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Influence of a Walnut Shell Biochar Additive on the Tribological and Rheological Properties of Vegetable Lubricating Grease

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Abstract: This paper presents the results of a study on the effect of a biochar additive produced via pyrolysis at 400 °C and 500 °C from waste biomass, i.e., walnut shells, on the tribological and rheological properties of vegetable lubricating compositions. Sunflower oil and amorphous silica, used as a thickener, were used to prepare the lubricants. To the base lubricant prepared in this way, 1 and 5% biochar additive were introduced, and for comparison, we took the same amounts of graphite. Tests were carried out on the anti-wear properties, coefficient of friction, and changes in dynamic viscosity during the tribological test, as well as on the anti-scuffing properties for the tested lubricant compositions. The effect of the applied modifying additive on the lubricating and rheological properties of the prepared lubricating greases was evaluated. On the basis of the study of vegetable greases, it was found that the addition of 5% biochar from walnut shells produced during pyrolysis in 500 °C had the most favorable effect on the anti-wear properties of the tested greases, while the 5% biochar from walnuts shell prepared via pyrolysis at 400 °C had the best anti-scuffing protection. The use of the biochar additive in vegetable greases resulted in a reduction in the dynamic viscosity of the tested greases, particularly for greases modified with 5% walnut shell biochar produced at 500 °C, which is particularly important with respect to the work of steel friction nodes, as well as in central lubrication systems.

Keywords: lubricants; sunflower oil; amorphous silica; tribological properties; biochar additive; dynamic viscosity; pyrolysis process; rheometer with tribological cell; Raman spectroscopy; friction coefficient

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1. Introduction

Modifying additives are special chemicals used in lubricants to improve their properties and adapt them to specific working conditions. They enable greases to protect surfaces more effectively against wear, seizure, corrosion, high temperatures, or extreme loads [1–3].

Additives increase the wear and friction resistance of greases by preventing damage to metal surfaces and improving the lubricating properties of greases by reducing the coefficient of friction, resulting in a significant reduction in wear with respect to the machine and equipment components [4–6]. The use of additives in lubricants increases the corrosion and oxidation resistance of the lubricated components and increases the adhesion of the lubricant to the surface, which prevents leaching. Another function of the additive is to adapt the grease to extreme working conditions, such as high loads and high temperatures [5–9].

Analysis of changes in the composition of the surface layer during contact between the lubricant and the elements of the tribosystem is of great importance with respect to

lubrication efficiency [10–12]. Elucidating the mechanism and establishing the kinetics of conversion under the influence of mechanical forcing and the catalytic interaction of the elements of the tribological system will enable the determination of the parameters of these processes and contribute to the optimization of the composition of lubricants in order to obtain the desired lubricating properties [13–15]. Finding a correlation between the type of active additive and changes in its structure as a result of tribochemical reactions occurring under mechanical and thermal excitations will contribute to identifying the mechanism of these interactions and determining the optimum conditions for this process [16–18]. A lubricating grease in the vicinity of the machine components forms a separate near-surface phase called a boundary layer. When a lubricant is within the range of the surface forces of the machine elements, the interaction of these forces results in a greater ordering of the components of the lubricants, which is associated with an increase in intermolecular interactions between the lubricant composition and the elements of the tribosystem [19–24]. As a result of these interactions, a boundary layer is formed, which prevents intensive wear and seizure processes. In order to effectively protect equipment components from excessive wear, it is necessary to learn the characteristics of the changes occurring between the components of the lubricating composition and the surface of the tribosystem [25–28].

The general principles describing the interactions between lubricant components and trbosystem elements have been described with the use of model substances of a relatively simple chemical composition, while examination of the boundary layers, as a result of the interaction of the actual components of lubricants, such as oils, thickeners, and additives, may constitute an important contribution to the furthering of the scientific description of the physicochemical processes to which lubricants are subjected in the tribological node [29,30]. Identifying the changes occurring in the surface layer of the friction elements in the presence of lubricants will make it possible to extend our knowledge of the tribochemistry processes affecting the formation of boundary layers during mechanical excitations and acting on the lubricant components [28,31–34].

Finding the correlation between the structure of the surface layer, the lubricating properties of the lubricating compositions, and the mechanical excitations will enable the use of such components to increase the life of the tribosystem [35,36]. Control of the processes affecting the properties of a lubricating composition can be achieved by using thickeners or modifying additives containing active elements whose diffusion into the surface layer will increase wear resistance, influence the durability of steel–steel associations, and improve tribological parameters such as wear resistance [32,35]. Lubricant components degrade under mechanical forcing, and the resulting chemical compounds can be adsorbed onto the friction surface to form a boundary layer. A complete identification of the changes in lubricant components under the influence of mechanical forcing in frictional processes can only be realized by applying a system of complementary testing techniques [31,34,35].

Products formed from waste biomass during pyrolysis processes are becoming increasingly important in lubricant production technology. The biochars thus formed can be used as an additive with which to modify the functional properties—including the tribological properties—of lubricants [37–39]. The biochar additives can successfully replace previously commonly used additives such as amines, molybdenum disulphidem, or ZDDP. Therefore, the authors, taking into account the physicochemical properties of walnut shells, decided to subject the shells to a pyrolysis process and to introduce the obtained biochar into the structure of the lubricating grease to improve its lubricating properties [40–43].

Walnut shells have many properties that make them a valuable raw material in various fields, from natural medicine to industry. They contain juglone, which inhibits the growth of bacteria and fungi, and in the composition of walnut shells, you can also find many antioxidants that protect against oxidation processes. They are used as a dye in cosmetics

and other dyeing processes [44–46]. They are a component of ecological plastics and biocomposites and are also used to clean surfaces as an abrasive agent. In addition, walnut shells contain tannins, flavonoids, and phenolic acids, which have antioxidant effects, as well as vitamins B, C, E, K, and P and minerals, including zinc. In the aviation industry, they are used to clean jet engine turbines. Shells can also be used to produce biodegradable materials and composites that reinforce plastic [47–50].

Walnut shells are widely used in industry due to their hardness, biodegradability and rich chemical composition. They are also used in the production of light but durable materials, e.g., in the automotive industry [51,52]. Walnut shells constitute a raw material for the production of medicines and supplements in antiparasitic preparations. They are often used as food additives or as a natural preservative. They are also used as natural filters for purifying water and fats in the food industry, and they also help remove unwanted substances from oils. They can also be used as a component in biodegradable food packaging [53–55].

According to the available data, walnut production in Poland is around 6800 tons per year [56]. In the world, according to forecasts for the 2024/25 season, global walnut production is expected to increase by 2% to 2.7 million tons, mainly due to higher production in China, Chile, and other countries [57]. Shells constitute a significant part of the mass of a walnut, but the exact percentage may vary depending on the variety and growing conditions. It is assumed that shells constitute around 50–60% of the mass of the whole nut. Therefore, if we assume production at the level of 6800 tons, around 3400–4080 tons are shells. Similarly, shells constitute around 1.35–1.62 million tons of the 2.7 million tons of nuts produced worldwide. One way to solve the problem of walnut shell disposal is the pyrolysis process [56,57].

Biochar, obtained through the pyrolysis of biomass, has also attracted much interest as a potential additive to lubricants due to its functional properties that can improve their performance in various applications. The addition of biochar to lubricants can lead to improved tribological and rheological properties, making it a valuable ingredient in the development of sustainable and environmentally friendly lubrication systems [43].

In ref. [43], it was shown that the addition of biochar from chokeberries significantly improves the anti-wear and anti-scuffing properties of vegetable greases, surpassing the effectiveness of commercial activated carbon. In ref. [58], the authors analyzed the effect of sugarcane bagasse biochar and activated carbon as additives modifying the tribological properties of greases. Primarily, these additives affect the reduction of friction and wear in greases, highlighting the key role of the porous structure and specific surface area of the additives in improving the tribological properties of greases. On the other hand, in ref. [39], the authors presented the use of biochar with nanometric structures as an additive for lubricating oils, showing improved the tribological properties and better surface wetting compared to traditional additives. In ref. [59], the authors used biochar as a modifying additive for lubricating compositions obtaining a significant improvement in its anti-wear and anti-scuffing properties. In ref. [60], the authors used biochar to improve the tribological properties of lubricating oil. In refs. [61,62], the authors used biochar as a lubricant additive to improve the anti-wear properties of mineral oil-based lubricants. In the ref. [63], the authors used biochar as an additive to lubricants to improve the tribological properties of the tested lubricating compositions.

The aim of this study was to investigate the effect of a biochar additive produced via the pyrolysis of walnut shell biomass on the changes in the tribological and rheological properties of lubricants produced on the basis of vegetable oil, intended for use in the food industry, and developed at the Lukasiewicz Research Network—Institute for Sustainable Technologies in Radom.

2. Materials and Methods

A series of model lubricating greases were developed using non-toxic ingredients as dispersion and dispersed phases [64–67]. Sunflower oil was used as the dispersing phase. The sunflower oil used to prepare of grease compositions was defined by specific physicochemical parameters as follows: 0.756 g/cm^3 density; 32.44 cSt kinematic viscosity at 40 °C; 1.87 meq O_2/kg peroxide number; 113.34 g I_2/100 g of iodine index; 156.23 mg KOH/g of saponification index; and 1.43 mg KOH/g of acid index.

Aerosil®-type modified silica [7,9,68] were used as dispersed phases. The base oil and thickener were mixed together using a high-speed homogenizer at room temperature at 18,000 rpm for 30 min. The prepared vegetable-based lubricating composition was modified by introducing a 1% and 5% modifying additive into its structure. As an additive that modifies the tribological and rheological properties of the grease, the authors decided to use biochar made from walnut shells produced via pyrolysis at 400 °C and 500 °C. For comparison, a biochar grease was used in which graphite with a grain size of less than 30 µm was used as an additive. The selected components were used to produce vegetable lubricants of the second consistency class. The consistency class of the produced lubricant compositions was tested in accordance with the requirements of the ISO 2137:2021 standard [69] using a laser penetrometer produced by the Lukasiewicz Research Network—Institute for Sustainable Technologies [7,9]. The prepared lubricating compositions were designated as follows: grease based on sunflower oil (grease A); base grease modified with 1% biochar from walnut shells prepared at 400 °C (grease B); base grease modified with 5% biochar from walnut shells produced at 400 °C (grease C); base grease modified with 1% biochar made from walnut shells at 500 °C (grease D); base grease modified with 5% biochar made from walnut shells at 500 °C (grease E); and, for comparison, grease with graphite (grease F). The chemical composition of the tested lubricating greases are shown in Table 1.

Table 1. Chemical composition of tested vegetable lubricants.

Designation of the Test Grease	Dispersion Phase	Dispersed Phase	Content of Modified Additive	Pyrolysis Temperature in Which the Additive Was Produced [°C]
A	Sunflower oil	Amorphous silica	without additive	-
B	Sunflower oil	Amorphous silica	1% biochar from walnut shell	400
C	Sunflower oil	Amorphous silica	5% biochar from walnut shell	400
D	Sunflower oil	Amorphous silica	1% biochar from walnut shell	500
E	Sunflower oil	Amorphous silica	5% biochar from walnut shell	500
F	Sunflower oil	Amorphous silica	5% graphite	-

All lubricating compositions produced were subjected to tribological and rheological tests, and the results obtained were statistically processed using Student's *t*-test. The tribological and rheological tests of the tested lubricating compositions were carried out in triplicate.

The walnut shells were pyrolyzed over a temperature range of 400 °C to 900 °C, using carbon dioxide (CO_2) as a protective atmosphere (Figure 1).

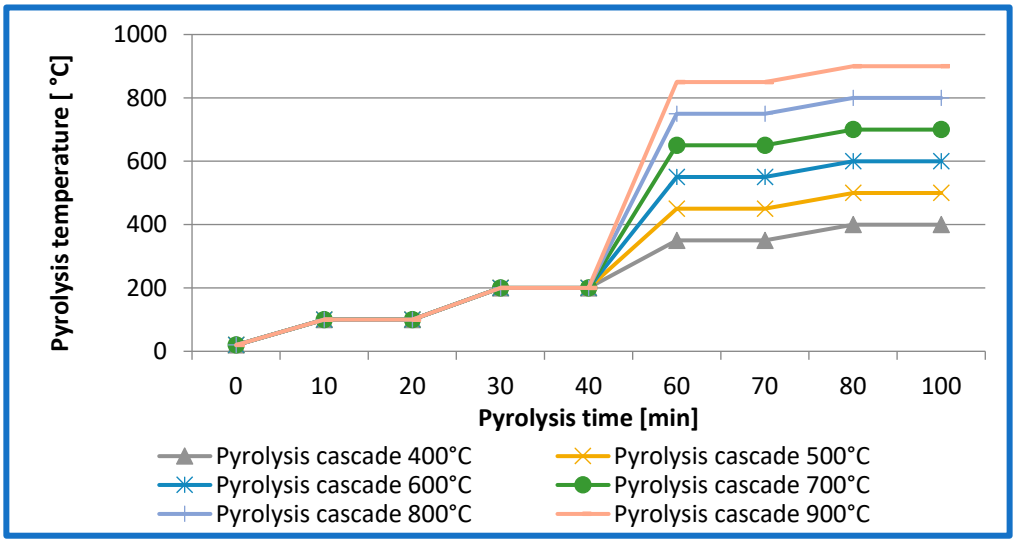


Figure 1. Temperature profile of the walnut shell pyrolysis process as a function of the final temperature.

The entire carbonization process was carried out according to a cascade programme with four temperature stages. In the first stage, the temperature of the material was raised from 20 °C to 100 °C within 10 min and then maintained for a further 10 min. The second stage involved further heating to 200 °C over 10 min, followed by a 10 min stabilization period. In the third stage, the temperature was increased to an initial level specific to the pyrolysis version: 350 °C (for 400 °C), 450 °C (for 500 °C), 550 °C (for 600 °C), 650 °C (for 700 °C), 750 °C (for 800 °C), and 850 °C (for 900 °C). The heating time in this stage was 20 min, followed by 10 min of holding. The fourth and final stage involved heating the material to a maximum temperature of 500 °C, 600 °C, 700 °C, 800 °C, or 900 °C depending on the process variant. It took 10 min to reach this temperature, which was then stabilized for 20 min. The entire process was carried out in a Czylok chamber furnace, model FCF-V12RM, with a continuous flow of CO_2 inert gas at 5 L/min. After pyrolysis, the samples were seasoned for 22 h, during which time the gas flow was reduced to 2 L/min [43].

The biochar produced during the pyrolysis process was fractionated using a laboratory sifter with a set of vibrating screens to obtain a uniform grain fraction. The biochar fraction was limited to particle sizes below 200 µm to match the grain size of the commercial graphite used in the study, which also has a particle size <200 µm; this ensures the direct comparability of the additives and eliminates the effect of different granulations on the tribological properties of the analyzed lubricants.

Tribological examinations: The tribological characteristics of the lubricants under investigation were assessed using a four-ball testing device. The evaluation of the surface wear defects, i.e., the scar diameters, was conducted utilizing an optical microscope, to ensure meticulous analysis. The collected data enabled the calculation of the $G_{oz/40}$ and poz parameters, which represent the anti-wear and anti-scuffing performance of the tested lubricants [70,71]. The tribological behavior of the investigated materials was evaluated by determining two key metrics: the limiting load of wear ($G_{oz/40}$) and the limiting pressure of seizure (Poz). In the course of the experiments, steel balls with a nominal diameter of 12.7 mm were utilized. These materials were fabricated from ŁH 15 bearing steel and exhibited a surface roughness of Ra = 0.32 µm, in addition to a hardness of 60–65 HRC.

The limiting load of wear was determined by applying a constant load of 392.4 N on the frictional contact for the full period of the test, which was 3600 s. The ball was rotated at a speed of 500 rpm in accordance with the guidelines specified in WTWT-94/MPS-025 [70].

The parameter characterizing the anti-wear characteristics is employed as a metric for the lubricant's wear resistance, calculated using the following formula:

$$G_{oz} = 0.52 \times P_n / d_{oz}^2$$

where P_n denotes the load of the friction node assembly equal to 392.4 N, and d_{oz} denotes the dimensions of the scars formed on the steel balls utilized for the experiment.

The evaluation of lubricating performance under scuffing conditions, i.e., when the load increases without interruption throughout the testing process, was conducted in accordance with the procedure established by the Lukasiewicz Research Network—Institute for Sustainable Technologies. The experimental procedure involved the application of a progressively greater load, initiated at 0 N and elevated to a maximum of 7200 N over a duration of 18 s. This was conducted while the spindle rotated at a speed of 500 revolutions per minute and the load increased at a rate of 409 Newtons per second. The occurrence of a sharp rise in scuffing stress, denoted as P_t [71], was identified as the point at which the frictional torque exhibited a significant increase.

The test continued until either the value of frictional moment reached 10 Nm, or the ultimate strength of the machine (7200 N) was obtained. This threshold was designated as the limiting load of scuffing, also known as Poz [71]. In order to ascertain the ultimate result, the numerical average of a minimum of three measurements was calculated, with a maximum permissible variation of 10%. In order to analyze the data statistically, the Q-Dixon method was employed at a degree of assurance of 95% reliability.

The limiting pressure of seizure, which is indicative of a grease's anti-scuffing performance under high-stress conditions, was calculated using the following equation:

$$p_{oz} = 0.52 \times P_{oz} / d_{oz}^2$$

where P_{oz} is the limiting load of scuffing, and d_{oz} is the dimensions of the perforation observed on the steel balls after testing [18,19,25,72,73].

Rheological experiments: To evaluate the rheological characteristics of the studied lubricant mixture, a portable MCR 102 rotating triborheometer from Anton Paar (Graz, Austria) was employed, equipped with a T-PTD 200 tribocell with a specimen in a concentrated plate-to-ball geometry. In this configuration, three solid steel parallelepiped plate elements were applied with an appropriate load and rotational speed against a ball mounted on the rotating spindle. The balls, with a size of 12.7 mm in aperture, and the plates, with a size of 15 × 5 × 2 mm, were both manufactured from LH 15 bearing steel (surface roughness: Ra = 0.3 μm; hardness: 60–63 HRC). The experiments were performed under dip lubrication conditions. Viscosity experiments were performed on the friction node at a force of 10.00 Newtons and a velocity of 500 revolutions per minute for a period of 3600 s and at a constant temperature of 20 degrees Celsius [74].

Spectral studies: Spectroscopic analysis was carried out using a high-resolution confocal scanning Raman spectrometer (NRS 5100, Jasco Corporation, Tokyo, Japan) to investigate the chemical structure of lubricants. The instrument was designed with a 532.12 nm wavelength emission laser and a CCD sensor. The device had a diffraction grating thickness of 600 lines/mm, a maximum laser intensity of 5.2 mW, a numerical aberration of 4000 μm, and provided spectrum data in the region of 3700–100 cm^{-1} with a spatial resolution of 4.2 μm. The objective used offered 20-fold imaging power, and every single observation was made with an exposure time of 200 s. Grease mixtures were prepared for examination on microscope slides [75].

Fourier transform infrared (FTIR) spectra were recorded using a Jasco FTIR 6200 spectrometer in the reflection method, employing a Pike accessory with a diamond-based

quartz. Each spectrum was obtained over an exposition period of 30 s, covering a spectral region from 650 cm^{-1} to 4000 cm^{-1}.

3. Results and Discussion

3.1. Analysis of the Efficiency of the Pyrolysis Process

During the pyrolysis of walnut shells, the amount of biochar obtained decreases with increasing temperature because at higher temperatures, more organic components are decomposed and converted into gaseous products. At 400 °C, the efficiency is 38.6%, and at 900 °C, it drops to 16.6%. This decrease is the result of more intensive thermal processes, which lead to greater mass loss in the form of gases. In the first phase of pyrolysis, already at temperatures below 200 °C, water and light volatile compounds are removed. Then, in the range of 200–400 °C, the decomposition of hemicellulose begins, which leads to the release of carbon dioxide, carbon monoxide, and small amounts of hydrocarbons. At 400 °C, the efficiency is highest because most of the organic components still remain in their solid form. In the range of 400–600 °C, the decomposition of cellulose and initial decomposition of lignin begin, which causes further mass loss and a drop in efficiency to 29.5% at 600 °C. At this stage, the amount of gaseous products, such as hydrogen and methane, increases, and the structure of biochars begins to take on a more aromatic character. After exceeding 600 °C, lignin degradation is increasingly intensive, and at temperatures of 600–800 °C, graphitization processes begin to dominate. Lignin, which is the most difficult component of biomass to decompose, gradually decomposes, which leads to a further decrease in efficiency to 21.3% at 800 °C. In this temperature range, the amount of volatile products, such as hydrogen, carbon monoxide, aromatic hydrocarbons, and tar fractions, increases, which contribute to the loss in mass of the remaining material. Above 800 °C, the pyrolysis process enters the final phase, in which further graphitization and the removal of residual heteroatoms, mainly oxygen and hydrogen, occur. At 900 °C, the efficiency is only 16.6%, which means that most of the biomass has been converted into gaseous products. The structure of biochars becomes more and more ordered and resembles graphite, but it contains fewer reactive oxygen groups, which makes the material less chemically active but more thermally stable.

Spectral characteristic of biochar from walnut shell: The walnut shell before pyrolysis and after pyrolysis were presented in Figures 2 and 3. The resulting pyrolysis biochars from walnut shells was subjected to spectral testing using Raman spectroscopy and FTIR spectroscopy to assess the chemical structure of the resulting biochar additives (Figures 4a–g and 5A,B).

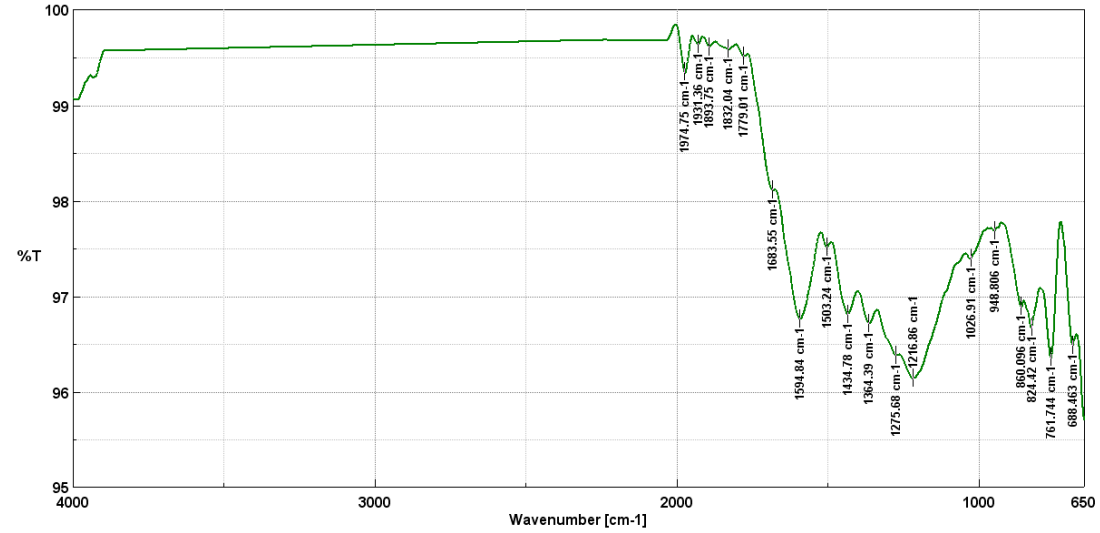


Figure 2. Walnut shells before pyrolysis.

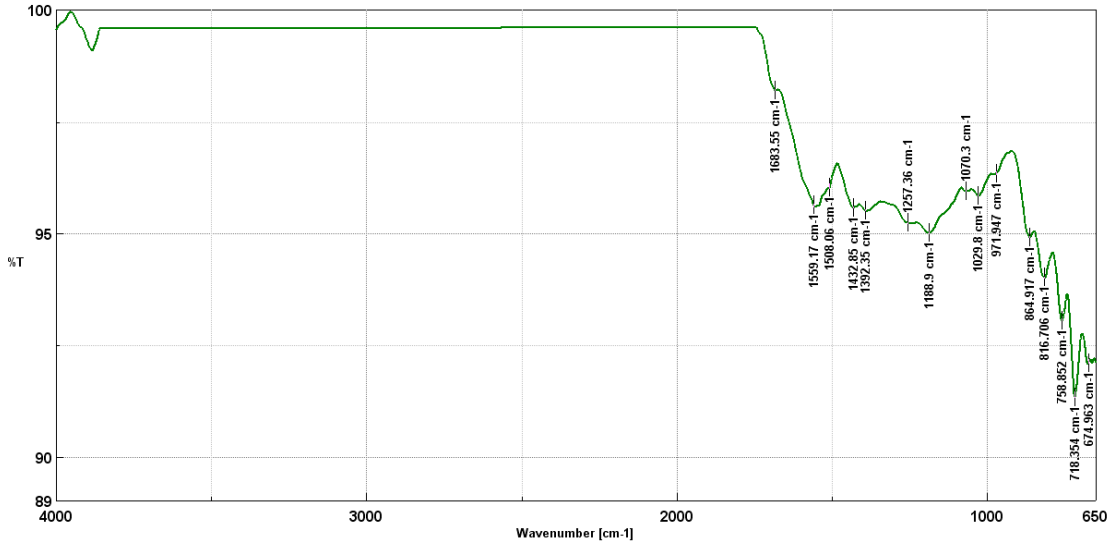


Figure 3. Walnut shells after pyrolysis in 500 °C.

The results of the chemical structures of the biochar produced via the pyrolysis of walnut shells at 400–900 °C and graphite using the FTIR technique are shown in Figure 4a–g.

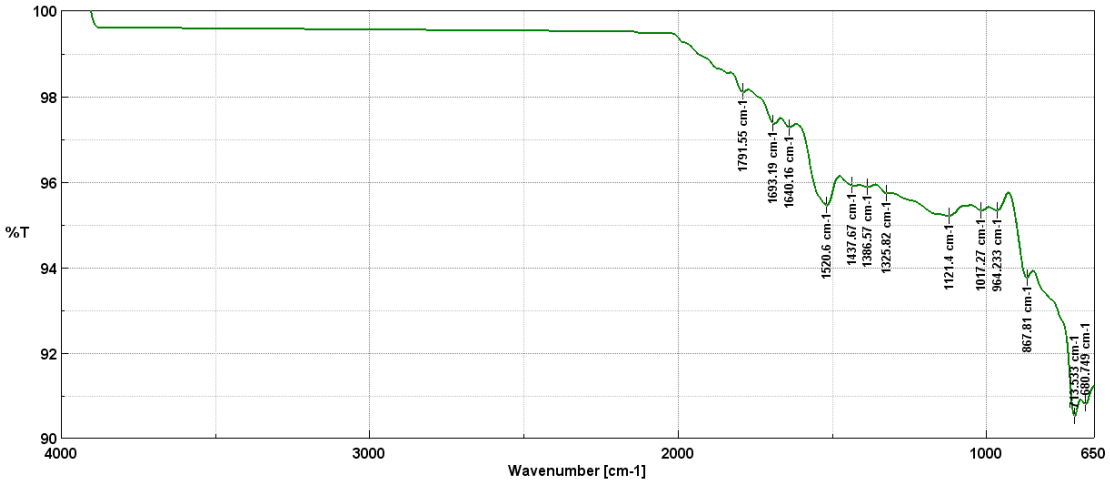


(**a**)



(**b**)

Figure 4. *Cont.*



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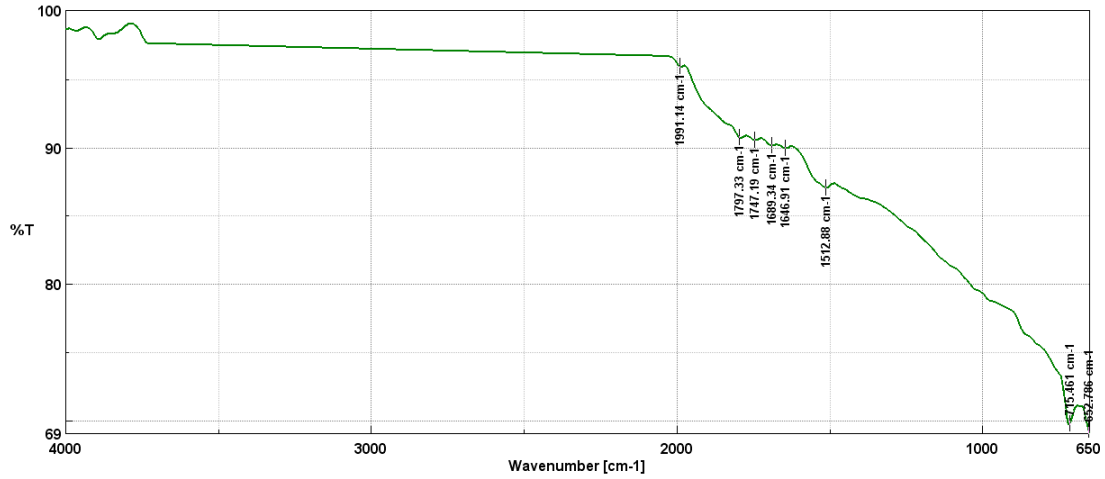

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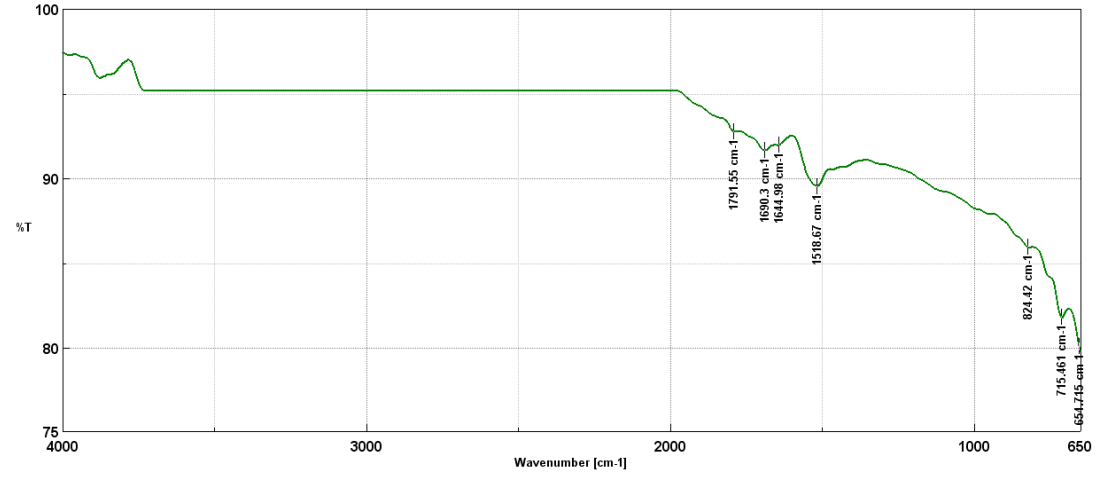
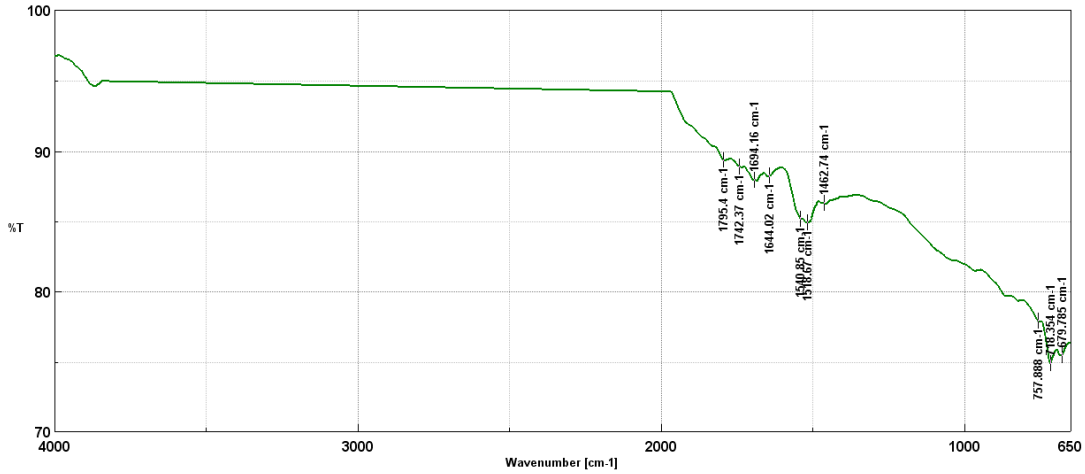


(**e**)

Figure 4. *Cont.*



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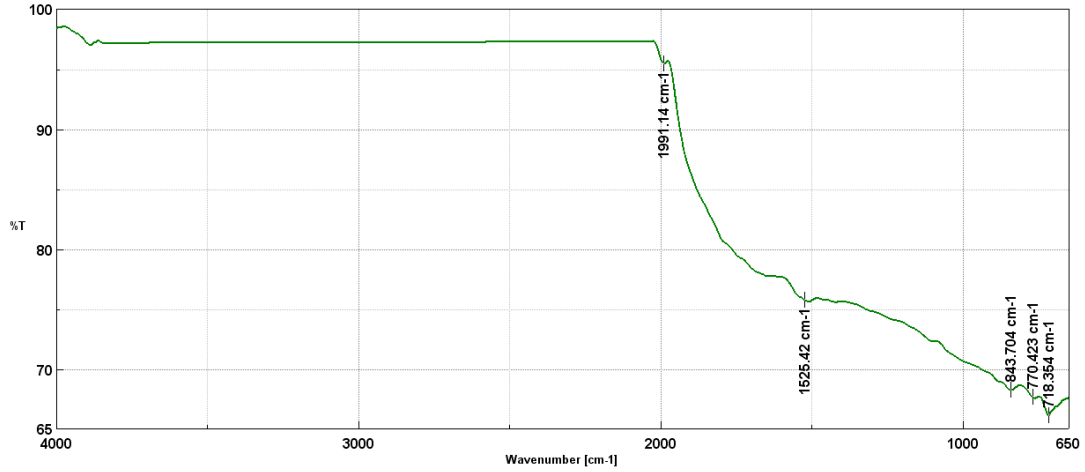


(**g**)

Figure 4. (**a**) The FTIR spectra of biochar from walnut shells obtained via pyrolysis at 400 °C. (**b**) The FTIR spectra of biochar from walnut shells obtained via pyrolysis at 500 °C. (**c**) The FTIR spectra of biochar from walnut shells obtained via pyrolysis at 600 °C. (**d**) The FTIR spectra of biochar from walnut shells obtained via pyrolysis at 700 °C. (**e**) The FTIR spectra of biochar from walnut shells obtained via pyrolysis at 800 °C. (**f**) The FTIR spectra of biochar from walnut shells obtained via pyrolysis at 900 °C. (**g**) The FTIR spectra of graphite.

FTIR spectroscopy allows for the analysis of functional groups present in biochars, which have a significant impact on their tribological properties, i.e., their ability to reduce friction and wear. FTIR analysis suggests the presence of specific functional groups but does not clearly define the structure of the material.

In the 400–900 °C range, the spectra contain numerous significant differences, both in terms of intensity and peak shifts corresponding to the presence of specific functional groups (e.g., C=O, C-O, O-H, Si-O). These subtle changes are crucial as it is the presence (or absence) of specific functional groups that can affect the adsorption of the additive on metallic surfaces and the formation of the lubricating film, with direct implications for tribological properties.

In [76], the authors analyzed the influence of different functional groups on the tribological properties of lubricants. It is shown that the presence of carboxyl, hydroxyl, and amine groups affects adsorption on metal surfaces, which translates into a reduction in the coefficient of friction and wear. Ref. [77] discussed how different functional groups affect the adsorption on titanium alloy surfaces and their tribological properties. Functional groups were found to play a key role in the formation of protective layers, reducing material

wear. In ref. [39], the authors showed how functional groups contained in a biochar additive with nanometric structures used in lubricating oils improved the tribological properties and produced better surface wetting compared to traditional additives.

Depending on the pyrolysis temperature, the chemical composition of biochars changes, which translates into their effectiveness as a lubricant component. A key aspect is oxygen groups, which affect the formation of protective lubricating layers that prevent surface wear. At 400 °C, FTIR spectra show a high content of carbonyl groups (C=O) in the range of 1974–1893 cm^{-1}, which can come from ketones, esters, and anhydrides. These groups can improve anti-seize and anti-wear properties by creating a stable lubricating layer. Additionally, the C=C band (~1683 cm^{-1}) indicates the presence of aromatic structures, and hydroxyl and ether groups (C-O in the range of 1275–1026 cm^{-1}) indicate high surface reactivity, which favors lubricating interactions.

A similar chemical composition is shown by biochars produced at a temperature of 500 °C, in which carbonyl groups (~1683 cm^{-1}), aromatic groups (~1559 cm^{-1}), and C-O groups (~1257–1029 cm^{-1}) are still present. At this temperature, bonds C-H are also present in methyl and methylene groups (1432, 1392 cm^{-1}), deformational bonds (C-H) in aromatic systems (971, 864 cm^{-1}), and deformational bonds (C-H) out-of-plane in aromatic rings (816–674 cm^{-1}).

Thanks to this, the material retains an appropriate number of active functional groups, which improve its ability to protect friction surfaces. As the pyrolysis temperature increases, above 600 °C, the oxygen functional groups gradually degrade, which leads to a decrease in lubricating properties. FTIR spectra indicate that the C=C bands start to dominate (~1520 cm^{-1} at 600 °C), which means that the structure becomes more graphitized and the number of reactive groups is reduced. Also present are the deforming bonds (C-H) in aromatic systems (867–680 cm^{-1}), stretching bonds (C=O) in ketones and amides (1791–1640 cm^{-1}), and bending bonds (C-H) in methyl and methylene groups (1325–1386 cm^{-1}). In addition, the decrease in the intensity of the C-O bands in the range of 1200–1000 cm^{-1} indicates the loss of phenolic and ether functional groups. Such a change means that biochars lose their ability to form a protective lubricating layer, which worsens their anti-wear and anti-scuffing properties.

During the pyrolysis of walnut shells at 700 °C, the following results were found: stretching bonds (C=O) in aromatic systems (aromatic quinones) at 1991 cm^{-1}; stretching bonds (C=O) in ketones and amides (1747–1797 cm^{-1}); stretching bonds (C=C) in aromatic rings at 1646–1689 cm^{-1}; deformational bonds (C-H) in aromatic systems at 1512 cm^{-1}; and deformational bonds (C-H) in aromatic systems at 715 and 652 cm^{-1}.

However, at 800 °C, the following results were found: stretching bonds (C=O) in ketones and amides at 1791 cm^{-1}; stretching bonds (C=C) in aromatic rings at 1644 and 1690 cm^{-1}; deforming bonds (C-H) in aromatic systems at 1518 cm^{-1}; deforming bonds (C-H) in aromatic systems at 824 cm^{-1}; and deforming bonds (C-H) in aromatic systems at 715 and 654 cm^{-1}.

At 900 °C, the following results were found: stretching bonds (C=O) in residual ketones at 1694–1795 cm^{-1}; stretching bonds (C=C) in aromatic rings at 1644 cm^{-1}, which means that the structure becomes more graphitized, and the number of reactive groups is reduced; deforming vibrations (C-H) in aromatic systems at 1518–1540 cm^{-1}; bending vibrations (C-H) in aromatic groups at 1462 cm^{-1}; and deforming vibrations (C-H) in aromatic rings (679–757 cm^{-1}).

The following were found for graphite: deformational bonds (C-H) in highly ordered aromatic systems at 718 cm^{-1}; deformational bonds (C-H) out-of-plane in graphitic systems at 770 cm^{-1}; stretching bonds (C=C) in highly ordered in aromatic rings at 1525 cm^{-1}; and stretching bonds (C=O) in residual carbonyl groups at 1991 cm^{-1}.

Biochars obtained at temperatures of 400 °C and 500 °C were selected for tribological studies because they offer the best compromise between chemical activity and structural stability.

The results of the chemical structure of the biochar produced via the pyrolysis of walnut shells at 400–900 °C and graphite using Raman spectroscopy are shown in Figure 5A,B.

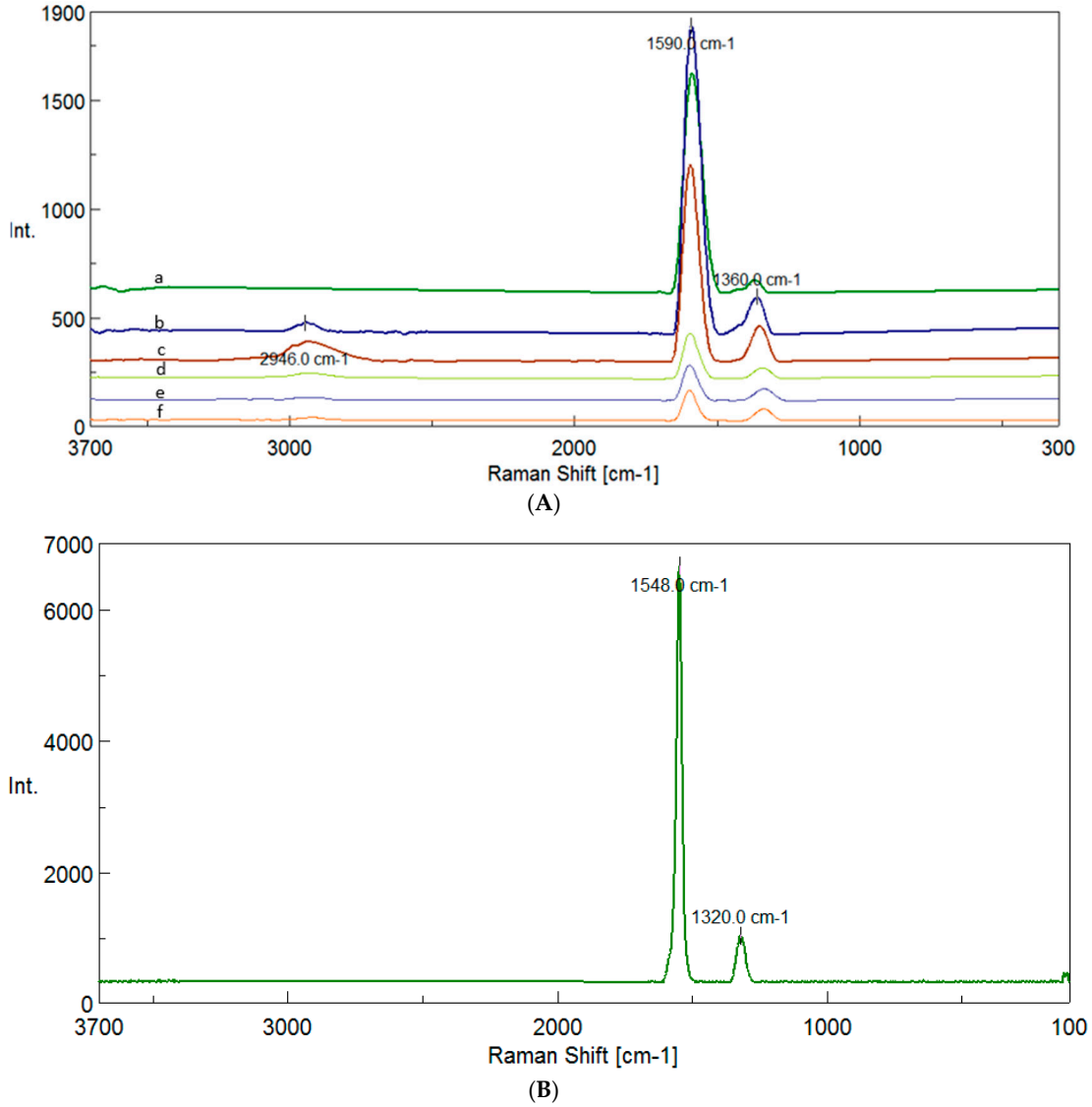


Figure 5. (**A**) The Raman spectra of biochar from walnut shell obtained in the pyrolysis process at the following temperatures: (a) 400 °C; (b) 500 °C; (c) 600 °C; (d) 700 °C; (e) 800 °C; (f) 900 °C. (**B**) The Raman spectra of graphite.

Raman spectroscopy allows us to determine how ordered the structure of biochars is and how many defects it contains. Two main bands can be distinguished in the tested materials: the G band around 1590 cm^{-1}, which indicates a well-ordered graphite structure; and the D band around 1360 cm^{-1}, indicating the presence of defects and less organized fragments. At temperatures of 500 °C and 600 °C, an additional 2D band appears in the range of about 2946 cm^{-1}, which is associated with the formation of graphene layers and may indicate a more ordered structure of the material. To assess the degree of ordering of biochars, the ratio of the D band intensity to the G band (I_D/I_G) is analyzed. The lower its value, the more ordered the material. At 400 °C, this ratio is 0.05, and at 500 °C, it increases to 0.12, which means that these materials are exceptionally well-ordered. In comparison, at 600 °C, the ratio is 0.18, and at higher temperatures, it gradually increases to 0.23 at 700 °C, 0.38 at 800 °C, and 0.44 at

900 °C, indicating an increasing number of defects and a more chaotic structure. Interestingly, commercial graphite has an I_D/I_G ratio of 0.16, which means that biochars obtained at 400 °C and 500 °C may be even more ordered than graphite. Since more structured materials have better tribological properties, biochars formed at temperatures of 400 °C and 500 °C were selected for the production of the lubricant. Their low I_D/I_G ratio means fewer defects, which improves their lubricity and makes them behave similarly to graphite. Additionally, the presence of a 2D band at 500 °C indicates the beginning of graphene layers formation, which can improve the stability of the structure and reduce friction. However, at temperatures above 600 °C, the number of defects increases, making the material less favorable from a tribological point of view.

Two main bands can be distinguished in the graphite: the G-band at around 1550 cm^{-1}, which indicates a well-ordered structure; and the D-band around 1320 cm^{-1}, indicating the presence of defects and less-ordered fragments.

Table 2 shows all FTIR peaks and characteristic bonds for biochar produced via pyrolysis at 400–900 °C and for graphite.

Table 2. FTIR spectra analysis of the produced biochars and graphite.

Pyrolysis Temperature (°C)	FTIR Peaks (cm^{-1})	Characteristics of Chemical Bonds
400	1974, 1931, 1893	Stretching (C=O) in anhydrides or carbonyl groups (aldehydes, ketones, and esters)
	1832, 1779	Stretching (C=O) in anhydrides or esters (intense carbonyl bands)
	1683	Stretching (C=C) in aromatic systems
	1594, 1503	Stretching (C=C) in aromatic rings (lignin, phenols)
	1434	Stretching (C-H) in methyl ($-CH_3$) and methylene ($-CH_2-$) groups
	1364	Bending (C-H) in methyl and methylene groups
	1275, 1216	Stretching (C-O) in alcohols, phenols, and ethers (cellulose derivatives)
	1026, 948	Stretching (C-O) in phenols and ethers
	860, 824, 761, 688	Deformational (C-H) out-of-plane in aromatic rings
500	1683	Stretching (C=O) in amides, ketones, and carbonyl groups
	1559	Stretching (C=C) in aromatic rings (lignin)
	1432, 1392	Bending (C-H) in methyl and methylene groups
	1257, 1188	Stretching (C-O) in phenols
	1070, 1029	Stretching (C-O) in alcohols and ethers
	971, 864	Deformational (C-H) in aromatic systems
	816, 758, 718, 674	Deformational (C-H) out-of-plane in aromatic rings
600	1791, 1693, 1640	Stretching (C=O) in ketones, amides, and lactones
	1520, 1437	Stretching (C=C) in aromatic rings
	1386, 1325	Bending (C-H) in methyl and methylene groups
	1121, 1017, 964	Stretching (C-O) in phenols and ethers
	867, 713, 680	Deforming (C-H) in aromatic systems

Table 2. *Cont.*

Pyrolysis Temperature (°C)	FTIR Peaks (cm^{-1})	Characteristics of Chemical Bonds
700	1991	Stretching (C=O) in aromatic systems (aromatic quinones)
	1797, 1747	Stretching (C=O) in ketones and amides
	1689, 1646	Stretching (C=C) in aromatic rings
	1512	Deformational (C-H) in aromatic systems
	715, 652	Deformational (C-H) in aromatic systems
800	1791	Stretching (C=O) in ketones and amides
	1690, 1644	Stretching (C=C) in aromatic rings
	1518	Deforming (C-H) in aromatic systems
	824	Deforming (C-H) in aromatic systems
	715, 654	Deforming (C-H) in aromatic systems
900	1795, 1742, 1694	Stretching (C=O) in residual ketones
	1644	Stretching (C=C) in aromatic rings
	1540, 1518	Deforming vibrations (C-H) in aromatic systems
	1462	Bending vibrations (C-H) in aromatic groups
	757, 718, 679	Deforming vibrations (C-H) in aromatic rings
Graphite	1991	Stretching (C=O) in residual carbonyl groups
	1525	Stretching (C=C) in highly ordered aromatic rings
	770	Deformational (C-H) out-of-plane in graphitic systems
	718	Deformational (C-H) in highly ordered aromatic systems

3.2. Tribological Tests

A measure of the anti-scuffing characteristics of the tested greases under a scuffing operating regime constitutes the limiting pressure of seizure (p_{oz}). The experimental data determined for this parameter are presented in Figure 6.

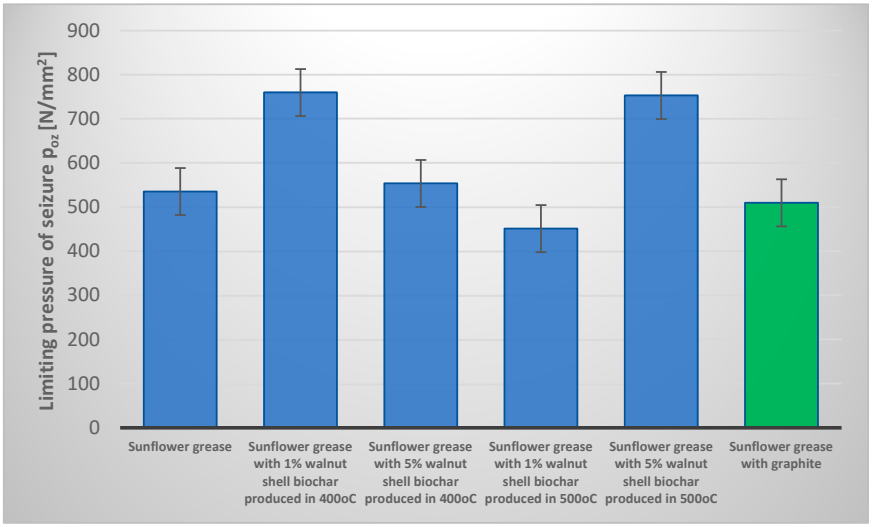


Figure 6. Limiting pressure of seizure of tribological system lubricated with greases based on sunflower oil with various contents of biochar additive.

The obtained data on the limiting pressure of seizure indicated various degrees of anti-scuffing characteristics for the studied lubricating greases. Depending on the quantity of biochar additives used, the level of the calculated factor p_{oz}, characterizing the anti-scuffing characteristics of the investigated lubricants, changes considerably. In this paper, we introduce a modifying additive into the structure of sunflower grease in the form of a biochar produced via pyrolysis at 400 °C and 500 °C from walnut shells. Modification of the vegetable grease with 1% biochar produced at 400 °C from walnut shells increased the anti-seizure properties, characterized by the p_{oz} parameter, by 42%. Modification of vegetable grease with 5% biochar additive produced at 400 °C did not show such favorable changes in the parameter in question in comparison to the base grease (there was an increase in the parameter characterizing the anti-seizure properties of only 3%). In contrast, the addition of a walnut shell pyrolysis additive at 500 °C to the base grease indicated that the positive effect of this additive is observed at higher additive concentrations. Modification of the vegetable grease with 1% biochar from walnut shells produced at 500 °C had an antagonistic effect on the anti-scuffing properties of the tested lubricating compositions. In this case, an almost 16% decrease in the value of the p_{oz} parameter was achieved compared to the results obtained for the base grease without the modifying additive. Modification of sunflower grease with 5% biochar additive produced via pyrolysis at 500 °C showed a positive effect on the anti-scuffing properties of the tested lubricating composition. This amount of additive resulted in a 41% increase in the p_{oz} parameter compared to the results obtained for the base vegetable grease.

The use of biochar additives from walnut shells in vegetable greases was characterized by a more effective anti-seizure effect than the use of graphite as a lubricating additive in vegetable greases. The p_{oz} parameter provides information about the pressure prevailing in the friction zone at the moment of seizure. On the basis of the obtained test results, it can be concluded that the use of a biochar additive made from walnut shells has a positive effect on the formation of highly resistant structures that effectively protect machine and equipment components from seizure. The incorporation of walnut shell biochar into the structure of the lubricant increases the resistance of the surface layer to seizure processes, and the nature of the lubricating film formed promotes a significant reduction in wear.

The walnut shell biochar additive produced via pyrolysis is a typical EP additive introduced into a lubricant when very high friction pressures occur between cooperating surfaces. It forms a firmly adherent film on the metal surface by reacting with the layer of iron oxides adhering to the metal surface through the high pressures and temperatures accompany metal–metal friction processes. If the adherent layer of the additive is destroyed, it is rapidly reconstituted. The additive's mechanism of action is based on the phenomenon of absorption of the biochar additive molecules into the thickener molecules, which attaches to the chain of the oil base to form structures resistant to seizure processes. The addition of biochar in the lubricant prevents the excessive wear of mating surfaces and reduces the risk of seizure. The applied biochar additive can also act as a friction modifier. In the range of low sliding speeds, with insignificant specific pressures and low oil viscosity, fluid lubrication can easily change to mixed friction.

In order to prevent friction vibrations and scrapes and to reduce friction forces, substances are usually used that are effective at temperatures at which EP additives are not yet active, form single-molecule layers of physically adsorbed, oil-soluble additives, and reduce friction through re-aggregated layers that have a significantly lower coefficient of friction than a typical oil without additives. Particularly important is the biochar additive, which belongs to the group of additives with a mechanical effect of great importance at moderate temperatures and pressures, where mixed friction occurs.

The anti-wear properties of the evaluated plant-based greases were validated by calculating the limiting load of wear ($G_{oz/40}$) of the tribological system lubricated with the assessed greases. The experimental data are presented in Figure 7. The dependence of the friction factor on the sliding speed of the tested greases is presented in Figure 8.

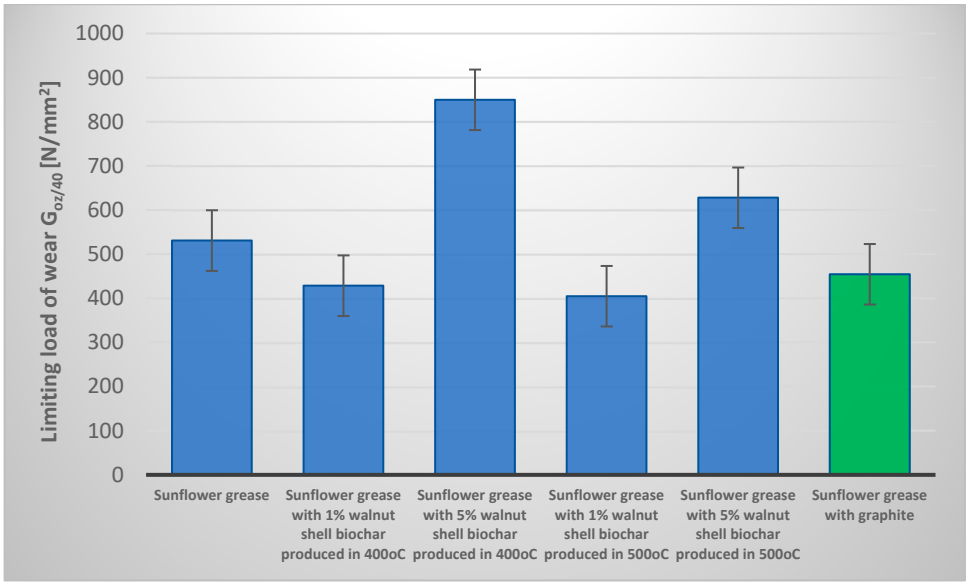


Figure 7. Limiting load of wear of a tribological system lubricated with greases based on sunflower oil with various quantities of biochar additive.

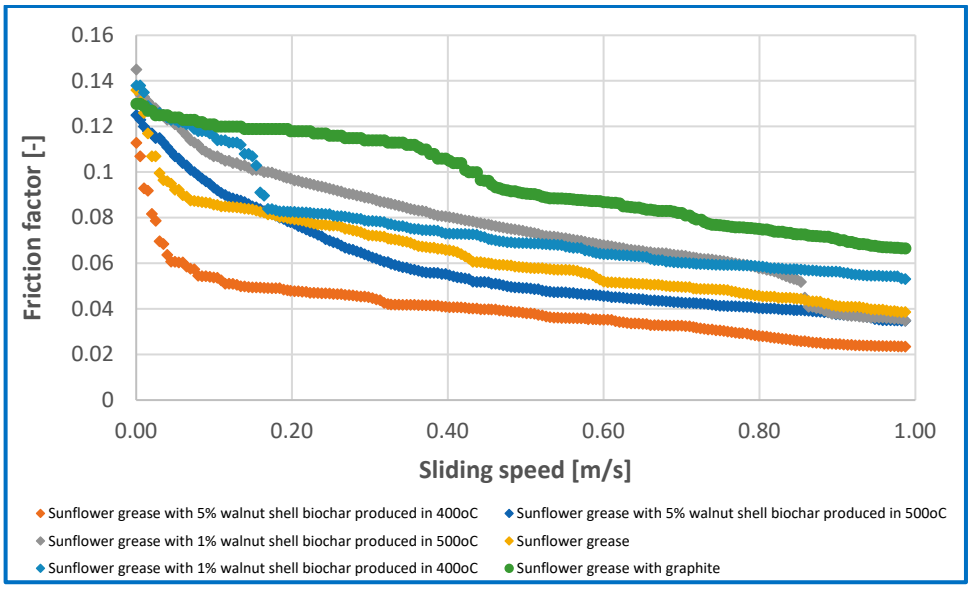


Figure 8. The friction factor from the sliding speed for compositions modified with different quantities of biochar additive.

Tribological tests, undertaken at a constant friction node load of 392.4 N, for the produced vegetable greases modified with different amounts of biochar from walnut shells produced via pyrolysis at 400 °C and 500 °C showed that the used additive changes the ability of the greases to protect the friction node against wear.

The durability of the boundary layer is indicated by the wear index $G_{oz/40}$. The higher the index, the greater the durability of the boundary layer and the reduction in wear. The sunflower oil-based vegetable grease had a $G_{oz/40}$ parameter value of 530.82 N/mm^2. Introducing 1% walnut shell biochar produced at 400 °C into the structure of the vegetable

grease resulted in a significant reduction in the anti-wear protection of steel friction nodes by 19% compared to the base grease without the modifier. On the other hand, increasing the concentration of the biochar additive in the lubricant to 5% resulted in a significant increase in the value of the parameter in question, which indicates the level of anti-wear properties. The value of the $G_{oz/40}$ parameter for this composition was 849.85 N/mm^2, which was 60% higher than the value of this parameter for the base grease, which indicates the positive effect of the additive used on the level of the anti-wear protection of the friction nodes lubricated with sunflower oil-based vegetable greases. Modification of the vegetable grease with a 1% biochar additive made from walnut shells obtained via pyrolysis at 500 °C did not produce positive results. In this case, a 24% decrease in the value of the $G_{oz/40}$ parameter was observed, indicating a deterioration in the anti-wear protection of friction nodes lubricated with this composition, while an increase in the concentration of the biochar additive produced at 500 °C increased the value of the parameter in question, improving the anti-wear properties of the tested lubricating composition by 18.5%. The use of the biochar additive at a concentration of 5% showed a significant effect on improving the anti-wear properties of vegetable greases. Increasing the anti-wear protection of steel friction nodes after the use of biochar additives for vegetable greases is more effective than the use of graphite as an anti-wear additive. Lubricants modified with a minimum of 5% biochar additive are lubricants that effectively protect the friction node against wear under constant tribosystem load. Introducing a 1% biochar additive made from walnut shells into the structure of a vegetable grease increases the coefficient of friction in relation to the results for the base grease without the modifier, while the introduction of a 5% additive into the structure of the grease reduces the coefficient of friction of the tested greases, indicating the effective protection of the friction node against wear.

The observed difference—i.e., lower wear in the sample containing 5% walnut biochar produced at 400 °C, even though the limiting pressure of seizure is not the highest—may be due to several factors. First, a higher amount of additive may result in a better "sealing" of the contact zone between surfaces, which reduces minor damage and reduces wear and seizure, even if the lubricating film does not significantly increase the load carrying capacity of the system. Second, with a higher concentration of walnut biochar additive in the vegetable grease, the biochar particles may be less evenly distributed in the grease, which may locally reduce seizure resistance, but at the same time, it may promote the formation of a more effective protective film to reduce wear. Third, it should be taken into account that different tests emphasize different lubrication mechanisms; mixed or hydrodynamic lubrication may dominate in wear tests, where the chemical nature of the additive is more important, while the seizure test more strongly exposes the film's resistance to sudden overloads. Therefore, lower wear at a slightly lower seizure limit load is not a contradiction in terms but is due to different mechanisms dominating in the two tests.

The main aim of this study was to evaluate the tribological and rheological properties of walnut biochar as a lubricating additive, while the detailed characterization of the wear surface, including the confirmation of the presence of a protective film, was planned as a further stage in the research.

Therefore, our information on the possible formation of a lubricating film by biochar particles currently takes the form of a hypothesis based on the behavior of the lubricating system and the results of friction and wear measurements.

In order to characterize the tribological wear mechanism, the images of the wear scars were taken for all tested lubricants (Table 3), and the percentage contents of elements such as C, O, Si, Cr, and Fe in the wear scar were determined (Table 4) using the SEM-EDS technique.

Table 3. Image of wear scars of tested vegetable lubricants after tribological tests.

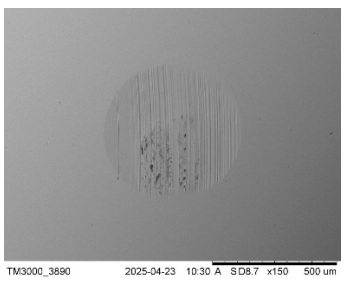
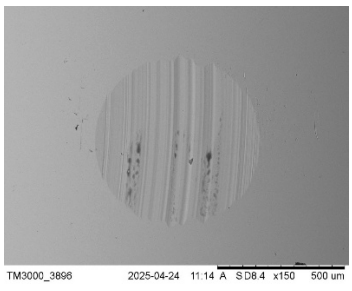
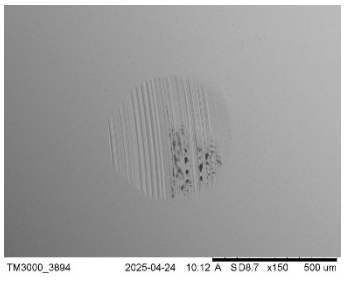
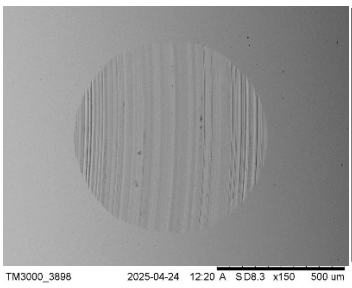
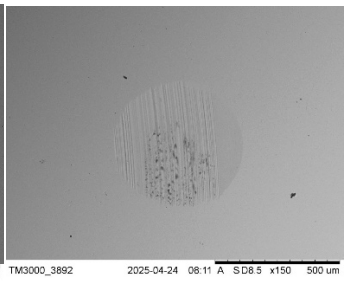
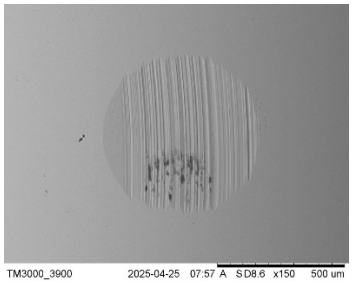
Tested lubricants	A	B	C
Image of wear scars after tribological tests			
Tested lubricants	D	E	F
Image of wear scars after tribological tests			

Table 4. Chemical composition of tested vegetable lubricants after tribological tests.

Tested Vegetable Grease	The Percentage Content of Elements in Wear Scars				
	C	**O**	**Si**	**Cr**	**Fe**
A	15.366	23.494	5.113	0.981	55.045
B	19.949	22.254	3.856	0.867	53.074
C	15.968	4.947	0.817	1.249	77.018
D	15.691	18.867	3.826	0.977	60.639
E	17.175	13.097	3.288	1.113	65.327
F	17.392	26.102	6.386	0.844	49.276

In all cases, the predominant mechanism of tribological wear was the so-called *gouging* processs, in which plastic displacement and the detachment of material occurs due to high contact forces and the relative movement of solids. As a result, deep grooves, cracks, or furrows form on the surface. This type of mechanism occurs at high pressures and hard irregularities. This is typical behavior in a situation where there is intense sliding contact. This type of wear can be a precursor to other forms of wear, such as adhesive or fatigue wear.

As part of the study of the surfaces of tested lubricating compositions after tribological tests, an analysis of the elemental composition was carried out using the EDS method, both in tribologically undamaged areas and in the tribological wear scar. The results demonstrate significant differences in the chemical composition of the analyzed areas, particularly in the content of elements such as oxygen (O), silicone (Si), and carbon (C), which occurred in significantly higher concentrations in the wear zone than in areas where tribological wear was not observed. The elevated carbon content and the presence of oxygen and silicon may indicate the adsorption of biochar particles on the steel surface during frictional contact, leading to the formation of a boundary layer containing its degradation products, such as organic oxides or the residues of functional groups (e.g., carbonyl, hydroxyl). At the same time, in the elemental composition of the wear surface, the presence of silicon

was detected, which does not originate from the biochar but from the amorphous silica (Aerosil®) present in the lubricant structure acting as a thickener; under working conditions, it can move into the frictional contact zone, resulting in the detection of elements such as Si and O in the wear scar. The presence of oxygen in the wear layer may therefore be the result of overlapping processes, namely, the functional groups in the biochar, the presence of the thickener (SiO_2), and tribo-oxidation processes on the steel surface resulting in the formation of iron oxides. The variation in iron (Fe) content between wear-damaged and undamaged areas via the tribological processes confirms the local wear of the carrier material, which correlates with microscopic observations and tribological test results. Altogether, the analysis of the EDS elemental composition confirms the hypothesis that the biochar acts as an active EP/AW-type additive, which, due to its chemical reactivity, participates in the modification of the tribological surface, forming a protective barrier limiting direct contact at the metal–metal interface and thus positively influencing on the lubricating properties of the tested compositions.

Analysis of the elemental composition using the EDS method carried out for the tested lubricating compositions after the tribological tests showed a clear variation in oxygen (O) and silicon (Si) content in the wear scar depending on the type of additive used and its amount. In the case of the reference sample, i.e., a base grease with no modifier in its composition, the high values of oxygen (23%) and silicone (5%) are due to the lack of an active lubricating additive, which results in the full exposure of the lubricant components to wear and tribo-oxidation processes. The introduction of graphite as a modifying additive into the base lubricant results in higher oxygen (26%) and silicone (6.4%) contents, suggesting that graphite, despite its good conductivity and lubricating properties, does not form a permanent protective layer on the steel surface but, in fact, allows the thickener to migrate more intensively into the friction contact zone.

Different results were obtained for greases containing a biochar additive in their composition. The introduction of 1% walnut shell biochar into the structure of the lubricant causes a reduction in oxygen content (to 18.8% for biochar obtained via pyrolysis at 500 °C and to 22.2% for biochar obtained via pyrolysis at 400 °C) and silicone content (to 3.8% in both cases), indicating the partial protection of the analyzed surface from exposure to the lubricant components. The applied biochar additive, thanks to the presence of functional groups and a more developed surface, forms a protective layer limiting the contact between the metal and the lubricant components and reducing the possibility of the intense oxidation of the surface subjected to intensive tribological processes.

Even more pronounced effects are observed for greases containing 5% walnut shell biochar in their composition, where the oxygen content decreases to 13.3% (biochar obtained from walnut shellss via pyrolysis at 500 °C) and 4.9% (biochar obtained via pyrolysis at 400 °C), and the silicone content decreases to 3.2% and 0.8%, respectively. Thus, the clear reduction in oxygen and silicone content indicates the more effective coverage of the analyzed surface by the biochar layer and a reduction in the migration of lubricant components into the wear zone.

The differences between biochar obtained at 400 °C and 500 °C can be explained by the degree of carbonization of the material: biochar produced at 400 °C contains more oxygen groups, which can lead to a higher proportion of oxygen in the surface layer; while biochar pyrolyzed at 500 °C is more decarboxylated and less polar, so its protective layer contains less oxygen components. At the same time, the higher pyrolysis temperature favors the formation of more ordered carbon structures, which may improve the stability of the protective mechanism against tribological wear processes.

The biochar produced from walnut shells effectively reduces the availability of silica and oil degradation products to the steel surface, reducing the oxygen and silicone content

of the wear layer. The effectiveness of this protection increases with the increasing amount of biochar additive and the degree of carbonization, confirming its active role as an EP/AW-type additive in the tested lubricating compositions.

The results show that the biochar additives made from walnut shells can be used as AW additives in vegetable-based greases, especially at higher additive concentrations in greases with vegetable oils as the oil base. The purpose of AW additives is to prevent excessive wear on cooperating friction surfaces and to reduce the coefficient of friction. Their mechanism of action is to form a permanent film of polar additive molecules on the metal surface. This film adheres to the metal surface without forming permanent chemical bonds with it. The additive molecules form chemical bonds with the metal surface or are bound to the substrate by intermolecular van der Waals forces.

The adsorption mechanism of the polar AW additive molecules is that the upper part of the additive molecules attach to the metal particles, while the lower, hydrocarbon part is directed towards the oil, forming a so-called oil film. Both cooperating faces of the friction association are covered by such a tightly adherent film, which reduces the possibility of direct contact between their surfaces and thus reduces wear. The layer of additive particles forming on the cooperating metal surfaces, the so-called oil film, facilitates their mutual displacement, thus also reducing the coefficient of friction.

3.3. Tests of Rheological Properties of Lubricating Greases

During tribological tests, the dynamic viscosity tests were carried out for lubricating compositions modified with different quantities of biochar additives. The dependence of dynamic viscosity on sliding speed is shown in Figure 9.

For each tested lubricant, the dependence of dynamic viscosity on sliding speed was determined. The presented test results testify to changes in the tribological properties of the tested lubricating compositions. Based on the changes in dynamic viscosity, it is possible to predict what effect the introduced biochar additives will have on the anti-wear properties of vegetable greases. The greatest changes in the dynamic viscosity of the tested lubricants were observed at low sliding speeds—i.e., those up to 0.2 m/s.

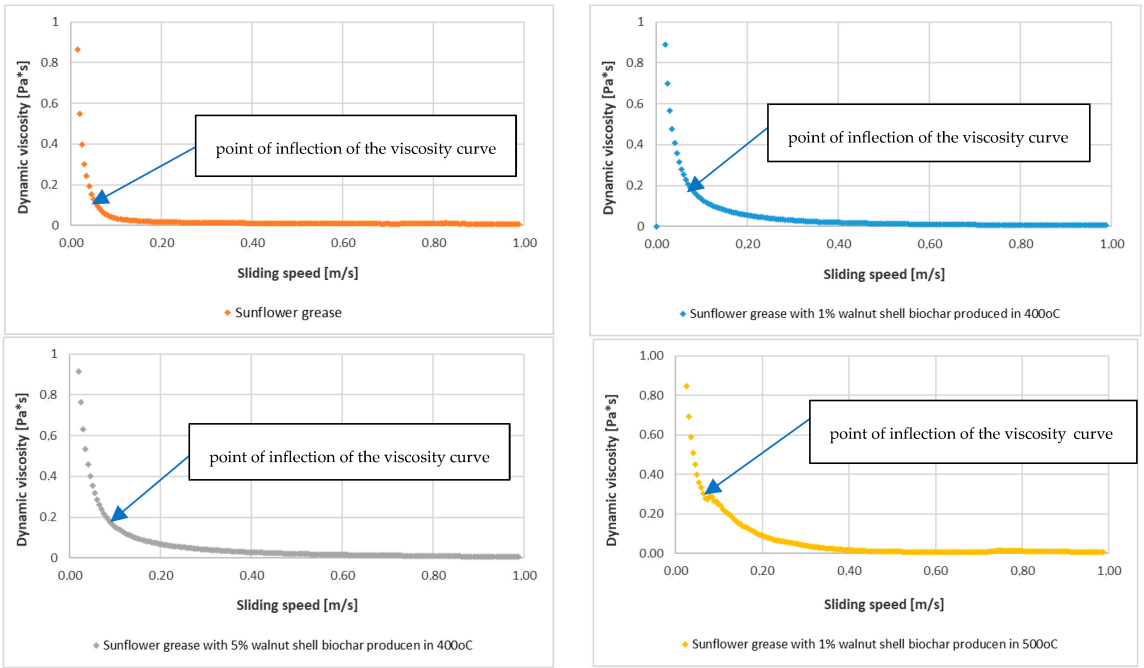


Figure 9. *Cont.*

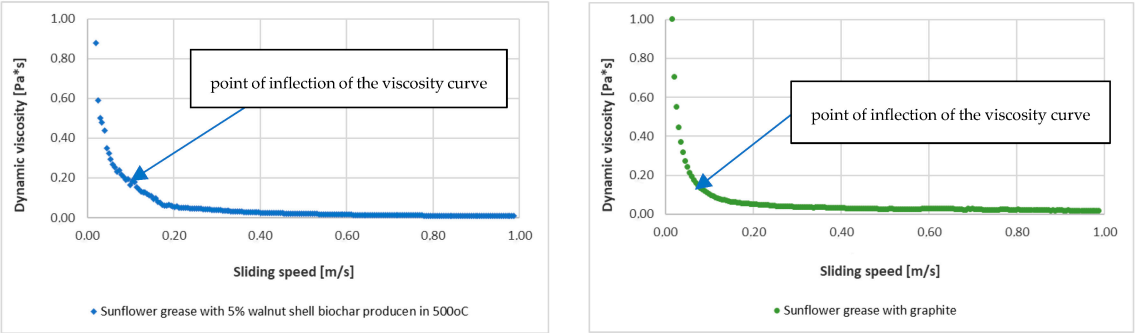


Figure 9. Dependence of dynamic viscosity on sliding speed for lubricating compositions modified with different quantities of biochar additive.

The introduction of a biochar additive to sunflower grease produced via pyrolysis at 400 °C and 500 °C from walnut shells changes the rheological properties of the biochar grease. Changes in the viscosity curve for grease B and grease D, in which 1% of the modifying additive was introduced, testify to the much weaker anti-wear properties of these compositions than for the base composition without the additive. In these cases, the inflection of the curve occurs at higher values of dynamic viscosity. On the other hand, for compositions C and E, the change in dynamic viscosity indicates better anti-wear protection of the friction node. In these two cases, the inflection of the viscosity curve occurs at lower dynamic viscosity values. On the other hand, the viscosity changes for grease F, which was modified with graphite, indicate the anti-wear properties fitting in between compositions modified with 1% biochar additive and compositions modified with 5% biochar additive. Thus, the level of anti-wear protection characterized by the $G_{oz/40}$ parameter can be verified on the basis of changes in the dynamic viscosity of the lubricants subjected to tribological tests.

The way in which the additive particles are incorporated into the base oil chain is indicative of the rheological properties of biochar greases. The introduction of a minimum of 5% biochar additive causes the distance and bond strength between the oil, the thickener, and the used additive to decrease, resulting in a stabiliation of the structure of the tested lubricants and an increased resistance to degradation due to shear forces.

From the results shown in Figure 9, it can be seen that the grease modified with 1% walnut biochar produced at 400 °C has a higher viscosity, but its wear resistance is not the most favorable. This may indicate that mixed lubrication dominates under the conditions analyzed, where it is not so much the dynamic viscosity that is crucial but the chemical interaction of the additive with the metal surface and its ability to form a protective adsorption layer.

Therefore, although viscosity is an important parameter from the point of view of hydrodynamic lubrication, in the case of the analyzed greases supplemented with walnut biochar, other factors, such as the chemical composition, the presence of functional groups, or the dispersibility of the additive, may be decisive, affecting the formation of the lubricating film and tribological efficiency.

4. Conclusions

Based on the results of the tests, it was found that the lubricating and rheological properties of the tested lubricating compositions changed significantly depending on the amount of the modifying additive used.

The tests were carried out on a four-ball apparatus at concentrated contact. Vegetable lubricating compositions containing 1 and 5% of the modifying additive were analyzed. The obtained results unequivocally confirmed the positive effect of the additive used in the grease during the friction of the steel tribosystems.

The determined values of the limiting pressure of seizure, determining the anti-seizure properties in scuffing conditions, showed that the use of 1% biochar produced via pyrolysis at 400 °C from walnut shells and 5% biochar produced at 500 °C—as an modifying additive of tested lubricating compositions—is most effective in influencing the change in the anti-seizure properties of the greases used in the experiment.

The use of the optimum amount of the modifying additive causes the formation of a low-friction surface layer on the steel surface, which is resistant to high unit loads, resulting in an increase in the durability and efficiency of many sliding associations.

The modification of vegetable plastic greases with biochar from walnut shells contributes to improving their lubricating properties. The base oil (sunflower oil) used in the experiment interacts synergistically with the walnut shell biochar, causing an increase in the indices describing the tribological properties of the tested lubricating compositions.

The introduction of walnut shell biochar into the structure of the plastic grease as a modifying additive contributes to the formation of a protective film on the surface, which increases the resistance of the tribosystem to seizure. As a result of the improved boundary layer properties, the beginning of the scuffing process occurs at higher loads on the friction node.

Under the influence of the modifier used, especially at a concentration of 5%, there is an increase in the G_{oz} parameter, which indicates a high resistance to boundary layer disruption. This indicates that the additive used has a positive effect on improving the tribological characteristics of vegetable plastic greases.

The introduction of the biochar additive into the structure of the vegetable plastic grease causes a decrease in dynamic viscosity with an increase in sliding speed, which consequently leads to a decrease in the friction coefficient for greases modified with a 5% biochar additive produced from walnut shells, and an increase in the anti-wear protection of steel friction nodes lubricated with vegetable lubricating compositions containing walnut shell biochar produced via pyrolysis at 400 °C and 500 °C.

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