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# Biochar from Wood Waste: Textural and Structural Properties Depending on Production Method

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Biochar is a carbonaceous product from biomass obtained by heat treatment of organic materials such as wood, by-products of wood processing, and plant residues. Biochar can be obtained as a result of thermal processing, which can be divided into three main processes: pyrolysis, torrefaction, and hydrothermal carbonization. The resulting biochar has different composition, structural and textural properties, strength, etc. Despite of numerous studies, the investigation of biochar production and properties are of great interest. Modification of carbonaceous products obtained from biomass is one of the actual tasks to produce the tailor-made materials. This article presents a comparison of the characteristics of biochar obtained by three methods: pyrolysis, torrefaction, and hydrothermal carbonization. Another task was the evaluation of the modifier addition to biochar to form materials with higher surface area and abrasion resistance for catalyst applications. The biochar obtained by the torrefaction from different lignocellulosic feedstock (hydrolysed and non-hydrolysed pine and birch sawdust) was found to have the highest carbonization degree, and the highest specific surface area. It was found that the additional modification of biochar with silica provides an increase its abrasion resistance, and higher porosity that allows the material obtained to be used for different purposes (as filler and binder for the construction materials, catalyst support etc.).

## 1. Introduction

Biochar is a carbonaceous product from biomass. Biochar is widely used as an energy supplier, sorbent, catalyst support, etc. The composition and properties of biochar strongly depend on the feedstock nature and the preparation methods. Among the methods of bio-coal obtaining, pyrolysis, torrefaction, and hydrothermal carbonization can be highlighted.

Pyrolysis destroys chemical bonds in the biomass components with the formation of new compounds in the absence of oxygen. It has high flexibility in biomass processing. This process thermochemically converts biomass into liquid (bio-oil), charcoal (bio-coal), and non-condensable gases by heating to about 480 °C or more (Pecchi and Baratieri, 2019). The pyrolysis product distribution depends on the composition of the feedstock. A high cellulose content in the raw material leads to a higher yield of liquid products, a high hemicellulose content leads to a high gas yield, and a high lignin content leads to a larger amount of solid residue (Kumar et al., 2020). The regulation of the process parameters also changes the proportion of products received. The low temperature of the process and the longer residence time contribute to the formation of coal. High temperature and longer residence time increase gas formation, while moderate temperature and short residence time ensure high bio-oil yield (Pecchi and Baratieri, 2019).

Torrefaction is defined as the thermal process of converting biomass into bio-coal (Hu et al., 2019). Torrefaction is a mild pyrolysis process that occurs at temperatures from 200 to 300 °C under conditions of oxygen deficiency for an appropriate duration (0.5-2 h). In the torrefaction process, the components of hemicellulose, lignin, and cellulose decompose and a large number of volatile compounds are formed. The yield of biochar during the torrefaction of hemicellulose is usually lower compared to cellulose (Zheng et al., 2015) and lignin (Chen et al., 2015). The gaseous products of torrefaction are mainly $CO_2$ and CO, with a small amount of $CH_4$ and $H_2$ (Chen et al., 2018). Torrefied biomass has lower atomic ratios of O/C and H/C, which makes it similar to coal. The

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parameters of the torrefaction process, such as temperature, feedstock residence time, heating rate, moisture content in the biomass, and particle size, have a direct effect on the final properties of the torrefied biomass (Abdulyekeen et al., 2021).

The hydrothermal carbonization process, also known as wet torrefaction, is usually performed in the subcritical water temperature range (180-250 °C) at the appropriate pressure. Hydrocoal is a material obtained as a result of the carbonization process. It resembles brown coal in its elemental composition and calorific value (Ghanim et al., 2017). The high moisture content in the biomass is especially favorable for this process since there is no need for pre-drying. In addition, coal obtained using the carbonization process has a reduced ash content (Makela et al., 2015).

Conversion of biomass waste into useful material like biochar is a prospective method of waste valorization. The development of biochar production technology makes it possible to produce nano-scale biochar with an enhanced surface area and structural behaviors (Sharma et al., 2023). However, further research is needed to provide the high yield of biochar, as well as the development of the methods for production of carbon-containing composites. The current techniques for biochar production from waste need to be improved. The key area for enhancement is the efficiency of the carbonization process, optimizing the process conditions, developing new approaches for carbonization, etc. This will allow the biochar yield and its quality to be increased.

Moreover, the modification of biochar can be carried out to obtain the tailoring materials. Sulfur acid, alkali and alkali-earth oxides, metal particles are widely used among the modifiers (Stepacheva et al., 2023a). Modification of biochar with metal particles is used for the formation of catalytically active materials (Wang et al., 2021). The addition of silicon in the form of Si or SIC can be used for environmental or agricultural application (Wang et al., 2019). In this work, the effect of the addition of modifying agents (metal salt and alkoxysilane) on the properties of biochar was studied to obtain the tailored materials for construction and catalysis. The systematic study of the influence of biomass type, carbonization methods, and modifying agent on the properties of biochar specifies the scientific contribution and novelty of this work.

## 2. Materials and Methods

In the current work, the comparison of the biochars from birch and pine sawdust obtained by pyrolysis, torrefaction, and hydrothermal carbonization was carried out. The biochar yield, specific surface area, carbon content, and abrasion resistance were estimated when the initial sawdust, and solid residues after hydrolysis and liquefaction were used as a feedstock.

### 2.1 Feedstock composition

Pine and birch sawdust were purchased from the harvesting company of Tver region, Russia, and used as a feedstock. The feedstock was dried at 90 ± 5 °C for 12 h and milled until the mesh of 0.25 mm. The sawdust was analyzed to estimate the elemental composition, fractional composition, humidity, and ash content. The humidity and ash content were estimated according to the standard procedure. The elemental composition was studied using CHNS-O EMA502 analyzer (VELP Scientifica, Italy). The content of extractives was estimated using the Soxhlet extraction apparatus according to Ostroukhova et al. (2018). Analysis of cellulose, hemicelluloses, and lignin content was carried out according to RU2534067 and EA005492 Patents. The composition of the wood waste used in this work is presented in Table 1.

*Table 1: Composition of wood waste*

| Parameter | Sawdust | |
|---|---|---|
| | pinewood | birch |
| Humidity, wt. % | 7.0 | 5.2 |
| Ash content, wt. % | 3.6 | 1.4 |
| Cellulose, wt. % | 53.1 | 39.5 |
| Hemicelluloses, wt. % | 7.9 | 17.9 |
| Lignin, wt. % | 25.4 | 31.1 |
| Extractives, wt. % | 6.3 | 4.9 |
| C, wt. % | 48.8 | 50.2 |
| H, wt. % | 6.1 | 6.0 |
| N, wt. % | 0.1 | 0.6 |
| O, wt. % | 44.6 | 42.2 |

### 2.2 Biochar production

For all biochar production processes, a fraction of air-dry sawdust with a particle size of 1-2 mm was used. The process temperature was chosen according to the thermogravimetric analysis performed in our previous studies (Lugovoy et al., 2018). The pyrolysis of sawdust was carried out using the experimental setup (Stepacheva et al., 2023b) using nitrogen as a gas carrier. The process was carried out for 50 min at a temperature of 500 °C. The torrefaction of sawdust was carried out using the same experimental setup as for pyrolysis. The process was carried out for 3 h at a temperature of 250 °C. The hydrothermal carbonization was performed in a stainless-steel reactor-autoclave (Parr Instrument, USA) at 250 °C and auto pressure (ca. 4.0 MPa). Feedstock to water ratio was 1 : 4 by weight. The carbonization was carried out for 3 h. Besides the initial feedstock, the hydrolyzed sawdust with lignin content over 95 wt.% and the solid residue after the sawdust liquefaction were used for biochar production.
For the production of functionalized biochar, the wetness impregnation of the feedstock with the aqueous solutions of modifying agents was carried out. 3 g of the air-dry feedstock were stirred with 10 mL of the solutions of cobalt nitrate, iron nitrate, and tetraethoxysilane (TEOS) at room temperature. The concentration of the modifying agent was calculated to provide the addition of 5 wt. % of Co, Fe, and $SiO_2$ respectively to the biochar. The mixture was evaporated at 100 ± 5 °C and the solid residue was dried in the air at 90 ± 5 °C for 12 h.

### 2.3 Biochar characterization

The characterization of the biochar obtained was carried out to analyze the specific surface area, carbon content, and abrasion resistance. To estimate the porosity of the bio-coal, the analysis by the low-temperature nitrogen physisorption was carried out using Beckman Coulter SA 3100 analyzer (Coulter Corporation, USA) equipped with Beckmann Coulter SA-PREP (Coulter Corporation, USA) apparatus for sample preparation. Before the analysis, the samples were outgassed in the Beckmann Coulter SA-PREP (Coulter Corporation, Brea, CA, USA) apparatus. The concentration of carbon in the biochar was measured using CHNS-O EMA502 analyzer (VELP Scientifica, Italy). To estimate the concentration of modifiers (Fe, Co, and Si), the analysis was performed by the X-ray fluorescent analysis using Zeiss Jena VRA-30 spectrometer (Carl Zeiss Industrielle Messtechnik GmbH, Germany). The mechanical abrasion resistance of biochar tablets was studied following the ASTM D 4058-96 method using Attrition and Abrasion Tester (Vinci Technologies, France) equipped with a cylindrical stainless steel drum nozzle.

## 3. Results and Discussion

To form biochar with the specified characteristics, the influence of the biomass carbonization method was studied. Biocoal obtained from hydrolyzed and non-hydrolyzed biomass, as well as the solid residue of liquefaction of wood waste, were studied. Three main methods were used to produce bio-coal: pyrolysis, torrefaction, and hydrothermal carbonization. The yield of biochar depending on the preparation method is presented in Figure 1. The highest yield of biochar was obtained while using the torrefaction method. Slow heating of the feedstock at low temperatures allows the gasification processes to proceed at a lower rate. Preliminary hydrolysis leads to an increase in the biochar yield in all cases. This is due to the higher content of lignin which is carbonized in a better way compared to celluloses and hemicelluloses. For the liquified residue, in contrast, the yield of biochar decreases compared to the initial sawdust. During the liquefaction, the partial hydrolysis and hydrogenolysis of the biomass components facilitate the formation of gaseous products during the carbonization.

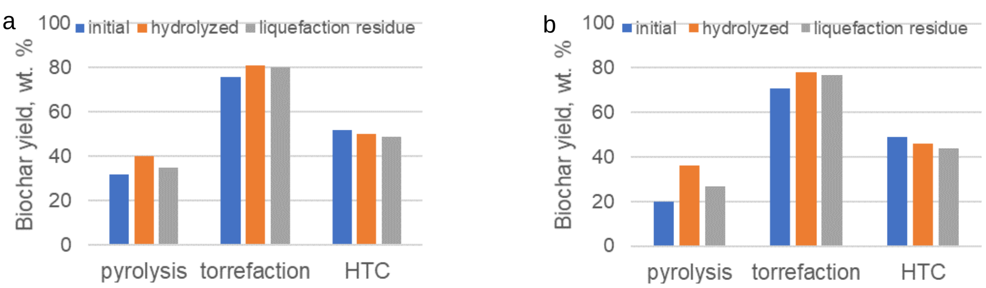


*Figure 1: Biochar yield depending on the preparation method for birch sawdust (a) and pine sawdust (b)*

Tables 1-3 present a comparison of carbon content, specific surface area, and abrasion resistance for biochar obtained by various methods from hydrolyzed and non-hydrolyzed sawdust, and liquefaction residue. It can be noted that for hydrolyzed and non-hydrolyzed biomass, as well as for solid residue of liquefaction, the largest surface area was noted using torrefaction and hydrothermal carbonation methods. At the same time, the biochar obtained by torrefaction is characterized by higher microporosity, whereas hydro coal has a mesoporous character. This can be explained by the nature of the process. Torrefaction is characterized by the slow heating of biomass, which allows the preservation of the original structure of plant cells to a greater extent than slow pyrolysis. During hydrothermal carbonization, excessive water pressure leads to the probability of cavitation, which consists of both "collapse" and "destruction" of internal cavities, therefore, the porous structure of hydro coals is rather heterogeneous. The abrasion resistance for torrefied biomass seems to be slightly higher, probably due to incomplete decomposition of organic components of biomass and the "binding" effect.

*Table 1: Characterization results for biochar obtained by pyrolysis method*

| Parameter | Birch sawdust | | | Pine sawdust | | |
|---|---|---|---|---|---|---|
| | initial | hydrolyzed | after liquefaction | initial | hydrolyzed | after liquefaction |
| Specific surface area, $m^2/g$ | 2.8 | 5.7 | 4.7 | 2.3 | 6.4 | 5.9 |
| Pore volume, $cm^3/g$ | 0.02 | 0.04 | 0.04 | 0.02 | 0.05 | 0.05 |
| Carbon content, wt.% | 71.3 | 79.8 | 87.4 | 69.9 | 81.6 | 89.2 |
| Abrasion resistance, % | 57 | 61 | 59 | 55 | 63 | 58 |

*Table 2: Characterization results for biochar obtained by the torrefaction method*

| Parameter | Birch sawdust | | | Pine sawdust | | |
|---|---|---|---|---|---|---|
| | initial | hydrolyzed | after liquefaction | initial | hydrolyzed | after liquefaction |
| Specific surface area, $m^2/g$ | 4.7 | 6.9 | 6.2 | 4.9 | 7.2 | 6.8 |
| Pore volume, $cm^3/g$ | 0.03 | 0.05 | 0.05 | 0.03 | 0.06 | 0.05 |
| Carbon content, wt.% | 70.8 | 78.2 | 85.3 | 67.7 | 79.5 | 86.9 |
| Abrasion resistance, % | 62 | 64 | 61 | 65 | 69 | 67 |

*Table 3: Characterization results for biochar obtained by hydrothermal carbonization method*

| Parameter | Birch sawdust | | | Pine sawdust | | |
|---|---|---|---|---|---|---|
| | initial | hydrolyzed | after liquefaction | initial | hydrolyzed | after liquefaction |
| Specific surface area, $m^2/g$ | 4.2 | 6.3 | 5.8 | 4.6 | 6.5 | 6.2 |
| Pore volume, $cm^3/g$ | 0.03 | 0.05 | 0.05 | 0.03 | 0.05 | 0.05 |
| Carbon content, wt.% | 74.7 | 82.5 | 89.8 | 72.6 | 80.4 | 87.1 |
| Abrasion resistance, % | 59 | 62 | 60 | 61 | 65 | 63 |

To form nanostructured materials in the process of carbonization of biomass, it was decided to use additional mineral components. The catalytically active materials based on bioderived carbons are formed by subsequent activation during treatment with sulfonating agents, alkalis, deposition of active metals, etc. In this work, carbon materials containing additional components such as metal oxides and silica were obtained by torrefaction methods. The initial biomass was impregnated with solutions of metal salts, as well as alkoxysilane (TEOS), then dried in air and used for carbonization. The obtained functional carbon materials were analyzed for porosity, element content, and abrasion resistance. The surface composition was studied by the XPS method to estimate the form of the elements introduced.

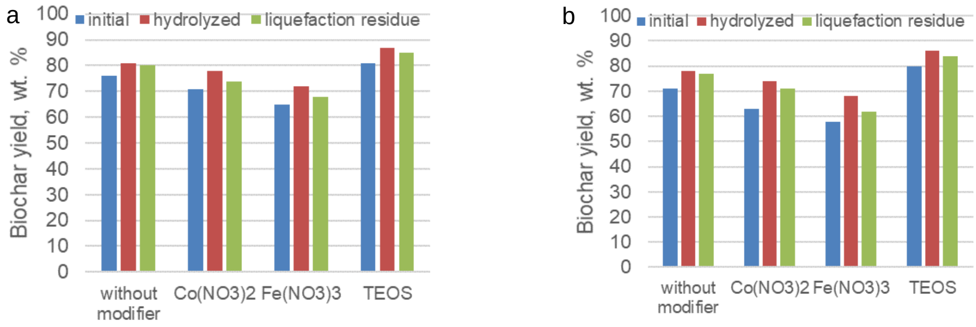


*Figure 2: Torrefied biochar yield depending on the mineral components introduced in biomass for birch sawdust (a) and pine sawdust (b)*

Figure 2 presents the influence of the mineral additives on the yield of biochar obtained by torrefaction. An increase in the biochar yield is observed when TEOS is used as a modifier for both pine and birch sawdust. This is due to the formation of the silica phase during the thermal decomposition of TEOS. The addition of TEOS did not show any catalytic effect, and the composed carbon-silica material was obtained. The addition of cobalt and iron nitrates, in contrast, leads to a decrease in the biochar yield in comparison with the non-treated sawdust. It is known that Fe and Co have a catalytic effect on the thermal decomposition of biomass facilitating the degradation of cellulose and hemicelluloses (Ryu et al., 2019). The catalytic effect of the metal used also leads to a decrease in the temperature of the decomposition of biomass components (Badr et al., 2019). Thus, the addition of cobalt and iron nitrates led to the formation of a higher amount of gaseous products.

The results of the biochar characterization are shown in Tables 4-6. When impregnating the feedstock with solutions of metal salts, an increase in the carbon content in the composition of bio-carbons was noted. This is associated with the catalytic effect of metal ions on the thermal decomposition of biomass. When impregnating biomass with alkoxysilane solutions, the catalytic effect is practically not observed, however, an increase in the specific surface area was noted for bio-carbons obtained by torrefaction due to the formation of the silicon oxide phase. The introduction of additional mineral components into the composition of biochar increases their abrasion resistance by creating an additional inorganic "framework". This effect was found to be higher for carbon materials with a silicon phase.

*Table 4: Characterization results for biochar obtained by torrefaction with iron nitrate*

| Parameter | Birch sawdust | | | Pine sawdust | | |
|---|---|---|---|---|---|---|
| | initial | hydrolyzed | after liquefaction | initial | hydrolyzed | after liquefaction |
| Specific surface area, $m^2/g$ | 4.2 | 6.4 | 5.9 | 4.4 | 6.9 | 6.1 |
| Pore volume, $cm^3/g$ | 0.03 | 0.05 | 0.05 | 0.03 | 0.06 | 0.05 |
| Carbon content, wt. % | 77.4 | 85.3 | 91.2 | 74.8 | 83.9 | 89.7 |
| Fe content, wt.% | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Fe state on the surface | $Fe_2O_3$ | $Fe_2O_3$ | $Fe_2O_3$ | $Fe_2O_3$ | $Fe_2O_3$ | $Fe_2O_3$ |
| Abrasion resistance, % | 71 | 82 | 76 | 74 | 83 | 78 |

*Table 5: Characterization results for biochar obtained by torrefaction with cobalt nitrate*

| Parameter | Birch sawdust | | | Pine sawdust | | |
|---|---|---|---|---|---|---|
| | initial | hydrolyzed | after liquefaction | initial | hydrolyzed | after liquefaction |
| Specific surface area, $m^2/g$ | 4.5 | 6.7 | 6.0 | 4.6 | 7.1 | 6.3 |
| Pore volume, $cm^3/g$ | 0.03 | 0.05 | 0.05 | 0.03 | 0.06 | 0.05 |
| Carbon content, wt. % | 79.2 | 86.8 | 92.3 | 76.1 | 85.4 | 90.6 |
| Co content, wt.% | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Co state on the surface | CoO | CoO | CoO | CoO | CoO | CoO |
| Abrasion resistance, % | 70 | 83 | 75 | 72 | 85 | 79 |

*Table 6: Characterization results for biochar obtained by torrefaction with TEOS*

| Parameter | Birch sawdust | | | Pine sawdust | | |
|---|---|---|---|---|---|---|
| | initial | hydrolyzed | after liquefaction | initial | hydrolyzed | after liquefaction |
| Specific surface area, $m^2/g$ | 9.6 | 13.5 | 12.4 | 9.8 | 14.0 | 13.1 |
| Pore volume, $cm^3/g$ | 0.08 | 0.11 | 0.11 | 0.08 | 0.12 | 0.11 |
| Carbon content, wt. % | 71.0 | 78.6 | 85.8 | 68.0 | 79.8 | 87.1 |
| Si content, wt.% | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Si state on the surface | $SiO_2$ | $SiO_2$ | $SiO_2$ | $SiO_2$ | $SiO_2$ | $SiO_2$ |
| Abrasion resistance, % | 77 | 86 | 81 | 79 | 88 | 82 |

## 4. Conclusions

In this work, the data on the effect of carbonization methods and structuring agents on the composition and structure of biochar obtained from the non-hydrolyzed and hydrolyzed sawdust, and solid residue of liquefaction were obtained. Three main methods were used to produce biochar: pyrolysis, torrefaction, and hydrothermal carbonization. It is noted that for hydrolyzed and non-hydrolyzed biomass, as well as for liquefaction solid residue, the highest biochar yield and the largest surface area were noted using the torrefaction method. At the same time, the biochar obtained by torrefaction is characterized by greater microporosity, whereas hydrocoal has a mesoporous character. The abrasion resistance for torrefied biomass was found to be slightly higher in comparison with the other methods used. To form nanostructured materials, biochars were obtained by torrefaction after impregnation of the feedstock with solutions of metal salts and alkoxysilanes. When impregnating raw materials with solutions of metal salts, an increase in the carbon content and a decrease in the biochar yield were noted due to the catalytic effect of metal ions on the thermal decomposition of biomass. When impregnating biomass with alkoxysilane, the catalytic effect is practically not observed. However, an increase in the biochar yield and specific surface area due to the formation of the silicon oxide phase was noted. The introduction of additional mineral components into the composition of bio-carbons increases their abrasion resistance by creating an additional inorganic "framework".

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