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# Production of potassium hydroxide-activated biochar and its use as a filler in polylactic acid for food packaging

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**Abstract** – Petroleum-containing packaging materials of the past and present have created serious ecological problems for the environment due to their resistance to biodegradation. In this context, researches have been conducted to promote the use of biodegradable films as an alternative to packaging materials. Among various biopolymers, poly(lactide) (PLA) has found application in the food industry owing to its promising properties and is currently one of the most industrially produced bioplastics. In this study, biomasses of olive pruning wastes, which are abundant in the Çanakkale region, were converted into biochar (BC) by slow pyrolysis, and their characterization was examined by adding them to PLA at different rates (5%, 10%, 15%, 20% by mass). Specific surface area analysis (BET), scanning electron microscopy (SEM) analysis, biochar yield, ash content, surface contact angles, and antimicrobial activity of film depending on the BC concentration were evaluated. As a result, potassium hydroxide (KOH) activated BC was successfully synthesized with a surface area of 1022 $m^2/g$. The hydrophobicity of films was improved with increasing BC ratio. Also, the film shows good antimicrobial activity toward gram-negative bacteria.

**Keywords:** *Biochar from olive branches, polylactic acid film, antimicrobial activity*

## 1. Introduction

Because petroleum-containing packing materials are resistant to biodegradation, they have caused significant ecological difficulties for the environment. The use and development of biodegradable packaging has been the general trend in the food packaging industry in recent years. Research has been done in this regard to encourage the use of biodegradable films in place of traditional packaging materials. Environmentally friendly plastics that can be substituted for petroleum-based plastics are biodegradable films [1]. Biopolymers are naturally formed by biomass and can be decomposed into their components by microorganisms in the environment. They are also defined as green polymers [2]. One of the most widely produced bioplastics today, poly(lactide) (PLA) is a biopolymer that has found use in the food sector due to its promising qualities [3]. The comparative characteristics of commercial and biobased polymers are displayed in Table 1.

Biomass has gained popularity as a renewable resource and organic solid waste in recent years. Lignocellulosic biomass can be thermochemically transformed into solid biochar. It is composed of aromatic and carbohydrate polymers, such as lignin and hemicellulose. Biochar is a porous, carbon-containing solid that is very resistant to breakdown and has a high degree of aromatization. It is created when biomass from plant or animal waste is thermally broken down without oxygen or in an atmosphere with low oxygen levels [4]. Biochar is made from biomass via a variety of thermochemical conversion processes, including carbonization, torrefaction, and

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pyrolysis. The slow pyrolysis process is the most often used of these. The biomass is broken down and transformed at a high temperature in an inert atmosphere during the pyrolysis process. Thus, the biomass forms a porous solid structure. Additionally, volatile condensable and non-condensable products are created. Liquid biofuels can be made from the condensable volatile compounds generated during the manufacture of biochar. Liquid bio-oils can be transformed into useful compounds or utilized as energy carriers in an inert atmosphere [5].

**Table 1.** Comparison of petroleum-derived plastic packaging and bioplastic packaging [6]

| **Petroleum-Based Plastics** | **Bio-plastic** |
|---|---|
| It is usually produced from fossil fuels and petrochemicals. | Produced from natural resources. |
| Causes high amounts of greenhouse gases. | Causes a small amount of greenhouse gases. |
| Cause environmental pollution. | Environmentally friendly. |
| It takes centuries to break down in nature. | Full biodegradation in nature takes 3-6 months after the end of use. |
| Used in the production of products such as construction, textiles, bottles, shoes, food packaging, and grocery bags. | Used in areas such as biodegradable food packaging, disposable biomedical instruments, carpets, and bags. |
| They consist of non-renewable resources. | They consist of renewable resources. |
| Some of the Traditional Plastics are high-density polyethylene, low-density polyethylene, polystyrene, polyethylene terephthalate, etc. | Some of the bioplastics are polylactic acid, polyamide11, starch, and cellulose-based protein-lipid-based biopolymers. |

Generally, biochar stability and properties depend on feedstock and pyrolysis conditions. Biochar produced at high temperatures has been reported to contain more fixed carbon, have predominantly stable carbon bonding, and have high thermal stability [7]. In recent years, biochar applications have received considerable attention in the fields of environment, agriculture, and industry. Biochar acts as a carbon sink in the soil, slowing down the chemical oxidation and reduction of biomass and preventing carbon emission to the atmosphere [8]. Especially activated biochar has a high adsorption capacity. Therefore, they can be used for the disposal of pollutants in wastewater [9]. There are numerous studies on the use of biochar for different applications. Sharma et al. [10] concluded that biochar can be used as activated charcoal and adsorbent in various applications by applying different activation methods to biochar. Singh et al. [9] reported that activated biochar with a high surface area can be used for phenol removal from refinery wastewater. Li et al. [11] reported that biochar produced from wood with high pyrolysis temperatures can be useful for organic carbon and nutrient retention in soil. They also stated that the biochar produced from corn husks and leaves at high pyrolysis temperatures will have a high effect on the improvement of acidic soils since the pH value is high. Mehdi et al. [12] stated that carbonaceous materials produced from biochar are suitable materials for super-capacitors due to their physical, chemical, and physicochemical properties and conducted studies on this subject.

Biochar is also effective in the use of filler components in polymers for various applications. It was reported that the addition of BC enhanced the hydrophobicity, mechanical, and sorption properties of packaging materials. Various amounts of biochar (0, 2, 4, 6, and 8% by mass) were added to the grta percha matrix to create black and biodegradable biochar/grta percha composite films. In comparison to the control film, the results demonstrated that biochar significantly enhanced the hydrophobicity and mechanical characteristics. High strength and barrier qualities were also demonstrated by the composite films. Specifically, the water

vapor permeability value was lowest, and the tensile strength was highest (18.3 MPa) for the 2% BC film. After 60 days in the soil, the composite films outgrew the polyethylene (PE) film in terms of biodegradability [13]. The impact of incorporating BC (derived from pine wood) into polypropylene composites including cellulosic fibers (such as rice husk, coffee husk, coarse wool, and waste wood) was examined by Das et al. [14]. By adding BC, the flame-retardant qualities were enhanced. They claimed that using biochar from a feedstock equivalent to biomass produced the greatest outcomes [14]. According to Bartoli et al. [15], several mechanical properties of epoxy composites are tunable as a result of the production parameters of olive-based BC. Through the examination of BCs produced by various pyrolysis procedures, they were able to determine the correlation between mechanical qualities and morphology [15]. In a study performed by Idrees et al. [16], BC was mixed with polyethylene terephthalate (PET) at different ratios and used as packaging material. It was observed that a composite filament appropriate for 3D printing applications was created by melting down BC that was formed from the carbonization of starch-based packaging material. In comparison to the undoped polymer matrix, the inclusion of BC particles increased the matrix's processability, producing composite products with better mechanical and thermal properties [16]. In their 2017 study, Moustafa et al. [17] created BC particles by grinding coffee, which they then combined with a biodegradable poly (butylene adipate-co-terephthalate) (PBAT) matrix. Applications for food packaging were made of it. Despite its hydrophobicity, BC was found to enhance the thermo-mechanical characteristics of the polymer matrix [17]. Arrigo et al. [18] developed PLA-based biocomposites using BC and two distinct processing techniques: melt mixing and solvent casting. Morphological and rheological analyses were used to determine the distribution of BC particles and the degree of polymer-filler interactions. Additionally, the thermal and mechanical behavior of the biocomposites was examined in order to determine whether the process actually had an impact on the PLA's actual properties [18]. Sobhan et al. [19] developed a packaging material that works as an ammonia sensor in packaging by producing activated BC from corn cobs and adding it to the PLA matrix at different ratios. Here, the percentage of BC was increased up to 85%. The mechanical properties and natural degradation of the desired material were not examined [19].

Literature studies have shown that biochar improves many properties of films. In this study, for the first time in the literature, biochar was produced from olive branches by KOH activation and its effect on some characteristic properties of PLA film was determined.

## 2. Materials and Methods

### 2.1. Biochar Production

The BC production procedure is shown in Figure 1. For the preparation of biochar; olive branches were first dried in an oven at 105 ºC for 1 day and cut into 2 mm lengths (Figure 1a). Biochar activation follows the following steps provided in [9]:

*i.* mixing and grinding KOH fractions with biochar in a 3: 1 ratio biochar

*ii.* thermal activation of the mixture in a tube furnace, in an inert environment, at 650°C for 1 hour at a temperature rise rate of 10°C/min (Figure 1b)

*iii.* soaking the mixture in 0.5 N HCl solution at room temperature overnight to remove $K_2CO_3$, excess KOH and other impurities

*iv.* removal of the mixture from the HCl solution by filtration and washing with distilled water until the pH value is stabilized

*v.* drying of the activated biochar in an oven.

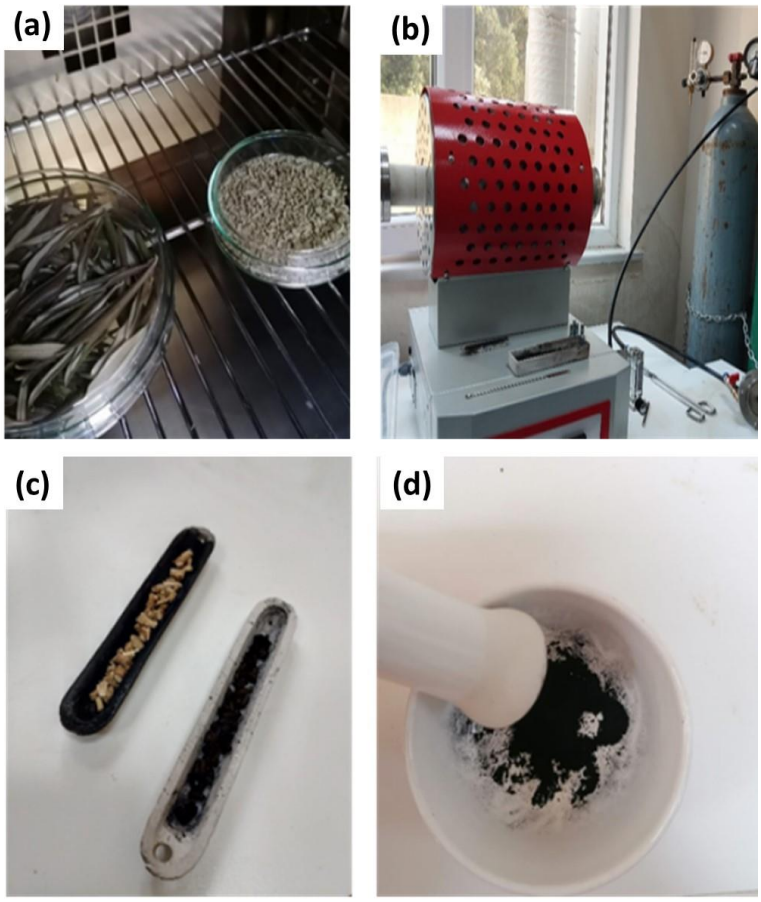


**Figure 1**. Biochar preparation stages

Figure 1c shows the olives branches before and after pyrolysis and Figure 1d shows the grounded particles below 10 μm.

The main reaction is given as (2.1) [20]. Potassium metals enable pore formation in biochar layers [9]. Thus, the biochar surface area is increased. Compared to physical activation, chemical activation using KOH gives biochar a higher surface area [21].

$$6KOH + C \leftrightarrow 2K + 2K_2CO_3 + 3H_2 \tag{2.1}$$

BC yield and ash content were calculated as shown in (2.2) and (2.3), respectively [22].

$$\text{BC Yield } (\%) = \frac{M_f(\text{g})}{M_i(\text{g})} \text{x} 100 \tag{2.2}$$

$$\text{Ash content } (\%) = \frac{M_r(\text{g})}{M_f(\text{g})} \text{x} 100 \tag{2.3}$$

where $M_i$ and $M_f$ are the weights of biomass and biochar before and after pyrolysis, respectively. $M_r$ is the ash content of BC after it was kept at 850 ºC in an ash oven for 4 hours.

## 2.2. Film Preparation

10% PLA by mass, 90% chloroform, and 10% DMF by volume formed the polymer solution, which underwent stirring at 40 °C until it was dissolved. In a separate experiment, 0–20% biochar (by mass of polymer) was dissolved in 5 ml of DMF and then mixed for 30 minutes using a homogenizer to distribute. Following the

homogenizer, the BC solution was added to the PLA solution and mixed for two hours at room temperature. The mixture was transferred onto a glass Petri dish, and the films were submerged in water to finish the phase separation. The films were then taken out and heated to dry at 60 °C.

The film preparation stage is shown in Figure 2.

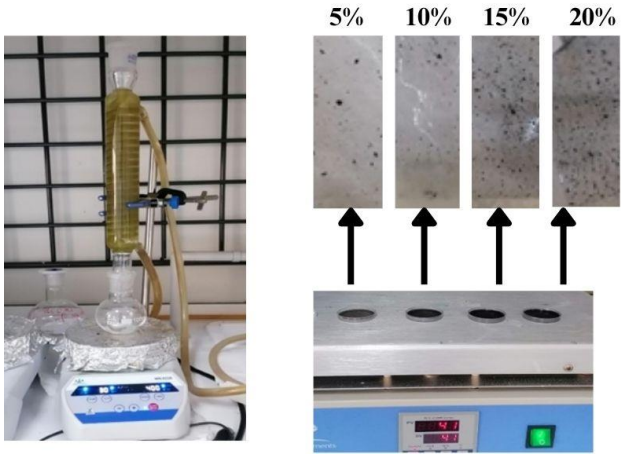


**Figure 2.** Film preparation stages

## 2.3. Characterization

The morphological analysis of the biochar produced was determined by scanning electron microscopy (SEM, JEOL JSM-7100-F). The surface hydrophobicity of the produced films was determined by contact angle tests. BC's pores were characterized using the Quadrasorb SI Brunauer-Emmett-Teller (BET) apparatus. The samples underwent an hour-long vacuum sealing and degassing process at 200°C. Adsorption of nitrogen gas was employed.

## 2.4. Antimicrobial Activity

*Escherichia coli* strain, a gram (-) bacterial strain, is used to determine antimicrobial activity. By the disk diffusion method, the antimicrobial activity of film samples against *E. coli* bacteria is observed. The bacteria to be tested are inoculated in a tryptic soy broth medium and the density is adjusted according to the 0.5 McFarland turbidity standard (108 microorganisms/ml). Spread 100 µm of the bacterial suspension on a petri dish containing Mueller Hilton agar medium with a sterile swab. 10 mm diameter samples were cut from the films, placed on the petri dishes, and incubated at 37°C for 24 hours. The test was repeated for three times.

# 3. Results and Discussion

## 3.1. Characterization

Figure 3 shows SEM analysis of biochar with and without KOH activation. As seen in Figure 3a, BC has macro pores. Activation is applied to increase the porosity and surface area of biochar. Activation is divided into two main groups: physical and chemical activation. Chemical activation can be carried out with suitable acids, bases or metal salts. In this study KOH base was used for activation. The main purpose of activation using base is to increase the surface area of the biochar and the functional groups it contains. As seen in Figure 3b, KOH activation significantly increased the porosity. This result was also supported by BET analysis.

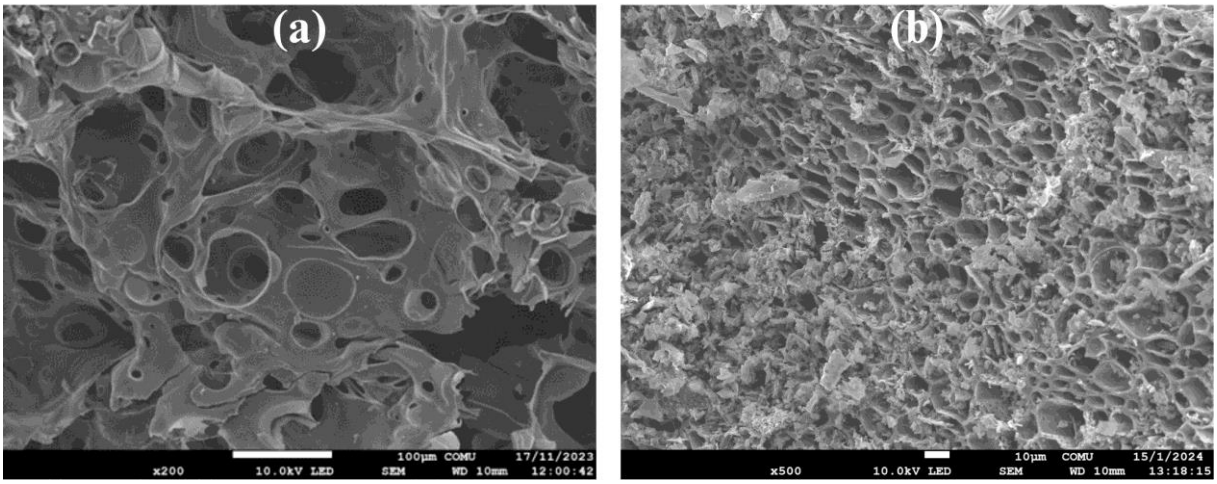


**Figure 3.** SEM micrographs of BC without (a) and with KOH activation (b)

Table 2 shows that the specific surface area of biochar increased from 1.477 $m^2/g$ to 1022.201 $m^2/g$ as a result of solid KOH activation. According to the literature, KOH provides a higher surface area to biochar compared to non-activated BC. Activation with KOH increases the specific surface area of biochar by increasing the number of mesopores [21]. There are many biochar and activated biochar studies in the literature. In order to compare the specific surface area results, biochar studies were collected as a result of a literature search (Table 2). As seen in the table, although different biomasses were used, the activation process at the same temperature increased the surface area. However, in tests with or without the same activation, the surface area increases again as the temperature increases. However, uncontrolled pyrolysis temperature increase decreases the yield. In this study, high surface area was obtained with KOH activation at relatively low temperature.

**Table 2.** Comparison of surface area

| Raw material | Pyrolysis temperature (°C) | Activation Method | Surface area ($m^2/g$) | Reference |
|---|---|---|---|---|
| **Olive Branch** | **600** | **-** | **1.477** | **This study** |
| **Olive Branch** | **650** | **KOH** | **1022.201** | **This study** |
| Corn husk | 600 | - | 86.750 | [21] |
| Corn husk | 600 | $K_2CO_3$ | 541.910 | [21] |
| Bamboo | 600 | - | 307.100 | [8] |
| Spruce Wood | 600 | - | 465.140 | [5] |
| Eucalyptus Branch | 700 | KOH | 1754.000 | [9] |
| Cone Cone | 800 | KOH | 1714.500 | [23] |
| Douglas Fir | 700 | KOH | 1050 | [24] |

Biochar yield varies according to feedstock and pyrolysis temperature. Biochar yield is inversely proportional to the amount of volatile matter in the biomass content and directly proportional to the amount of lignin in the biomass content [8]. For pyrolysis applied to the same feedstock, biochar yield decreases as the pyrolysis temperature increases [21]. As a result of biochar yield analysis, the yield of biochar produced at 650°C pyrolysis temperature, 1 hour cooking time, 10°C/min temperature increase rate, and 1L/min $N_2$ flow rate was calculated as 29.75%. As a result of the literature review, Table 3 shows the comparison of the achieved yield with the literature. As can be seen in the table, the efficiency decreased in pyrolysis at higher temperature

because it is a known fact that at higher temperature, more components are separated from the medium and porosity increases. In this study, lower yields were obtained from the pyrolysis process at lower temperature since it was performed at 650 ºC.

**Table 3.** Comparison of the yield

| Raw Material | Pyrolysis Temperature (ºC) | Yield (%) | Reference |
|---|---|---|---|
| Olive Branch | 650 | 29.7 | This study |
| Rice Husk | 500 | 34.85 | [22] |
| Bamboo | 600 | 27 | [8] |
| Eucalyptus | 500 | 30.2 | [25] |
| Spruce Wood | 600 | 25.6 | [5] |
| Wood | 700 | 22.71 | [26] |

As a result of biochar ash determination, the ash content of biochar (BC-650-1) was calculated as 3.37%. As a result of the literature review, Table 4 shows the ash content ratio comparisons.

**Table 4.** Comparison of ash contents

| Raw Material | Pyrolysis Temperature (℃) | Ash Content (%) | Reference |
|---|---|---|---|
| **Olive Branch** | **650** | **3.37** | **This study** |
| Corn husk | 600 | 4.1 | [21] |
| Bamboo | 600 | 4.65 | [8] |
| Spruce Wood | 600 | 1.5 | [5] |
| Duglas Fir | 700 | 2.89 | [24] |

The ash contained in lignocellulosic materials consists of calcium, magnesium, and potassium carbonates and oxides [6]. Biochar produced at higher pyrolysis temperatures has a higher ash content [8]. When Table 4 is investigated, it is seen that the ash ratios of biochar produced at similar temperatures are close to each other but different. The difference in ash content is due to the variability of the inorganic matter content of different raw materials [26]. Additionally, just like the yield, high-temperature pyrolysis results in less residue because a greater proportion of the ingredients are transformed. As can be seen in the table, in this study, less residue remained from the treatment at 600 ºC, while more residue remained from the treatment at 700 ºC.

The angle formed by the tangent line at the liquid's contact surface point and the film surface's baseline is known as the contact angle. It is frequently employed to gauge a film's water resistance [27]. When food is coated and exposed to water during storage, the ability of the edible coating to withstand water damage is crucial [28]. The contact angle serves as a gauge for a surface's wettability and establishes its level of hydrophobicity. It is well known that when the contact angle rises, surface hydrophobicity does as well [29]. The Sessile Drop method was utilized to measure the contact angles of the films. Results are shown in Figure 4. As can be seen in the figure, the contact angles decreased as the BC ratio increased.

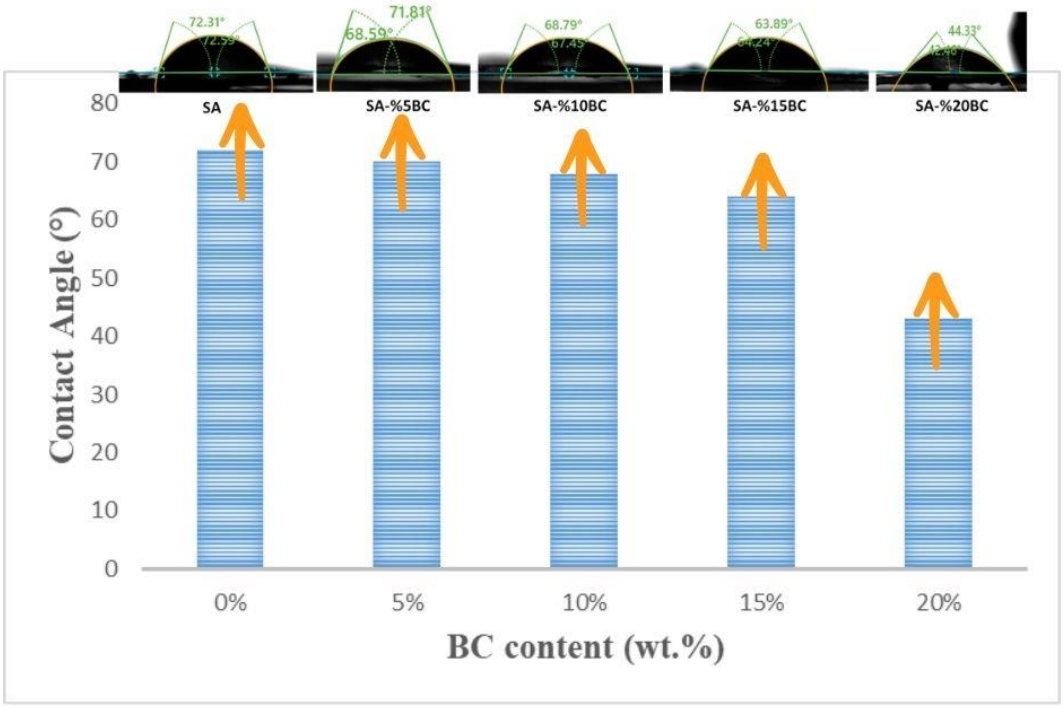


**Figure 4.** Effect of BC content on contact angle

According to a study by Vogler [30], hydrophobic surfaces show a contact angle greater than 65°, while hydrophilic surfaces show less than 65°. It has become widely acknowledged in recent years that a substance is considered hydrophilic when the contact angle is less than 90° and hydrophobic when it is larger than 90°. Surface chemistry and roughness are connected to surface wettability [31]. As seen in the figure, the contact angle values of the film samples were found to be in the range of 42.46-72.59˚. It is thought that PLA films containing 15% and 20% BC show hydrophilic properties [32].

The antimicrobial activity of the prepared samples was determined using the disk diffusion method. Figure 5 shows the petri dish after 24 hours of inoculation. There is no visible bacterial growth was observed when the bacteria planted on packaging films with different BC concentrations were kept in an oven under appropriate conditions. As shown in Figure 5, In films doped with 5% and 10% BC, microbial growth occurred around the film but not on it, indicating that the films are antimicrobially effective. In films doped with 15% and 20% BC, no microbial growth was observed, indicating that BC doping improves the antimicrobial properties of the films. The contribution of carbon-based materials to antimicrobial activity was described by Nishshankage et al. (2024). These processes include adsorption to bacterial and fungal cell walls via diffusion and electrostatic interactions. When BC pierces through the membranes and cell walls of bacteria and fungi, they cause cytoplasm to seep out of the cells. Additionally, when BC binds to DNA and RNA, they destroy the nucleic acid structures, which effectively stops bacteria from growing [33]. If bacterial growth had been observed, discoloration would have been observed on the parts of the packaging films where bacteria grew [34].

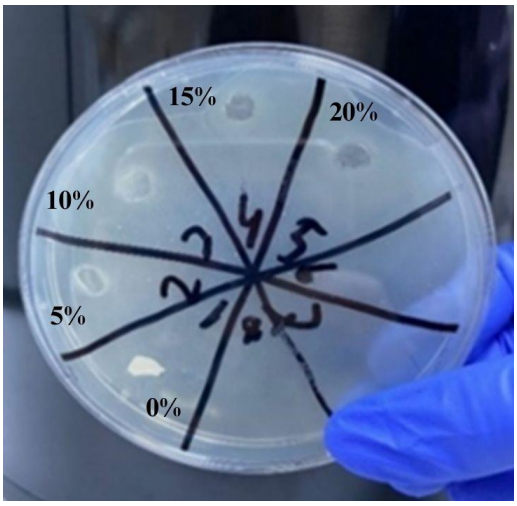


**Figure 5.** Antimicrobial test results after 24 hours

## 4. Conclusion

Avoiding plastic waste has become a necessity for a sustainable environment. The packaging industry accounts for more than 20% of the total plastics industry. Therefore, it is of great importance that food packaging is done with bio-based packaging instead of petroleum-derived packaging. In this study, biochar with high pore size was produced by KOH activation and added to the PLA matrix to form bio-based packaging film samples.

*i.* The results showed that the addition of BC improved the hydrophilicity of the film but reduced its resistance to water at overloading.

*ii.* The yield of the produced films was higher than those of the BC particles produced at the same conditions.

*iii.* The contact angle values of the samples were found to be in the range of 42.46-72.59˚. PLA films containing 15% and 20% BC showed hydrophilic properties, which is thought to be due to the fact that biochar has a water-affinity structure.

*iv.* According to the results of the antimicrobial activity test, no visible bacterial growth was observed when the bacteria planted on the BC loaded PLA packaging films. This proves that the films are antimicrobial. Antimicrobial packaging films are also extremely important for human health.

In the future, it is recommended to investigate the properties of KOH-activated films by performing basic tests necessary for food packaging.

## Author Contributions

All the authors equally contributed to this work. They all read and approved the final version of the paper.

## Conflict of Interest

All the authors declare no conflict of interest.

## Ethical Review and Approval

No approval from the Board of Ethics is required.

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