

The effect of biochar obtained from waste filter coffee grounds on plant germination

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Article History

Received: June 19, 2024

Revised: August 1, 2024

Accepted: August 4, 2024

Published Online : September 15, 2024

Article Info

Article Type: Research Article

Article Subject: Environmental Engineering

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Available at

<https://dergipark.org.tr/jaefs/issue/86361/1502611>



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Abstract

Nowadays, coffee consumption is quite high, and the consumption of filter coffee is steadily increasing. Consequently, there is a significant increase in waste filter coffee. This study aims to evaluate waste filter coffee grounds using a zero-waste approach. In this context, the solid product of pyrolyzed waste filter coffee grounds was added to the soil in specific ratios to improve soil quality and increase yield. The effects on the root and stem development of arugula (*Eruca vesicaria*) and garden cress (*Lepidium sativum*) plants were investigated. Waste filter coffee grounds was homogeneously mixed with soil at application rates of 1, 2, and 4 tons/ha. The results of the study observed that the pyrolysis solid product positively affected plant growth. Comparing the data, the highest yield in plants was observed in soil with added biochar, while lower yields were seen in soil with added raw waste filter coffee grounds, and the lowest yield was found in soil without biochar. Among the soils with added biochar, the most significant root and stem development was observed in plants with 2 tons/ha of added biochar.

Keywords: Waste Filter Coffee, Plant Growth, Biochar, Pyrolysis, Soil Improvement

Cite this article as: Ciner, M.N., Elmaslar Özbaş, E., Savuk, H., Günay, Ş., Özcan, H.K., Öngen, A. (2024). The Effect of Biochar Obtained from Waste Filter Coffee Grounds on Plant Germination. *International Journal of Agriculture, Environment and Food Sciences*, 8(3): 561-570. <https://doi.org/10.31015/jaefs.2024.3.9>

INTRODUCTION

Biomass is a renewable, naturally occurring material that stores solar energy as chemical energy through photosynthesis. Biomass consists of polymers composed of macromolecules containing C-C bonds. The main skeleton of these polymers is formed by C-C bonds, but they may also include bonds involving C-O, C-H, C-N, C-S, or other elements. The polymers that make up biomass are generally macromolecular materials consisting of large structures formed from smaller units (Wang et al., 2017).

Biomass sources that can be used for energy production can be classified into plant-based sources, animal waste, and urban and industrial waste. These sources can be listed as plant and agricultural residues (branches, stems, straw, roots, bark, etc.), sugar and starch crops (potatoes, wheat, corn, sugar beetroot), oilseed crops (canola, sunflower, soybean, etc.), fibre plants (flax, hemp, sorghum, miscanthus, etc.), protein plants (peas, beans, etc.), and forest and wood waste (Greiner et al., 2019; Zardzewiały et al., 2023; Bijarchiyan et al., 2020).

Used coffee grounds contain a high amount of solid matter. Coffee beans include various health-related chemicals, such as phenolic compounds, melanoidins, diterpenes, xanthines, and carotenoids. Additionally, used coffee grounds contain significant amounts of organic compounds such as fatty acids, lignin, cellulose, hemicellulose, and other polysaccharides. As a solid waste, used coffee grounds, rich in both organic and inorganic compounds, are produced in substantial quantities. Therefore, the management of coffee waste, which must be handled before being released into the environment, is becoming increasingly important (Machado et al., 2011). According to the International Coffee Organization, coffee is one of the most consumed beverages in the world. According to the World Coffee Consumption Report 2018 prepared by the International Coffee Organization

(ICO); Coffee consumption in Turkey, which has increased by 13.2 percent in the last 5 years, has reached 93.9 thousand tons. The US revenue is at the top of the list with 1.5 million tons per year, followed by Brazil with 1.3 million tons and Japan with 465 thousand tons. (International Coffee Organization, 2019). Coffee consumption in Turkey increased to 1.1 kilograms in 2021 (Gastronomi Journal, 2021). During the production of espresso or instant coffee, a significant amount of solid residue known as spent coffee grounds is generated, ranging from 550-670 g/kg of coffee beans (Lane, 1983; Farah, 2009). Following the consumption of brewed coffee, approximately 6 million tons of waste from spent coffee grounds accumulate annually worldwide on filter papers (Hardgrove and Livesley, 2016).

Spent coffee grounds contain a high amount of solid matter. As with most biological raw materials, the composition of spent coffee grounds depends on various factors such as brewing method, germination conditions, and coffee type. However, most have a similar composition. The largest component of spent coffee grounds is polysaccharides, which make up about 50% of the dry mass of the spent coffee grounds. These polysaccharides include cellulose and hemicellulose. Spent coffee grounds are an organic waste material containing high amounts of sugars, fats, antioxidants, and other compounds, making them an important biodegradable component (McNutt, 2019).

The disposal of industrial and urban solid waste is a significant problem, and new methods have been developed for the reuse of raw materials to obtain useful products. Pyrolysis is one of the primary thermochemical conversion methods used to transform biomass into solid (char), liquid (bio-oil), and gaseous products (biogas), as well as valuable chemicals (Tophanecioğlu, 2009).

The use of biochar produced by the carbonization of biomass through pyrolysis in soil improves soil reclamation, carbon sequestration, and soil fertility (Akgul, 2017). The application of biochar in agriculture has become increasingly popular over the past decade, with the primary aim of enhancing soil properties and structure and contributing to the soil's carbon content. The characteristics of biochar are closely related to the quality of the biomass and the pyrolysis process used to produce it (Weber and Quicker, 2018). The porous structure of biochar contributes to sustainable soil fertility by improving the physical, chemical, and biological properties of soils (Zhang, 2021). The application of biochar to clay soils significantly increases soil aeration by enhancing macropores (Lehmann and Joseph, 2015). Additionally, the porous structure of biochar reduces nitrate leaching in sandy soils (Chen et al., 2020).

A study conducted in 2017 observed that the application of biochar to sandy soil increased total porosity and reported a significant increase in water holding capacity by 127% (Liu et al., 2017). Additionally, the highly porous structure of biochar increases the total pore number in the soil, creating a habitat for microorganisms and enabling the enzymatic functions necessary for plant growth to occur (Baïamonte et al., 2019). Studies have reported that biochar can increase the water holding capacity of soil (Villagra-Mendoza and Horn, 2018), while others have reported that it can decrease (Carvalho et al., 2016) or have no effect on water holding capacity. Biochar contains a high amount of carbon and may also include basic cations such as K, Ca, and Mg (Novak et al., 2019). Due to its basic nature, biochar acts as a liming agent by increasing the pH in acidic soils and is recognized as a soil pH regulator (Li et al., 2019).

Historically, filter coffee has been a widely consumed beverage in most countries. Its consumption has steadily increased, and the resulting coffee grounds are typically discarded directly. This practice has raised economic and environmental costs. Therefore, alternative methods for utilizing waste filter coffee grounds are necessary. This study investigates the recovery of biochar from waste filter coffee grounds through pyrolysis and its effect as a soil improvement on plant growth. In the initial stage, biochar was produced through the pyrolysis of biomass. The effects of the obtained biochar on plant growth were assessed through pot experiments.

MATERIALS AND METHODS

Preparation of Biochar

Biochar was obtained from waste filter coffee grounds by pyrolysis methods, and the properties of raw waste filter coffee and the produced biochar were examined.

Pyrolysis of Waste Filter Coffee

For the pyrolysis process, waste filter coffee samples were initially dried in an oven at 50°C. The dried samples (50 grams) were brought to a constant weight and then placed in a fixed bed steel reactor with an inner diameter of 8 cm and a volume of approximately 3 liters. The system's heating rate was regulated via an electrical panel, and rock wool was utilized to minimize heat loss. To maintain an inert atmosphere during pyrolysis, nitrogen gas (N₂) was introduced at a flow rate of approximately 1.5 L/min. The pyrolysis was conducted for 4 hours. To prevent gas leakage from the reactor, pure graphite and graphite-lead spiral gaskets were employed between the reactor and its lid.

The reactor was equipped with a gas inlet line for nitrogen gas supply and an outlet line in the lid area to evacuate the synthesis gas produced. The internal temperature of the reactor was measured using a thermocouple. Two consecutive cooling columns were utilized to remove condensable organic volatiles from the synthesis gases, with the condensed liquids being collected in conical flasks attached to the columns. The synthesis gas passing

through the cooling columns was directed to a gas analyzer to determine its composition. To protect the analyzer from uncondensed volatile organic substances and particulate matter, one ceramic and two microfiber filters were used in the pipeline. The gas analyzer measured the volumes of CO, CO₂, H₂, CH₄, and O₂ in the released gases. Pyrolysis studies were performed at reactor temperatures of 400, 500, and 600°C under laboratory conditions with a heating rate of 5°C/min. The experimental setup used for the pyrolysis is shown in Figure 1.

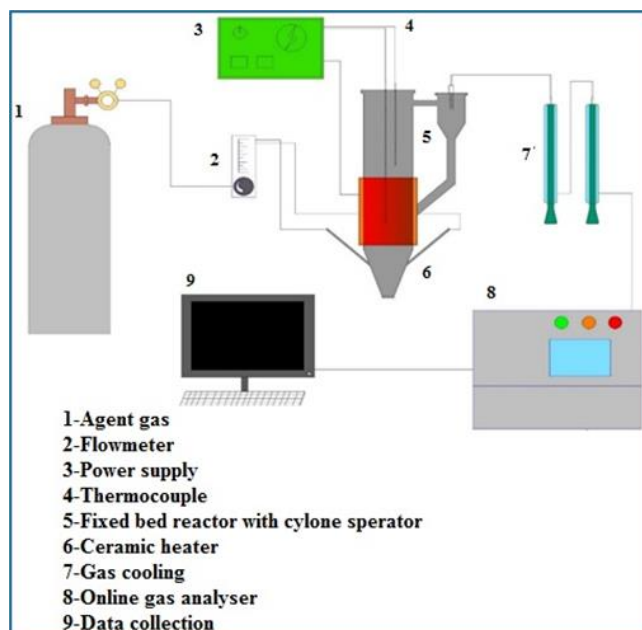


Figure 1. The experimental setup used for the pyrolysis.

Characterization Studies

Physical and chemical analyzes were carried out for biochar obtained as a result of pyrolysis and thermally activated biochar after the pyrolysis process. All analyzes were performed according to ASTM standards. The list of technical standards used for characterization is given in Table 1. Three technical replicates were performed for each analysis to minimize error.

Table 1. Technical standards for characterization.

Technical Standards	Analysis type
ASTM D-3173	Determination of Moisture Content and Solids Content
ASTM D-3174	Determination of Loss on Burning and Ash Content
ASTM D-5865	Determination of Calorific (Thermal) Value
ASTM D-5373	Elemental analysis
ATSM E-1252	Fourier Transform Infrared Spectrometer (FTIR)

Determination of Moisture Content and Solids Content

Total moisture and solids content of solid fuels are measured according to the oven drying method. This is achieved by placing the samples in a crucible and drying at 105°C for approximately 1 hour until constant mass is achieved. Then, the percent moisture content and solid content of the sample are calculated by equations (1) and (2), respectively. This analysis was carried out using a Memmert brand oven.

$$\text{Weight Moisture (\%)} = \frac{(A-B)}{A} \times 100 \quad (1)$$

$$\text{Weight Solids (\%)} = \left(\frac{B}{A}\right) \times 100 \quad (2)$$

Here,

A= First test sample, g

B= Dried test sample, g

Determination of Loss on Burning and Ash Content

Combustion loss analysis involves examining and evaluating the losses that occur during the combustion process in various systems such as engines, power plants, furnaces, or industrial processes. This analysis includes measuring and understanding energy losses, efficiency, and emissions related to combustion. Determining ash content is also a common analytical procedure used to measure the total mineral content in a sample. Ash is the inorganic residue remaining after the complete combustion of the sample's organic components, providing an estimate of the total mineral or inorganic content. In this analysis, combustion loss and ash content are determined using a muffle furnace. Samples are placed in a ceramic crucible and heated in an oven at 550°C for 120 minutes; then, the oven is cooled to 105°C. Finally, ash content and combustion loss are calculated using Equations (3) and (4). This analysis was conducted using a Magma Therm Primary MTP Series Model No. MTP-1000-8-P Furnace.

$$\text{Weight Loss on Combustion (\%)} = \frac{(A-B)}{A} \times 100 \quad (3)$$

$$\text{Weight Ash Content (\%)} = \left(\frac{B}{A}\right) \times 100 \quad (4)$$

Here,

A= First test sample, g

B= Dried test sample, g

Determination of Calorific Value

Calorific value analysis, also known as energy content analysis, is a process used to determine the amount of heat energy released when a substance burns. It is a crucial parameter for various applications, particularly in energy production and combustion. In this study, the process was conducted using the IKA C200 bomb calorimeter, which measures the higher heating value (HHV) of solid samples. The analysis follows the IKA guideline, describing the determination of the HHV of a solid biofuel in a bomb calorimeter at constant volume and a reference temperature of 25°C. Fuel samples are burned in a closed vessel with a pressurized (30 bar) oxygen-rich atmosphere, immersed in a known amount of water. The sample is placed in a quartz crucible within a plastic bag and ignited using a cotton thread connected to an electrode. The increase in the water temperature in the calorimeter system (ΔT) due to combustion is measured, and the HHV is evaluated on a wet basis.

Elemental Analysis

This method provides information about the elemental composition of samples, including the elements carbon (C), hydrogen (H), and nitrogen (N). The final analysis determines the percentage of these major elements (C, H, N) in the samples. The analysis is often conducted using techniques such as combustion analysis or instrumental methods like elemental analyzers. In this study, elemental analyses were conducted at the Central Research Laboratory of Bursa Technical University.

Fourier Transform Infrared Spectrometer (FTIR)

FTIR (Fourier Transform Infrared Spectroscopy) is a technique used to analyze both organic and inorganic materials by examining their chemical bonds and composition (Nandiyanto et al., 2019; Lopes et al., 2018). This method utilizes the light absorption properties of a material, leveraging how different molecular compounds react to infrared light to determine the material's structure. FTIR works by using multiple different frequencies in the beam, as each frequency interacts uniquely with the material, allowing for accurate determination of the composition of an unknown substance. In this study, FTIR analysis was employed to determine the chemical characterization of the samples. The FTIR analyses were conducted at the Central Research Laboratory of Istanbul University-Cerrahpaşa.

SEM Analysis

Scanning electron microscope analyses were performed at Recep Tayyip Erdoğan University Central Research Laboratory.

Plant Germination Experiments

Commercial soil samples were mixed with the pyrolysis product biochar and non-pyrolyzed waste filter coffee grounds in various proportions, and the prepared mixtures were placed into pots. Polyethylene-coated plastic pots with a capacity of 1.5 kg of soil were used. Before sowing the seeds (arugula and garden cress), biochar and waste filter coffee grounds were homogeneously mixed with the soil at application rates corresponding to 1, 2, and 4 tons/ha, equating to 6, 12, and 24 g/pot. Before sowing the seeds, the pots containing the mixtures were conditioned by watering daily with distilled water for one week. Subsequently, arugula and garden cress seeds were sown into each pot. At least three pots were used for each mixture to obtain a sufficient number of samples. Additionally, a control group with soil samples without the addition of biochar or waste filter coffee grounds was prepared. After 20 days, measurements were taken of the germinated plants. After harvesting, the lengths and weights of the roots and stems of the plants were measured. Throughout the experiments, the plants were irrigated with tap water and periodically rotated.

RESULTS AND DISCUSSION

Moisture, Ash Amount, Solid Matter and Burning Loss Tests

The moisture, ash, and solid matter values presented in Table 2 indicate that the waste filter coffee grounds samples used in this study are suitable for the pyrolysis process.

The literature suggests that the moisture content of raw materials for thermal processes should be in the range of 5-35% (Demirbas, 2009; Belgiorno et al., 2003). The moisture content obtained in this study falls within this range (8.28%). Additionally, the solid matter content of 91.72% further indicates that the waste filter coffee grounds samples are suitable for the pyrolysis process.

The characteristics of the raw material used in the pyrolysis process significantly affect the efficiency of the process. Materials with high carbon and hydrogen content are the most suitable for pyrolysis. Therefore, to determine the suitability of waste filter coffee grounds samples for the pyrolysis process, calorific value analyses were conducted. The calorific value results of both the raw waste filter coffee grounds and the biochar obtained from the waste filter coffee grounds are presented in Table 2.

Table 2. Results of moisture, Ash amount, solid matter and burning loss tests and energy values of biochar after pyrolysis.

Sample	Moisture (%)	Ash amount (%)	Solid matter (%)	Combustion loss (%)	Energy value (cal/g)
Raw	8.28	98.08	91.72	98.08	5121
Pyrolysis product biochar at 400°C	2.67	84.38	97.33	84.38	7259
Pyrolysis product biochar at 500°C	2.13	94.5	97.87	94.5	6970
Pyrolysis product biochar at 600°C	3.34	82.7	96.66	82.7	6358

SEM Images

In the study, SEM images were taken to investigate the pore structure of biochars obtained through the pyrolysis method. To observe the changes in the pore structure after pyrolysis, SEM images of both the raw samples and the post-pyrolysis samples are presented in Figures 2-5.

When examining the SEM image of the raw waste filter coffee grounds, it is observed that the untreated raw waste filter coffee grounds has a relatively smooth, non-porous structure (Figure 2). As the pyrolysis temperature increases and the pyrolysis products are subsequently thermally activated, an increase in porosity is observed (Figures 3-5).

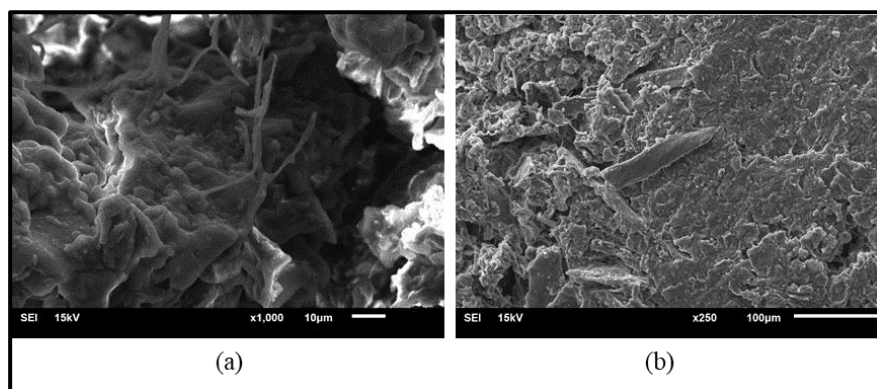


Figure 2. SEM images of raw waste filter coffee grounds a- magnified 1000 times b- magnified 250 times.

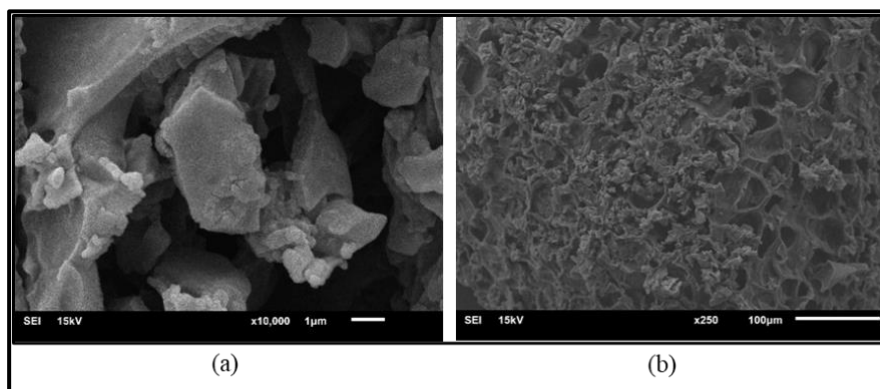


Figure 3. SEM images of biochar obtained from waste filter coffee grounds after pyrolysis at 400°C a- Magnified 10,000 times b- magnified 250 times.

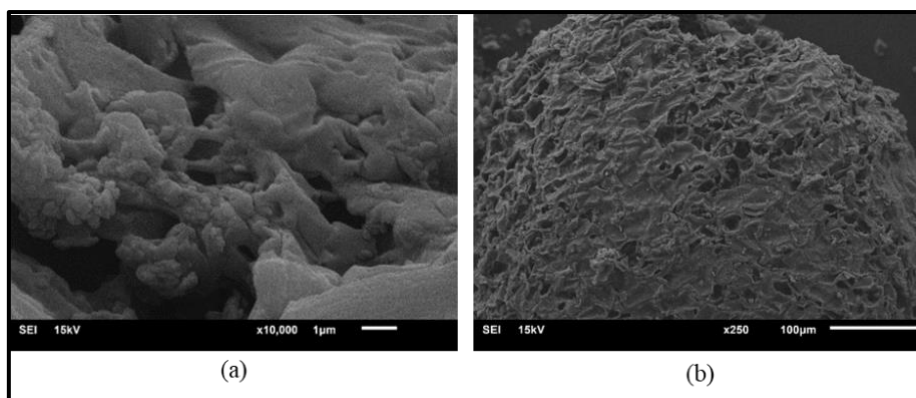


Figure 4. SEM images of biochar obtained from waste filter coffee grounds after pyrolysis at 500°C a- Magnified 10.000 times b- Magnified 250 times.

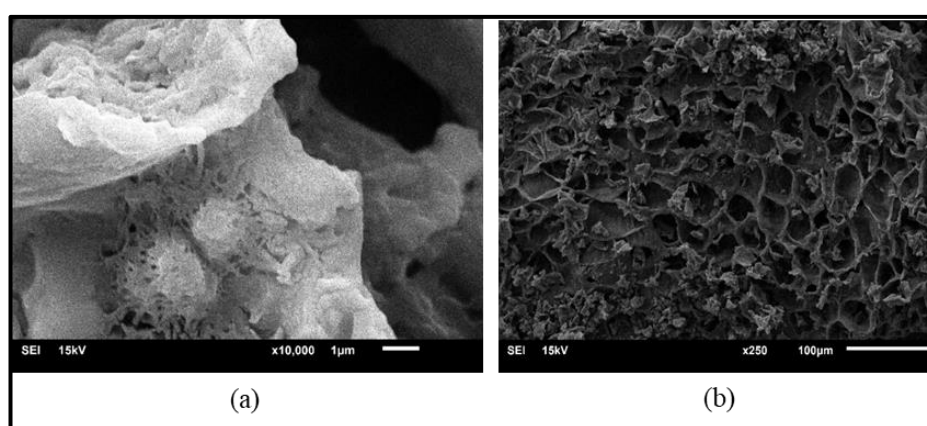


Figure 5. SEM images of biochar obtained from waste filter coffee grounds after pyrolysis at 600°C a- Magnified 10.000 times b- magnified 250 times.

C, H, N, S Elemental Analysis Results

Materials with high carbon and hydrogen content are the most suitable for pyrolysis. Therefore, an elemental analysis was conducted to determine the suitability of waste filter coffee grounds samples for the pyrolysis process. The elemental analysis results of both the raw waste filter coffee grounds and the biochar obtained from the waste filter coffee grounds are presented in Table 3. Upon examining the elemental analysis results, it was found that the raw waste filter coffee grounds sample contains 50.84% carbon (C) and 6.87% hydrogen (H). These values indicate that the waste filter coffee grounds samples are a suitable raw material for pyrolysis methods (Tsai et al., 2012).

Table 3. C, H, N Elemental analysis results.

Sample	C%	H%	N%
Waste filter coffee grounds	50.84	6.87	2.29
Biochar obtained by pyrolysis at 400°C	66.61	4.99	1.99
Biochar obtained by pyrolysis at 500°C	74.34	2.90	2.15
Biochar obtained by pyrolysis at 600°C	68.93	2.87	2.43

FTIR Analysis Results

Approximately 1-2% of waste filter coffee grounds content consists of caffeine. When examining Figure 6, the stretching vibrations observed at 2923 cm^{-1} and 2854 cm^{-1} are attributed to the caffeine in the coffee content. Additionally, 5-10% of coffee content is composed of chlorogenic acids. The presence of ester groups in these acids is evidenced by the bands at 1704 cm^{-1} and 1635 cm^{-1} in the spectra. Furthermore, the stretching vibration of the C-N bond related to tertiary amines from the structures of caffeine, theobromine, and theophylline molecules in coffee is typically seen in the 1350 cm^{-1} to 1200 cm^{-1} region, appearing at 1247 cm^{-1} in the figure. Additionally, coffee contains 2-5% moisture-derived H_2O , depending on storage conditions. The band corresponding to the

stretching vibration of water molecules is observed at 3294 cm^{-1} in the FTIR spectra (Barrios-Rodríguez et al., 2021).

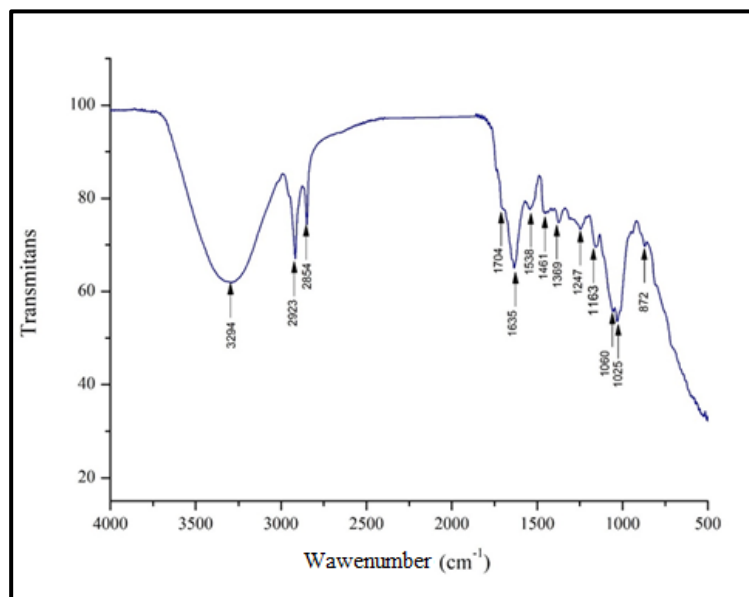


Figure 6. FTIR Spectra of Waste filter coffe grounds.

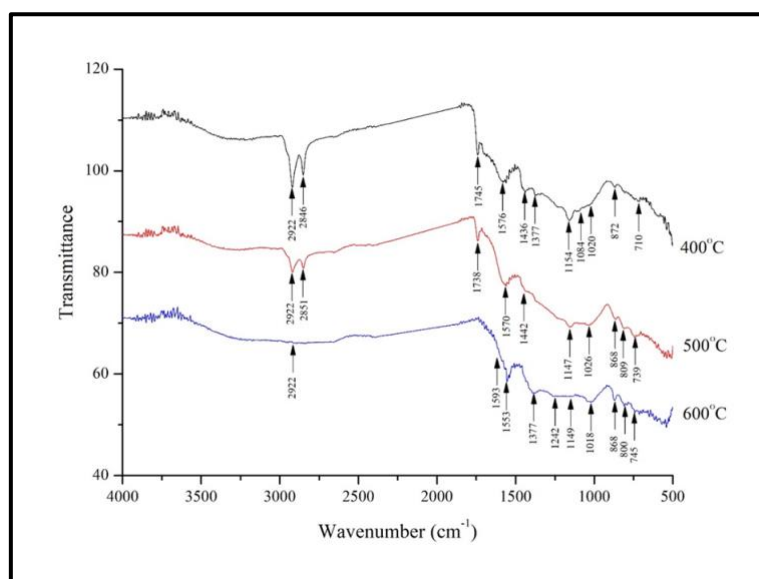


Figure 7. FTIR Spectra of the final structure at different pyrolysis temperatures.

When examining Figure 7, the stretching vibrations observed at 2922 cm^{-1} and 2846 cm^{-1} (occurring at 2831 cm^{-1} at 500°C) are attributed to the caffeine content in the coffee. The decrease in the absorbance values of these peaks with increasing temperature, and particularly the disappearance of the corresponding band in the residue after pyrolysis at 600°C , indicates that this molecule is removed from the environment post-pyrolysis. The presence of ester groups in the chlorogenic acids and groups originating from caffeine is evidenced by the phenomenon observed in the range of 1750 cm^{-1} to 1550 cm^{-1} , shown in Figure 9 at 1745 cm^{-1} (which occurs at 1738 cm^{-1} at 500°C). The peak intensity of this band initially decreases with increasing temperature during pyrolysis, and at 600°C , it creates a minimal effect, nearly disappearing from the environment (Barrios-Rodríguez et al., 2021). The bands located between 1300 cm^{-1} and 1150 cm^{-1} are also related to the chlorogenic acid structure. Pyrolysis temperature has a similar effect in this region; the intensity of the 1154 cm^{-1} peak, particularly noticeable at 400°C , decreases at 500°C , and shifts to 1147 cm^{-1} at 600°C . It appears at 1149 cm^{-1} , again with significantly reduced intensity (Briandet et al., 1996). This demonstrates that the relevant molecules in the content gradually dissipate from the environment at these temperature values.

Plant Germination Experiments

As a result of the elemental analyses of the biochars, the biochar obtained through pyrolysis at 500°C, which had the highest carbon content, was used in the plant germination experiments. Figures 8-9 show the photographs of the prepared pots and the growing plants.

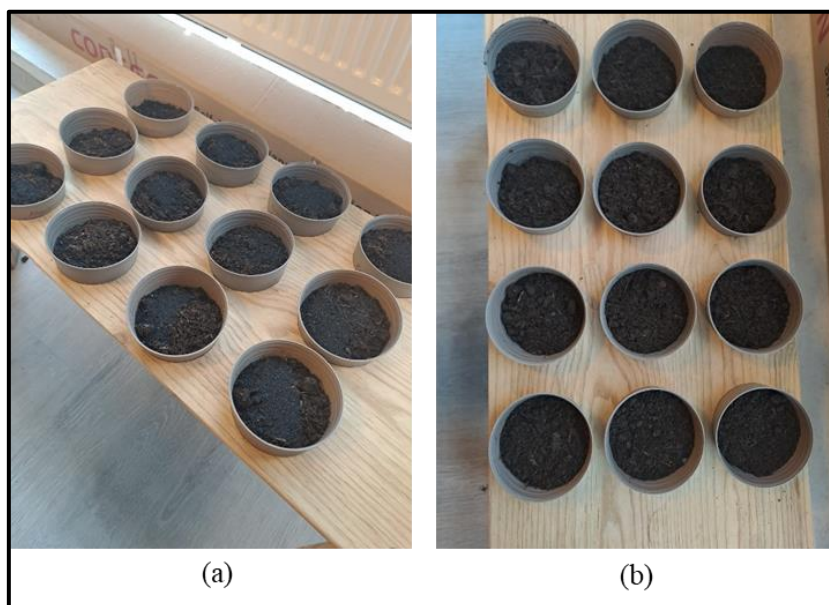


Figure 8. a- Soil not mixed with Biocar b- Soil left for conditioning.



Figure 9. Adult plants.

In this study, the effects of biochar on the root and stem length of plants were experimentally investigated. The results are presented in Table 4. Upon measuring the results, it was observed that the most suitable biochar application for arugula and garden cress plants was 2 tons/ha. The longest and heaviest plants were obtained when 2 tons/ha of biochar was used. Plants in pots with added biochar showed more growth compared to those grown in soil without biochar. Although plants in soil mixed with raw coffee developed less than those in soil with biochar, they still showed more growth compared to plants grown in just soil. The arugula plant demonstrated better development with 12 tons/ha biochar application and when grown solely in soil, having three leaves compared to the garden cress. In all other conditions, the plants had only two leaves (Table 4). The use of biochar obtained through pyrolysis in soil contributes to soil reclamation, carbon sequestration in soil, and increased soil fertility (Akgul, 2017). The porous structure of biochar enhances physical, chemical, and biological properties of soils, contributing to sustainable soil fertility (Zhang, 2021). The results obtained from this study support these positive attributes of biochar mentioned in the literature.

Table 4. Measurement results of plants after germination.

Mixtures in Pots	Root Length (cm)	Fringe Length (cm)	Plant Length of the whole (cm)	Plant Rhizome Length (cm)	Rooted Weight (g)	Rootless Weight (g)	Leaf Number (number)
Arugula (1 ton/da biochar)	1.8	0.3	7.6	5.8	0.03805	0.03735	2
Arugula (2 ton/da biochar)	3	-	10.25	7.25	0.0568	0.0534	3
Arugula (4 ton/da biochar)	1.9	1.1	7.95	6.05	0.0456	0.0438	2
Garden cress (1 ton/da biochar)	1.45	-	8.1	6.65	0.02335	0.0228	2
Garden cress (2 ton/da biochar)	1.3	0.3	9.85	8.55	0.03585	0.03505	2
Garden cress (4 ton/da biochar)	1.75	-	9.45	7.7	0.03375	0.03305	2
Arugula +soil	1.9	-	7.2	5.3	0.0379	0.0371	3
Garden cress +soil	2.8	-	7.8	5	0.0229	0.0211	2
Arugula + raw coffee	2	0.5	7.4	5.4	0.03795	0.03715	2
Garden cress + raw coffee	2.8	-	7.9	5.1	0.02314	0.02302	2

CONCLUSION

The results of this study indicate that biochar obtained from the pyrolysis of waste filter coffee grounds is suitable for use as a soil improvement to support plant germination. The findings demonstrate that when biochar derived from pyrolyzed waste filter coffee grounds is added to soil, it supports plant germination and growth.

Moreover, this study is significant as an application supporting the "zero waste" approach. The utilization of waste filter coffee grounds not only enhances environmental sustainability but also offers innovative solutions to waste management issues. It was determined that the biochar obtained after the pyrolysis of waste filter coffee grounds at 600°C contained 68.93% Carbon and 2.43% Nitrogen. It was observed that the best results were obtained in the application of 2 tons/da biochar to the soil for both arugula and garden cress plants. However, despite the promising initial results, further research is needed to enable the widespread application of this method in agricultural practices. To better understand the effects on soil and plants, field trials should be conducted in addition to laboratory-scale studies. These studies will help determine how biochar performs under different soil types and climatic conditions.

In conclusion, the pyrolysis of waste filter coffee grounds provides dual benefits in terms of waste management and soil improvement. This approach is seen as an important step towards the efficient use of resources.

Compliance with Ethical Standards

Peer-review

Externally peer-reviewed.

Declaration of Interests

The authors declare no conflict of interest.

Author contribution

Conceptualization, M.N.C., E.E.Ö., H.S., Ş.G., H.K.Ö. and A.Ö.; methodology, M.N.C., E.E.Ö. and H.K.Ö.; investigation, M.N.C., E.E.Ö., H.S., Ş.G., H.K.Ö. and A.Ö.; writing—original draft preparation, M.N.C., E.E.Ö. and H.K.Ö. All authors have read and agreed to the published version of the manuscript.

Funding

This article is derived from the TUBITAK 2209-A Project (Investigation of the Effects of Biochar Obtained from Biomass Recovery on Plant Growth) as part of an undergraduate thesis.

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