented in liquid extracts, after digested protocol, was investigated using atomic absorption spectroscopy (AAS). Also, qualitative analysis of K, Na, Ca, and Mg was performed using a multielement standard solution. Fig. 4 (a, b) shows the qualitative and quantitative analysis of heavy metals found in the HTC liquid extracts compared with black and a multielement solution of concentration 0.5 mg/L.

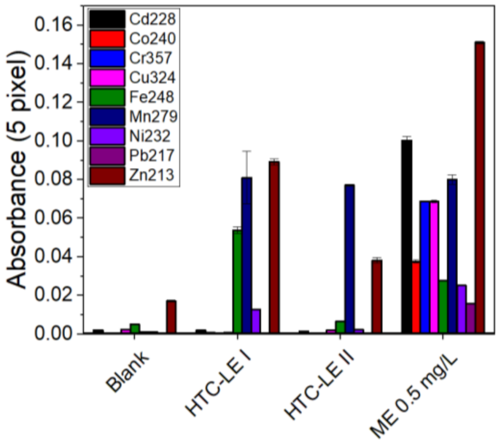


Fig. 4. (a) Atomic absorption spectra recorded for the HTC liquid extracts compared with blank and standard multielement solution (ME): Qualitative

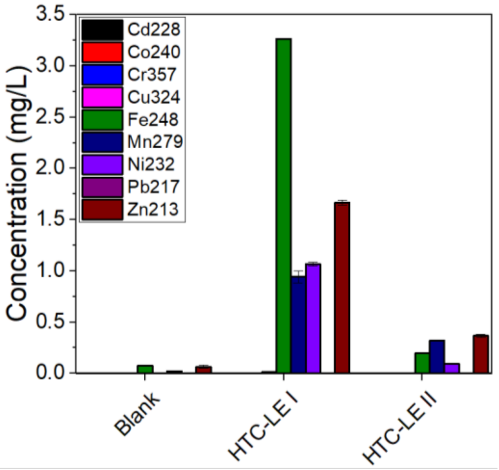


Fig. 4. (b) Atomic absorption spectra recorded for the HTC liquid extracts compared with blank and standard multielement solution (ME): Quantitative

Qualitative analysis reveals that the HTC-LE I exhibits, generally, higher absorbance values compared to HTC-LE II, particularly at wavelengths associated with Fe (248 nm), Mn (279 nm), Zn (213 nm), and Ni (232 nm) (Fig. 4a). This suggests that the shorter hydrothermal treatment may result in a higher concentration of soluble or complexing species capable of interacting with these metal-associated signals. However, only in the case of Fe, the absorbance of HTC-LE I exceeded that recorded for ME 0.5 mg/L.

Quantitative analysis shows 3.188 ± 0.0074 mg/L Fe for HTC-LE I, 0.318 ± 0.026 mg/L Mn for HTC-LE II, 1.041 ± 0.0208 mg/L Ni for HTC-LE I, and 1.6013 ± 0.024 mg/L Zn for HTC-LE I (Fig. 4b). HTC-LE I sample indicates a strong extraction efficiency during the shorter hydrothermal treatment, which favors the release of metal ions or metal-complexing species from the coffee grounds matrix. The rest of elements were detected at low concentrations or were below the detection limit. Similar, Campbell [20] quantified the heavy metals using the liquid resulted from the processing of spent coffee grounds by the hydrothermal carbonization process conducted 5 h at 200 °C, with a 1:9 the ratio between biomass: deionized water. The authors found concentrations of 0.34 mg/L Fe, 0.36 mg/L Mn, 3.09 mg/L Zn, and 0.02 mg/L Ni.

The observed differences between HTC-LE I and HTC-LE II can be mechanistically explained by the treatment duration on matrix degradation, metal mobility, and secondary phase formation [21]. Under shorter hydrothermal conditions (HTC-LE I), partial disruption of the spent coffee ground structure enhances the release of metal ions and soluble metal–organic complexes into the liquid phase. This results in higher measured concentrations of heavy metals and increased absorbance at metal-associated wavelengths, likely due to the presence of soluble ligands (e.g., organic acids and partially degraded polyphenols) that stabilize metals in solution.

In contrast, the extended hydrothermal time condition to 12 h (HTC-LE II) promoted further degradation of organic matter and transformation of dissolved species, leading to a reduction in detectable metal concentrations in the liquid phase. This may be attributed to several competing processes, including re-adsorption of metal ions onto the developing hydrochar surface rich in oxygen-containing functional groups, precipitation of less soluble phases, and incorporation of metals into more stable carbonaceous or mineral-organic structures [22]. Fig. 5 shows the presence of Na, K, Ca, and Mg ions in HTC liquid extracts compared with blank and ME samples.

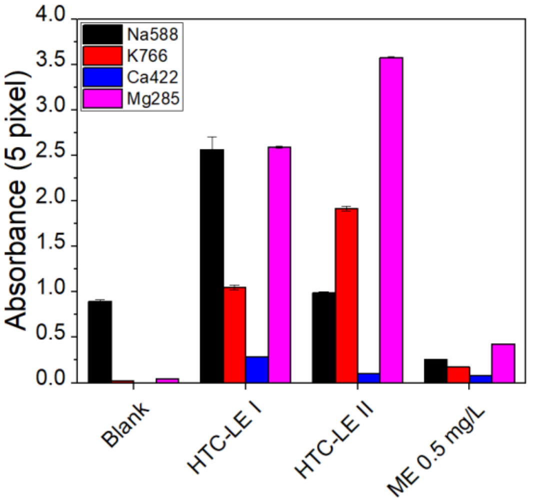


Fig. 5. Qualitative analysis of the main nutrients absorbed by the HTC liquid extracts

Following the qualitative analysis of the presence of Na, K, Ca, and Mg ions in the liquids obtained from coffee grounds processing, a higher signal intensity for Na and Ca is observed after 1 hour of treatment, while processing for 12 hours leads to a more pronounced release of Mg and K. These qualitative results are consistent with the values obtained in the TDS test, suggesting a correlation between processing time and the total amount of dissolved minerals.

### 3.3. Microbiological assay

The antimicrobial activity of the liquid extracts showed that the inhibitory concentrations are 25-50%, varying among the bacterial strains and extraction methods (Table 3). For *E. faecalis* and *P. aeruginosa* strains here is no difference of inhibition between the two tested extracts. In the case of *S. aureus,* the most effective sample is HTC-LE I, whereas, in the case of *E. coli,* a 25% solution of HTC-LE II inhibited bacterial growth (Table 3).

For Gram negative bacteria (*E. coli, P. aeruginosa)*, the best inhibition was obtained using the extract obtained after the 12 hours treatment (HTC-LE II). The high concentrations required to inhibit bacterial growth recommend the use of these extracts for further testing to determine their effects as biostimulants on plant growth.

Table 3

**Minimal inhibition concentrations (%)**

| Strain<br>Compound | *Staphylococcus aureus* | *Enterococcus faecalis* | *Escherichia coli* | *Pseudomonas aeruginosa* |
|---|---|---|---|---|
| HTC-LE I | 25% | 50% | 50% | 25% |
| HTC-LE II | 50% | 50% | 25% | 25% |

## 4. Conclusions

The results suggested that hydrothermal processing time influenced the chemical composition of spent coffee ground liquid extracts, which in turn affects their antimicrobial activity and interaction with metal-associated species or their spectral response profile. Analysis of the liquid extracts revealed the presence of Fe, Mn, Ni, Zn, Na, K, Ca, and Mg ions. These results highlight the need for further characterization to evaluate the potential of these liquid extracts as fertilizers and sources of bioactive compounds. Also, next studies are necessary to evaluate the concentrations of Na, K, Ca, and Mg from liquid extracts and their effect on the plant growth.

**Acknowledgements**

This work was supported by a grant of the Ministry of Research, Innovation and Digitalization, CNCS—UEFISCDI, project number 83PCE/2025 under PNCDI IV.

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