Metallurgical Coke Production with Biomass Additives. Part 2. Production and Characterization of Laboratory Biocokes

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1 Introduction

The development of technological processes in the modern world follows the path of reducing the environmental impact, using renewable sources of energy and raw materials while simultaneously obtaining products of the required quality [[1–](#page-14-0)[4\]](#page-14-1).

Based on the results of the annual UN conferences on climate change [\[5](#page-14-2)], further commitments are planned to reduce greenhouse gas emissions globally [[6,](#page-14-3) [7](#page-14-4)]. This requires a significant modernization of production to ensure the environmental sustainability of the industrial development of the metallurgy [[8–](#page-14-5)[10\]](#page-14-6).

In the next five years, a significant proportion of the world's existing integrated steel plants will reach the 60-year age limit and be decommissioned. In addition, it is expected that in 2040–2060 blast furnaces (BF) with a total production of about 200 million tons per year will be decommissioned before reaching final depreciation. There will probably be no need to build new blast furnaces.

Ensuring the sustainable development of metallurgy, particularly in light of future restrictions on $CO₂$ emissions, requires the modernization of the industry with the introduction of innovative technologies and the improvement of the existing ones [[11–](#page-14-7)[14\]](#page-14-8).

Coke production technology greatly impacts the natural environment due to using fossil coal. The primary consumer of coke is blast furnace production, the technology of which imposes specific requirements on the quality of the coke $[15-17]$ $[15-17]$. To meet the high requirements, which are constantly increasing, it is necessary to use raw

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287

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materials with suitable initial characteristics (according to proximate analysis, coking ability, and coarseness) to produce coke.

Along with the blast furnace industry, which uses up to about 70% of the coke produced, several other industries use coke and have related requirements. For example, for ferroalloy production, using more reactive coke with a grain size of 10–25 mm is desirable. For the sintering of ores, coke fines (<10 mm) are needed, along with high reactivity. In foundry production, coke should be coarse and lowreactive [[15\]](#page-14-9). Non-blast furnace consumers need coke for high-temperature reactions. The function of carbon materials in these processes is to provide the necessary temperature for reactions and carbon transfer for carbonization.

As an alternative to fossil fuels, renewable energy sources from organic raw materials are increasingly used [[17–](#page-15-0)[23\]](#page-15-1). The most important of them is the vegetable raw material formed in photosynthesis. Biomass is the product of converting solar energy into chemical energy. Plant biomass is a valuable renewable chemical raw material from which unique compounds and fossil fuel substitute products can be obtained.

It is known that charcoal was widely used in the production of cast iron until the middle of the twentieth century $[24]$ $[24]$. Charcoal is used as the main fuel and reducing agent in small blast furnaces in Brazil [[25–](#page-15-3)[27\]](#page-15-4). However, charcoal is mainly used in the blast furnace process for injection into the BF via the tuyeres [[25,](#page-15-3) [28](#page-15-5)[–31](#page-15-6)], as well as the carbon iron ore composite [[32\]](#page-15-7) and steel recarburazer [\[33\]](#page-15-8). At the same time, Brazil is one of the leading countries in using charcoal for steel production [\[34](#page-15-9)].

The advantages of using charcoal in the BF process, as opposed to coal coke, are low ash, sulfur, and high porosity. As an injecting component, charcoal has high reactivity and low mechanical strength. However, the use of charcoal for feeding into BF is ineffective precisely because of these features of mechanical and chemical properties.

At the current state of industry development, biomass is important as a renewable source of raw materials that do not increase the amount of $CO₂$ in the atmosphere. Biomass, as a substitute for fossil fuels, is actively used to produce biofuels [[35](#page-15-10)[–39](#page-16-0)], in the energy $[38, 40-42]$ $[38, 40-42]$ $[38, 40-42]$ $[38, 40-42]$, obtaining nanomaterials $[43-47]$ $[43-47]$, bioplastics $[48-51]$ $[48-51]$, as an effective adsorbent [[52–](#page-16-8)[55\]](#page-16-9), as well as fertilizer [\[56](#page-17-0)[–58](#page-17-1)].

Over the past couple of decades, the issue of using biomass and its products in the metallurgical process, namely in the BF process [[59–](#page-17-2)[63\]](#page-17-3), sintering ores [\[64](#page-17-4)– [71\]](#page-17-5), electrical arc furnace (EAF) to enable carburizing and slag foaming [[72,](#page-17-6) [73](#page-17-7)], submerged arc furnace (SAF) [\[74](#page-18-0)[–76](#page-18-1)], reduction of iron [\[77](#page-18-2)[–79](#page-18-3)] has been actively studied. At the same time, the amount of replacing the use of reducing raw materials instead of the conventional one depends on the type of process and the properties of biomaterials. Studies demonstrate that it is possible to increase the share of biomass use and its efficiency through the use of biocoke [\[80](#page-18-4)[–83](#page-18-5)].

Co-pyrolysis of biomass and coal can be considered as one of the possible ways of controlling the thermochemical conversion of coal and the formation of molecular structure and physical and chemical properties of the solid residues. However, for the effective use of biomass as a fuel and raw material for chemical products, it should be considered that biomass from different sources has different properties [\[84](#page-18-6)].

Studies on the effect of biomass additives on the caking process and coke properties show that a minor proportion of some biomaterials (up to 3 wt. $\%$) in the mixture does not significantly affect the caking of coals [[85,](#page-18-7) [86\]](#page-18-8). When studying the behavior of the removal of volatile matters from mixtures of biomass and coal, some authors found that no interaction occurs, and the yield of pyrolysis products is associated only with the amount of biomass and coal in the blend [\[87](#page-18-9)[–90](#page-18-10)]. Other authors have observed interactions between coal and biomass in co-combustion [[91\]](#page-18-11) and co-pyrolysis of biomass and brown coal [\[92\]](#page-18-12).

When studying the issue of using biomass for co-processing with coal, it is necessary to have a clear, theoretically substantiated, confirmed-by-practice understanding of the processes of thermal destruction of the mixture with the formation of a solid residue and chemical products (tar, gas). Analysis of publications on the co-pyrolysis of coal and biomass [\[93,](#page-18-13) [94](#page-19-0)] highlights the main patterns of ongoing processes, depending on the temperature stage of processing (Table [1\)](#page-3-0). At the same time, lignocellulosic biomass is considered a natural polymer consisting of hemicellulose, cellulose, and lignin, the thermal destruction of which has its characteristics [[95\]](#page-19-1).

Thus, the relevance of the use of biomass in metallurgy, as well as a small number of studies on the production and analysis of biocokes, formed the main aim of the chapter is to establish the effect of biomass additives on the yield and quality of biocokes. Co-pyrolysis of coal blend and various types of biomass was carried out via a laboratory shaft furnace with different portions of biomass additives (up to 45 wt.%) and their forms (pellets and chips). The supplementary aim considered in this chapter is to analyze the influence of the type of biomass, its quantity, and form on the technical and physical–mechanical properties of biocokes and quality analysis of biocoke for compliance with the requirements of metallurgical processes.

2 Materials and Methods

Materials

Table [2](#page-4-0) shows the characteristics of the coals that were used to prepare the coal blend. Coals are characterized by high sulfur content, typical for Ukrainian caking coals. It should be noted that the thickness of the blend's plastic layer (y, mm) was 12 mm, which is 2 mm less than conventional coal blends used for industrial coking. The indicators of the proximate analysis of coals were determined according to ASTM D3172-13 [[96\]](#page-19-2) and plastometric values according to [\[97](#page-19-3)[–99](#page-19-4)].

Indicators of reflectance of vitrinite, vitrinite reflectogram, and petrographic composition of the coal used in the research are given in Table [3](#page-5-0). The indicators were determined according to [\[100](#page-19-5)–[102\]](#page-19-6).

The average vitrinite reflectance (R_0) is 1.01%, while the values corresponding to industrial charges with good coking characteristics are 1.1–1.2% [\[97\]](#page-19-3).

Temperature range, ^o C	Component	Description	Main processes	
up to 105 °C	Hemicellulose Moisture removal. The duration of the stage is Cellulose determined by the Lignin moisture content and the weight of the charge Hard Coal		Dehydration. Evaporation of water occurs in a narrow range of about 100 $\rm ^{\circ}C$ This stage determines the subsequent course of the pyrolysis process in terms of the temperature field formation of the charge	
$105 - 270$ °C	Hemicellulose	Drying and start of decomposition at temperatures of about 200 $^{\circ}C$	Removal of adsorbed moisture Formed mainly CO, CO ₂ , and acetic acid	
	Cellulose	Drying and decomposition start early, even at 240 \degree C		
	Lignin	Drying and minor decomposition start early, even at 260 \degree C		
	Hard Coal	Removal of colloidal moisture and occluded gases (CO ₂ , CO). Drying	Removal of adsorbed moisture Pre-plastic state	
270-350 °C	Hemicellulose	Active decomposition	Isolation of combustible gases CO, H ₂ , CH ₄ , CO ₂ , acetic acid, methanol, acetone, etc., and tar	
	Cellulose			
	Lignin	Beginning of active decomposition		
	Hard Coal	Softening of coal with the beginning of the formation of coal plastic layer	Pre-plastic state	
350-420 °C	Hemicellulose	Completion of	The release of combustible gases CO, H_2 , and CH ₄ , along with $CO2$, acetic acid, methanol, acetone, etc., and tar	
	Cellulose	decomposition		
	Lignin	Active decomposition		
	Hard Coal	Plastic state	The main process of pyrolysis. Active physical and chemical processes with the participation of coal plastic layer and the release of volatiles	
420-500 °C	Hemicellulose		Formation of charcoal	
	Cellulose	$\overline{}$		

Table 1 Main processes that occur during the pyrolysis of biomass and hard coal

(continued)

Table 1 (continued)

As is known, coals' size strongly influences their thermal processing processes [[103–](#page-19-7)[105\]](#page-19-8). To exclude the influence of coal particles of different distributions on the quality of coke, a stable granulometric composition of coal of various types was taken, which was maintained during the preparation of coal blends. Firstly, coal of a specific granulometric composition was prepared, and afterward, it was mixed to the particle size distribution according to Table [4](#page-6-0).

Wood, sunflower husks, and straw were used as biomass additives in the form of pellets with a diameter of 8 mm and a length of 4 to 12 mm. Additionally, for comparison, when coking the blend, wood chips were used as an additive. The biomass additives' properties are summarised in Table [5](#page-6-1).

Coal type	Content of sizes, %					
	$10-6$ mm	$6-3$ mm	$3-1.5$ mm	$1.5 - 0.5$ mm	< 0.5 mm	
Coal A	4	10	31	30	25	
Coal B	4	22	30	24	20	
Coal C		17	24	33	23	
Coal D	2	8	27	32	31	
Blend	3.5	15.5	28.2	29.3	23.5	

Table 4 Particles size distribution of coals

Table 5 Characteristics of additive properties, wt.%

			Type of additive Moisture (W_t^r) Volatile matters (V^r) Total sulfur content (S_t^d) Ash (A ^r)	
Wood	9.4	81.3	0.01	
Sunflower husks $ 9.6$		75.7	0.01	2.6
Straw	4.7	75.0	0.01	5.4

Methods

Laboratory Coking

Coking was carried out in a laboratory shaft furnace [[80\]](#page-18-4) at 800 °C. The load was 1 kg, and after being charged in a retort, it was placed into a heated furnace. After finishing coking, the retort with the obtained coke was taken out and left to cool to room temperature. The coking time was 80 min.

Yield of Coke

After naturally cooling to room temperature, the obtained cokes were subjected to the analysis of the yield, %. The yield was calculated using Eq. [1.](#page-6-2)

$$
Yield = B \times 100/A, \,\%
$$
 (1)

where A is the initial mass of the blend, g; B is the mass of coke/biocoke after coking, g.

Particle Size Distribution of Coke

Further, the resulting cokes were sieved into sizes > 40 , $40-25$, $25-10$, and < 10 mm according to ASTM D293/D293M-18 [[106\]](#page-19-9).

Structural Strength

To determine the structural strength, coke and biocoke samples of 6–3 mm were prepared. Then samples were charged into two special steel cylinders according to [[107\]](#page-19-10). Likewise, five steel balls were placed in the cylinders. These two cylinders were set through screws in the cross-to-cross position in brackets, which were put on a shaft rotating with 0.417 s^{-1} (25 rpm). During the tests, the cylinders performed 1000 revolutions, after which the contents of each cylinder were poured separately on a sieve with a mesh of 3 mm and 1 mm. Coke or biocokes were sieved to separate into 3–1 mm and 1–0 mm fractions. The yield of more than 1 mm from the initial weight in percent characterizes the structural strength.

Abrasive Hardness

The abrasive hardness was determined by the abrasion of an aluminium plate against coke or biocoke powder and the evaluation of its mass loss. 4–5 g of samples of less than 0.5 mm in size was filled on an aluminum plate. A stamp was placed on top of the samples of coke or biocoke, which were loaded so that a pressure of 0.25 MPa was obtained. During the rotation of the rotor (500 rev.), the samples abraded the aluminum plate. The weight loss of the aluminium plate during the test run (in milligrams) was taken as the value of the abrasive hardness. Five tests were conducted for each sample.

Electrical Resistivity

The electrical resistivity measurement was carried out according to [\[108](#page-19-11)]. The method aims to determine the electrical resistivity of the particles with a size < 2 mm placed in the cylinder between two stainless steel plungers under a pressure of 3 MPa. The resistivity measured using a four-point mode is advantageous because it allows measuring resistivity close to the actual resistivity of the sample [[109\]](#page-19-12).

3 Results and Discussions

The characteristics of biocokes obtained from coking are summarized in Table [6.](#page-9-0) The yield of laboratory biocokes decreases with an increase in biomass additive, which has a higher yield of volatile matter than coal. The ash content of biocokes decreases due to the lower content of mineral components in the biomass. There is also a decrease in sulfur content since biomass additives practically do not add it to the mixture.

The yield of volatile matters of biocokes is determined by the level of readiness and varies from 0.8 wt.% to 2.5 wt.%. However, there is a tendency that with an increase in the proportion of biomass addition, the yield of volatile matters of the obtained biocokes increases.

The effect of biomass additives on the physical and mechanical properties of biocokes is more significant. The amount of 1 wt.% and 3 wt.% pellets of three types of biomass, as well as 5 wt.% wood pellets do not cause a deterioration in the particles size distribution of biocokes compared to the reference coke. At the same time, the use of wood chips has a more noticeable effect on the size of biocokes in the direction of a decrease in large sizes (>25 mm) and an increase in small ones (<10 mm). In general, with an increase in the proportion of the additive, there is a trend towards a decrease in the yield of large sizes and a corresponding increase in small ones.

Adding up to 3 wt.% of sunflower husks and straw pellets has a positive effect on structural strength. The additives of wood pellets and wood chips reduce the structural strength. At the same time, the negative effect of using chips is more pronounced than for pellets (Fig. [1](#page-10-0)). Similar dependences are observed when studying the impact of biomass additives on the abrasive hardness of the obtained biocokes (Fig. [2\)](#page-10-1).

Thus, the addition of sunflower husk pellets up to 5 wt.% and straw up to 3 wt.% led to an increase in abrasive hardness, as well as the use of wood chips up to 3 wt.%. At the same time, adding 3 wt.% chips significantly increases the abrasive hardness index, although adding wood pellets decreases this index. This can be because fine coke (<0.5 mm) is used to study abrasive hardness. Therefore the value of the index depends to a greater extent on the degree of biocoke readiness, the number of charcoal particles in the sample, and their distribution.

Electrical resistivity as an indicator that characterizes the structural features of carbon materials shows an improvement in the structural ordering of biocokes when using sunflower husk additives up to 3 wt.% compared to the reference one. The use of other additives leads to a decrease in the quality of biocokes in this indicator (Fig. [3](#page-11-0)). With an increase in the number of additives, electrical resistivity increases, although not linearly.

Therefore, the addition of sunflower husk up to 3 wt.% leads to an improvement in the physical and mechanical properties of biocokes, which can be explained by the presence of a certain amount of oily substances [\[110](#page-19-13), [111\]](#page-19-14), which during thermal destruction, positively affect the process of formation and properties of biocoke. Straw pellet additives up to 3 wt.% positively affect structural strength and abrasive

hardness, but electrical resistivity increases when using this type of additive. The use of wood additives shows a tendency to reduce the physical and mechanical properties of biocokes. At the same time, the use of pellets has a less negative effect on the structural features of biocokes (structural strength, electrical resistivity) than the addition of chips.

A study of the effect of adding a high proportion of wood pellets on the quality of biocokes demonstrates notable dependencies. Thus, the yield of biocokes decreases non-linearly and increases the yield from the expected one. The physical and mechanical properties of biocoke obtained with additives of 15 and 30 wt.% remain acceptable for non-BF productions. At the same time, inclusions of charcoal in the form of pellets are clearly observed, which, with an increase in the amount, are better separated from the bulk of the solid residue of coal pyrolysis.

Fig. 3 Effect of addition of biomass on electrical resistivity of biocokes

Therefore, the results of earlier studies are confirmed [[80,](#page-18-4) [83\]](#page-18-5), which shows the local effect of wood pellet additives on the change in the properties of the resulting biocoke.

Analysis of the obtained results shows that biomass additives affect the process and quality of biocoke production. The effect of influence is determined by qualitative and quantitative factors, namely the type, form, and proportion of the additive in a mixture with coal. The mechanism of the influence of biomass additives can be explained by the scheme of the co-pyrolysis of coals and biomass, which is presented in Fig. [4](#page-11-1).

Fig. 4 Scheme of the mechanism of co-pyrolysis of coal and biomass: V_b —vapor of water from biomass; S_b —solid biomass; VM_b—volatile matter from raw and pyrolyzed biomass; V_{hc}—water vapor from hard coal; V_{og} —occluded gas from hard coal; S_{hc} —solid hard coal; VM_{hc} —volatile matter from hard coal; PM_{hc} -plastic layer from hard coal

The scheme shows the course of the process of co-pyrolysis of biomass and coal, namely drying and subsequent parallel and sequential processes of dehydration and condensation with the formation of liquid and solid products and depolymerization processes with the formation of volatile and tarry liquid substances, in the case of biomass pyrolysis; as well as dehydration processes followed by sequential-parallel polycondensation reactions with the formation of metastable phases in the case of coal pyrolysis.

Consequently, there is a transition from the solid state to the solid, a systematic but limited at each stage, decrease in the mass of the solid, a change in the yield of volatile matters, and the appearance, growth, and disappearance of the plastic layer of coal. The pyrolysis process is non-isothermal and continuous, forming and destroying new compounds. As can be seen from the scheme, during the co-pyrolysis of biomass and coal, different phases interact.

Biomass, namely cellulose and hemicellulose, begins to decompose before coal; thus, the volatile decomposition products formed in the charge further process the coal. Considering the predominantly oxygen-containing composition of volatile biomass, their effect on coal is oxidative, which ultimately reduces the caking ability of coals [[82,](#page-18-14) [112](#page-19-15)[–115](#page-19-16)].

The treatment of biomass with volatile pyrolysis products should affect the kinetics of the yield of steam-gas products of coal. In turn, with increasing temperature, higher molecular weight volatile products of coal act on solid biomass degradation products, processing charcoal.

In the coal pyrolysis process, all phases interact with each other, and each is involved in polycondensation. As a result, new metastable products are synthesized, namely liquid, solid and gaseous, forming phases of the next stage. Evidence of this should be the formation of various amounts of vapor–gas, liquid, and solid products. The most active changes occur within the temperature range of 250–500 °C when biomass decomposes and the coal plastic layer is formed.

It is also important to consider that under higher temperatures and industrial conditions, thermal degradation of biomass pyrolysis tar and light products of primary coal tar will take place. The yield level of various products is determined by the depth of transformations that coal and biomass undergo at the stages of pyrolysis. Therefore, it is necessary to distinguish between conclusions on the co-pyrolysis of biomass and coal, obtained based on pyrolysis under laboratory conditions, and industrial-scale ones.

Additionally, essential to note that during pyrolysis, the destruction-synthesis processes are limited by the rate of thermochemical transformations of the coal substance and not by heat transfer inside the coal particle.

Thus, the addition of biomass actively affects the process of thermal destruction of the charge and, subsequently, the qualitative indicators of the solid residue: proximate analysis, physical–mechanical and physical–chemical properties. Accordingly, using biomass additives makes it possible to obtain biocoke of different qualities for various production processes.

4 Conclusions

Studies on the production of biocoke under laboratory conditions and its study showed that the addition of biomass in the form of wood pellets, wood chips, pellets from sunflower husks, and pellets from straw into coal blend leads to a change in the properties of coke: yield, particles size distribution, structural strength, abrasive hardness, and electrical resistivity.

The use of up to 3 wt.% biomass pellet additives and 5 wt.% wood pellets do not lead to a deterioration in the granulometric composition of biocokes. At the same time, the output of large sizes $(>25 \text{ mm})$ and small sizes $(< 10 \text{ mm})$ changes noticeably when using the addition of wood chips and with more than 5 wt.% pellet additions.

Regarding structural strength, abrasive hardness, and electrical resistivity, biocokes obtained with the addition of sunflower husks have better characteristics. This can be explained by the presence of a residual amount of oily substances, which improve the caking of coal particles. The addition of sunflower husks generally carries fewer oxygen-containing groups, negatively affecting the co-pyrolysis process with coal.

Straw pellet additions up to 3 wt.% also do not adversely affect the particle's size distribution and structural strength, with a noticeable improvement in abrasive hardness. However, the electrical resistivity of biocokes obtained with the participation of straw increases.

The addition of wood leads to a decrease in the physical and mechanical properties of biocokes. However, the deterioration of properties is not linear, which is more clearly seen when studying the effect of increased wood pellet addition. At the same time, using chips compared to pellets has an even more negative impact. Therefore, the effect of the pellets is local in nature on the change in the properties of the resulting biocoke, and the effect of the chips can be considered volumetric due to the uniform distribution of the chips in the charge volume.

Using biocoke as a metallurgical fuel and reducing agent can improve the technical and economic performance of non-blast furnace industries by using carbon material with more suitable physical, mechanical, and physicochemical properties than conventional coke. In addition, using biomass additives is an environmentally friendly approach to obtaining carbon fuels. The advantages of using biomass as a component of coal blends in the coking process are the utilization of biomass; use of renewable raw materials, instead of fossil fuels, in particular, deficient coking coal; obtaining a new product, namely biocoke, the properties of which meet the modern requirements of consumers; reducing the negative impact on the environmental situation in the region and the world. The cost of biocoke may be lower than that of obtaining the corresponding grades of conventional coke. Therefore, using biomass additives to produce coke will solve complex problems in industrial regions and enterprises of ferrous metallurgy, agriculture, the chemical industry, and many others that process biomass.

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