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Optimization of charge composition for coke production with spent anode material and evaluation of agglomerate properties

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Abstract

The purpose of this study was to optimize the batch composition for coke production by introducing spent anode material and to evaluate the properties of the resulting agglomerates. Experimental batches of coke with different levels of spent anode material residue were investigated, and the resulting agglomerates were subjected to comprehensive testing. The investigations included determination of chemical composition, spectroscopic analysis, and phase characterization of the coke using Fourier-transform infrared (FTIR) spectroscopy. It was found that samples with 20-30% spent anode material have an optimal ratio of aromatic and oxygen-containing groups, demonstrating a high degree of carbonization while maintaining structural strength. With an increase in the proportion of spent anode material to 40%, there is a decrease in the number of binding functional groups, which can worsen the strength characteristics. Laboratory agglomeration tests using modified coke were carried out. The obtained agglomerates showed satisfactory technological and chemical properties. The yield of the suitable agglomerate was 72.4–73.5 %, which confirms the effectiveness of using coke with 20–30 % spent anode material for metallurgical sintering processes. The results indicate that the inclusion of anode residue in the batch composition can enhance certain operational parameters of coke, such as structural integrity and resistance to degradation, while maintaining acceptable levels of strength and reactivity. These findings provide a basis for the industrial application of spent anode material in coke production, ensuring both resource efficiency and potential cost savings.

Keywords: Agglomeration, Coal pitch, Coal, Coke, FTIR, Man-made waste, Spent anode material.

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1. Introduction

Modern metallurgical processes, particularly agglomeration, demand stable, high-quality carbonaceous materials that ensure efficient thermal energy delivery and structural integrity of agglomerates. With the rising cost of coking coal and accumulation of industrial waste, interest in developing alternative coke feedstocks has grown significantly [1-4].

Today, a resource-efficient approach to the development of any country's economy is at the forefront. First of all, there are issues of saving energy and materials. Man-made materials, especially carbon-rich ones, are becoming promising sources, accumulations of which occupy huge areas, causing irreparable harm to the environment. Using waste, reducing the consumption of natural resources and the harmful impact of waste storage facilities on the environment, at the same time, it is possible to solve a wide range of economic, environmental and social problems [5-8].

In Kazakhstan, a significant amount of industrial waste is generated annually, and these issues are not given due attention. According to the latest data, about 31.6 billion tons of industrial waste have been accumulated in Kazakhstan, and about 1 billion more tons are added annually. The majority (70 %) are technogenic-mineral formations. Another 10 % is accounted for by waste from the manufacturing industry, and the remaining 20 % is accounted for by other types of activities. These wastes, including coal dust, sludge, and other by-products of coal mining and processing, represent significant potential for processing into valuable products such as coke [9-11].

Thus, the processing of coal industry waste into coke not only reduces the environmental burden but also provides an additional source of valuable product for the metallurgical industry of Kazakhstan.

In this regard, the problem of applying new progressive technologies for the thermal processing of coals into coke, an environmentally safe and energy-saving fuel and a reducing agent for industrial use, is of great practical importance [12, 13]. The need for further improvement of these methods has determined the conduct of a complex of research, experimental and pilot-industrial work on the development of scientific foundations and the development of new technological solutions in the field of thermal processing of coal, ensuring the production of high-quality products for technological and energy purposes.

Efforts are being made in Kazakhstan to recycle man-made waste from the coal industry. For example, Rentan LLP in Karaganda has introduced technology for the production of coke briquettes using coke production waste or cheap energy coals. This technology eliminates the need to use scarce coking coals and reduces energy consumption [14].

In addition, Qaz Carbon LLP is engaged in the disposal of industrial waste, such as coal and coke dust, by adding them to the charge for coke production. This helps to reduce the amount of waste and increase the efficiency of using raw materials [15].

Despite the efforts being made and individual projects implemented to recycle man-made waste from the coal industry in Kazakhstan, the scale of such work remains insufficient to significantly reduce the accumulated environmental damage. Existing technologies cover only a small fraction of the waste generated, while the potential for their recycling, in particular for the production of metallurgical coke, remains largely unrealized. In this regard, it is necessary to continue and expand scientific research aimed at the development and implementation of progressive, energy-efficient and environmentally friendly technologies for thermal processing of carbon-containing waste.

The present work is a continuation of a previously published study in which the composition of a briquetted charge based on man-made wastes such as coal fines, spent anode materials and coal pitch was developed, as well as its thermogravimetric analysis was carried out and the energy characteristics of the coke obtained were determined and the coke production technology was experimentally implemented. The differential thermal analysis of the charge materials made it possible to determine the optimal temperature conditions for coking, as well as to evaluate the calorific value and electrical conductivity of the resulting coke. It was found that this material has good thermal characteristics and can be used as an alternative fuel in the sintering process.

However, for a deeper understanding of the structure of the obtained coke and prediction of its behavior in high-temperature processes, it is necessary to study its microstructural and chemical characteristics.

The purpose of this work is to perform spectroscopic and phase analysis of coke obtained using the previously proposed technology, as well as to test it in a laboratory agglomeration process. Within the framework of this study, the main focus is on the structural and chemical characterization of the obtained coke using FTIR spectroscopy methods and further testing of the material in the agglomeration process.

The scientific novelty is that for the first time, the FTIR spectral analysis of coke obtained from a specific composition was performed: Ekibastuz coal mixed with spent anode material and coal pitch, and this coke was tested in sintering production.

In recent years, FTIR spectroscopy has been widely used to study the structure of carbon-containing materials, including coals, semi-coke, and coke, to identify chemical changes occurring during thermal processing. Studies show that this method is effective for identifying functional groups, assessing the degree of aromaticity, the residual content of binders, as well as studying the transformation of hydrocarbon structures during coking. In particular, FTIR analysis was used to quantify the content of carbonyl, hydroxyl, and aromatic fragments in coals of various degrees of enrichment, as well as to determine the microstructural differences between the initial and heat-treated materials. This made it possible to establish correlations between the chemical composition and the quality of the coke produced [16-20].

2. Materials and Methods

2.1. Materials.

The following raw materials were used for the preparation of metallurgical coke:

- Coal fines from the Ekibastuz coal deposit (fraction size 0.3–3 mm), a by-product of coal mining operations, were selected as the primary carbon source due to their availability and low cost.
- Spent anode material, a carbon-rich residue (96–98% carbon) from the electrolysis process in aluminium production, was used as a carbon concentrator.
- Coal pitch was used as a binder because of its favorable coke-forming ability and low viscosity in the molten state, which ensured uniform binding of the components during briquetting.

2.2. Methods

The previously obtained coke samples with different percentages of components were subjected to a detailed study using FTIR (FTIR – Fourier Transform Infrared Spectroscopy). Table 1 and 2 show the content of the components and their chemical composition, respectively.

Table 1.The content of the charge components of the coke samples studied, %.

| Sample | The content of components in the charge, % | | | | | | |
|--------|--|----------------------|------------|--|--|--|--|
| № | Ekibastuz coal | Spent anode material | Coal pitch | | | | |
| 1 | 50 | 10 | 40 | | | | |
| 2 | 50 | 20 | 30 | | | | |
| 3 | 50 | 30 | 20 | | | | |
| 4 | 50 | 40 | 10 | | | | |

Table 2. Chemical composition of the charge materials of the coke samples studied, %.

| Indicator | Ekibastuz coal (KCH), avr | Spent anode material, avr | Coal pitch, avr | | |
|------------------------------------|---------------------------|---------------------------|-----------------|--|--|
| Carbon (C), % | _ | 98 | 92 | | |
| Hydrogen (H), % | _ | _ | 4.5 | | |
| Oxygen (O), % | _ | _ | 0.9 | | |
| Nitrogen (N), % | _ | _ | 1.7 | | |
| Sulfur (S), % | 0.7 | 2.0 | 0.5 | | |
| Moisture (Wr), % | 5.4 | _ | _ | | |
| Ash content (Ad), % | 39 | 0.3 | 0.2 | | |
| Volatile substances | 32 | _ | <u> </u> | | |
| (Vdaf), % | | | | | |
| Qir, kcal/kg | 4000 | _ | <u> </u> | | |
| Melting point of ash, °C | 1490 | _ | | | |
| SiO ₂ , % | 62.1 | _ | <u> </u> | | |
| Al ₂ O ₃ , % | 28.0 | _ | <u> </u> | | |
| Fe ₂ O ₃ , % | 5.83 | _ | | | |
| CaO, % | 1.98 | _ | _ | | |
| MgO, % | 0.72 | _ | _ | | |
| TiO ₂ , % | 1.37 | _ | | | |
| SO ₃ , % | 1.43 | | | | |
| Density, g/cm ³ | | 1.56 (t/m³) | 1.2 | | |
| Softening point, °C | _ | _ | 115 | | |

The FTIR method was used to analyze the functional groups in the structure of the resulting coke. The main purpose of this analysis is to determine the degree of degradation of organic compounds and binder residues during coking.

Infrared spectroscopy (IR) is an analytical technique that uses light from the infrared part of the spectrum to analyze a sample. Peaks in the IR spectrum indicate the selective absorption of part of the radiation that coincides in frequency with the vibrations of atoms in the molecule, that is, they correspond to the frequencies of vibrations of chemical bonds in the sample. The spectra can be recorded in the transmission mode, in which case the spectra will be a line with certain "dips" corresponding to absorption bands, or in the absorption mode – absorption bands – peaks, "rises" directed upward [21].

The Shimadzu IRTracer-100 Fourier spectrometer is designed to measure optical transmission, absorption, diffuse and specular reflection, and disturbed total internal reflection in the infrared (IR) range; determine the concentration of various organic and inorganic substances in solid, liquid and gaseous states (Figure 1).



Figure 1. Fourier spectrometer Shimadzu IRTracer-100.

The principle of operation of spectrophotometers is based on determining the travel difference between interfering rays when moving mirrors in a two-beam interferometer. The spectrophotometer is based on the Michelson interferometer.

The radiation emitted by the light source passes through the aperture, with the help of a collimator, the beam of rays is reflected, becoming parallel, into the beam divider at an angle of incidence of 30°. The beam divider is a germanium film deposited on a substrate of potassium bromide. It divides the beam into two, one of them reflects onto a fixed mirror, and the other passes onto a movable one. Both mirrors reflect the beams into the divider. Each of the reflected beams becomes interfered with when passing through the divider. The returning beams of rays are transmitted and reflected radiation. The transmitted radiation from a fixed mirror and the reflected radiation from a moving one combine and intersect with each other, heading into a collective mirror. This fixed mirror has an automatic adjustment function that always ensures maximum interference efficiency. Using a collective mirror, a parallel infrared interfered beam of rays creates an image of the light source in the center of the cuvette compartment. The beam that passed through the sample mounted in the center of the cuvette compartment is reflected by a collecting mirror and enters the detector, where it is determined as an interferogram, which is a Fourier image of the detected optical spectrum. The spectrum itself (in the scale of wave numbers) is obtained after performing special mathematical calculations of the interferogram [22].

Subsequent stages of the study focused on evaluating the practical application of the produced coke materials in sintering processes. Agglomeration is the process of pretreatment of fine-grained material, in which this material, which cannot be directly loaded into a blast furnace, undergoes high-temperature solidification during sintering to obtain a material that meets the requirements for melting in a blast furnace [23].

For the agglomeration trials, coke samples containing 20 % and 30 % of spent anode material were selected based on prior analyses of their microstructure and spectral characteristics. The charge composition for sintering included screened coke produced from the experimental blends, rolled scale obtained from Casting LLP (Pavlodar, Kazakhstan)—a byproduct rich in iron oxides—and limestone sourced from the Keregetas deposit (Pavlodar region), operated by a subsidiary of Aluminum of Kazakhstan JSC.

The carbonaceous part of the mix consisted of coal from Bogatyr Komir LLP (Ekibastuz, Kazakhstan), spent anode material, and coal pitch supplied by the Kazakhstan Electrolysis Plant JSC (Pavlodar). The specific mass ratios are detailed in Table 3.

Table 3. The content of the charge components of the studied samples for agglomeration, %.

| Experiment number | Charge components, % | | | | | |
|-------------------|----------------------|-----------|-------|--------|----------|--|
| | Rolling scale | Limestone | Coke* | Refund | Moisture | |
| Experiment No. 1 | 55 | 10 | 7 | 20 | 8 | |
| Experiment No. 2 | 55 | 10 | 7 | 20 | 8 | |

Note: * - In the first experiment, coke containing 20 % spent anode material was used, in experiment No. 2 – 30 %.

Before the sintering step, all charge components were homogenized and pelletized using a plate-type granulator to obtain granules ranging from 5 to 10 mm in diameter. To achieve the required moisture content of 10–15 %, water was added after two minutes of dry blending. These parameters were selected based on empirical optimization. The total pelletizing time amounted to 3 minutes. After pelletizing, the charge was weighed, documented, and readied for sintering trials.

To maintain experimental consistency, the sintering bowl was pre-cooled to a stable baseline temperature. A preliminary layer of crushed return agglomerate (0.5 kg, 15–20 cm thick) was evenly placed over the grate. The pelletized charge was manually loaded and leveled using a guide template to ensure uniform bed thickness; excess material was

removed to match the set height. The weight of the prepared charge was determined by recording the mass difference of the loading container before and after charging.

An ignition mixture comprising fine coke particles (0–5 mm) and moistened sawdust was applied across the top surface of the bed to enhance gas permeability and initiate combustion. This was subsequently covered with wood shavings. The coke component constituted approximately 2 % of the total charge mass.

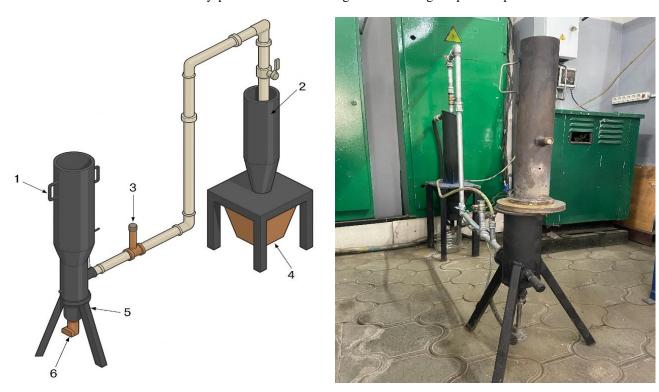
Sintering experiments were performed using a laboratory-scale setup (Figure 2) at the NJSC Toraighyrov University [24]. Surface ignition was used to trigger the sintering reaction, which progressed downward through the charge layer under vacuum generated by a dedicated pump. Airflow was controlled via an adjustable slide gate, while process temperature was monitored using a thermocouple. Exhaust gases were filtered and extracted through the ventilation system.

The operation of the laboratory sintering unit (Figure 5) begins with the creation of reduced pressure in the vacuum chamber (5), which ensures the downward movement of the combustion front through the charge bed placed in the agglomeration bowl (1). Combustion is initiated at the top surface and proceeds downward under the influence of suction created by the connected vacuum system.

Airflow into the system is regulated by the slide gate (3), which allows adjustment of the draft intensity and combustion rate. The temperature within the sintering bed is continuously monitored using the thermocouple (6), placed near the base of the agglomeration bowl.

The gaseous combustion products are drawn from the sintering zone into the dust collection unit (2) through a pipeline system. There, solid particles are separated from the gas stream. The purified gases, along with fine particulates, are collected in the storage bunker (4) located beneath the dust collector.

This configuration ensures stable operation of the sintering process under controlled thermal and airflow conditions, with efficient removal of combustion by-products and monitoring of the sintering temperature profile.



1 - the agglomeration bowl; 2 - dust collection unit; 3 - slide gate; 4 - storage bunker; 5 - vacuum chamber; 6 - thermocouple.

Figure 2. Installation for agglomeration.

The chemical composition of the synthesized agglomerate was determined by X-ray fluorescence (XRF) analysis using a ProSpector 2LE spectrometer (serial number P1775, Integrated Analytical Systems). The device enables the detection of elements ranging from magnesium (Mg) to uranium (U) with a detection limit of approximately 0.01 wt%.

Impact strength tests of the agglomerate were carried out according to GOST 24707-81, using the drop method. The analysis of the strength of the agglomerate makes it possible to assess its suitability for subsequent use in metallurgical aggregates, where high integrity and resistance of granules to mechanical destruction are important. The test samples weighing 5.00 kg were dropped from a height of 2 meters onto a steel plate. After the fall, the material was sieved, and the mass of the fraction with a particle size of more than 5 mm was determined. This indicator characterizes the impact strength of the agglomerate.

The impact strength (P) was calculated using the formula:

 $P = (m_1 / m_0) \times 100\%$

where:

 m_0 – the mass of the agglomerate before the test, kg;

 m_1 – fraction weight >5 mm after testing, kg.

3. Results and Discussion

In this section, the results of the experimental studies on the preparation and characterization of coke and sinter samples are presented and analyzed.

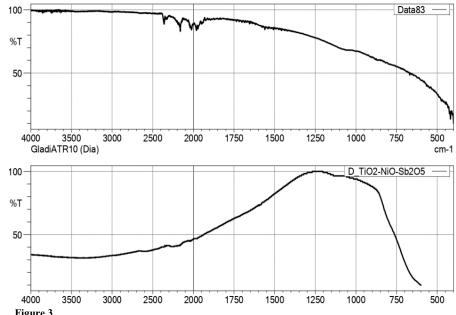
The chemical composition of the resulting coke is shown in Table 4.

Table 4. Chemical composition of coke.

| Carbon (C), % | Sulfur (S), % | Ash Content (Ash), % | Volatiles, % | Moisture (Wr), % |
|---------------|---------------|----------------------|--------------|------------------|
| 85 | 0.9-1.1 | 19.0 | 1.5–2.5 | ≤1 |

The resulting coke is characterized by a high carbon content (85%) and low humidity (<1%), which confirms its fuel suitability. Despite the increased values of ash content (19%) and sulfur (0.9–1.1%), this composition is acceptable for use in sintering production. The increased ash content and sulfur content are due to the use of high-ash Ekibastuz coal and spent anode materials as residues of electrolysis production of aluminum (spent anode material) containing sulfur compounds in the charge.

The results of the FTIR analysis of coke samples according to Figure 3 are presented below.



FTIR analysis results for coke sample 1 containing 50% Ekibastuz coal, 10% spent anode material, and 40% coal pitch.

The analysis of the FTIR spectrum of the Coke Sample 1 allowed us to identify the following key functional groups:

- 1. Aromatic compounds (C=C, ~1600 cm⁻¹): There is a pronounced absorption band in the range of 1580–1620 cm⁻¹, indicating the presence of aromatic hydrocarbons. These signals are characteristic of graphite-like structures, confirming the high degree of aromatization of the product.
- 2. Hydroxyl groups (O–H, 3200–3600 cm⁻¹): A wide band in the range of 3200–3600 cm⁻¹ is observed, indicating the presence of hydroxyl or phenolic groups. The intensity of the signal suggests residual moisture or the presence of oxygencontaining compounds after coking.
- 3. Carbonyl compounds (C=O, 1700-1750 cm⁻¹): A peak in this region indicates the presence of carbonyl fragments such as ketones or carboxylic acids, which may be the result of incomplete dehydration.
- 4. Aliphatic C–H compounds (2800–3000 cm⁻¹): Weak peaks of C–H valence vibrations in aliphatic fragments indicate a low content of volatile hydrocarbons, which confirms a high degree of carbonization.
- 5. Ether and phenolic groups (C–O, 1050–1150 cm⁻¹): The presence of a signal corresponding to C–O oscillations indicates the presence of oxygen-containing fragments (phenols, esters).
- 6. Mineral components (500–900 cm⁻¹): Peaks in this region are characteristic of deformation vibrations of metal oxides (TiO₂, Fe₂O₃, Al₂O₃) originating from ash components.

Sample 1 demonstrates a high degree of graphitization and a low content of volatile substances. The moderate content of oxygen-containing functional groups indicates a good level of coking. However, due to the relatively low proportion of

spent anode material, the degree of carbonization is lower than that of other samples, which makes this composition acceptable, but not optimal for metallurgical applications. Also, the increased content of ash components requires further control when used in metallurgical processes.

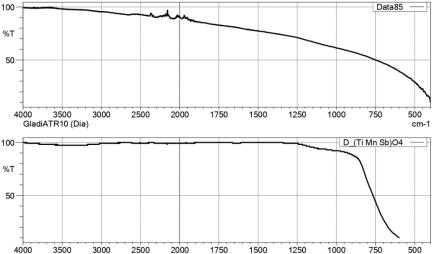
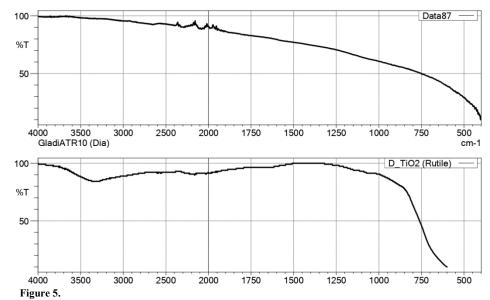


Figure 4. FTIR analysis results for coke sample 2 containing 50% Ekibastuz coal, 20% spent anode material, and 30% coal pitch.

The analysis of the FTIR spectrum of the coke sample 2 showed the following spectral features:

- 1. Aromatic compounds (C=C, ~1600 cm⁻¹): An intense peak in this region indicates the presence of aromatic carbon structures, indicating a high degree of aromatization.
- 2. Hydroxyl groups (O-H, 3200-3600 cm⁻¹): A high-intensity wide band indicates the presence of hydroxyl or phenolic groups, probably due to residual moisture or incomplete dehydration.
- 3. Carbonyl compounds (C=O, 1700–1750 cm⁻¹): A more pronounced band than in sample 1 indicates the presence of carbonyl compounds such as quinones or residual acids, which may indicate the oxidation of hydrocarbons.
- 4. Aliphatic C–H compounds (2800–3000 cm⁻¹): Very weak peaks confirm a high degree of carbonization and utilization of volatile compounds during heat treatment.
- 5. Ether and phenolic groups (C–O, 1050–1150 cm⁻¹): Peaks in this range are more intense than in sample 1, which may indicate a higher proportion of residual resinous substances due to the higher content of spent anode material.
- 6. Mineral components (500–900 cm⁻¹): More pronounced peaks indicate the presence of metal oxides (Fe₂O₃, TiO₂, etc.).

Sample 2 shows a high degree of aromatization and carbonization, while retaining a moderate number of functional groups that ensure structural integrity. Although the presence of oxygen-containing compounds and ash components requires adjustments, the balance between graphitization and structural connectivity makes this sample promising.

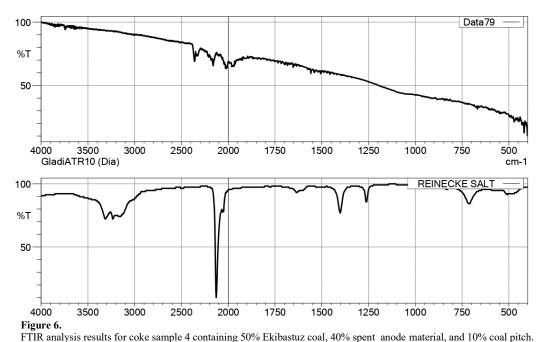


FTIR analysis results for coke sample 3 containing 50% Ekibastuz coal, 30% spent anode material, and 20% coal pitch.

The analysis of the FTIR spectrum of coke sample 3 revealed the following key functional groups:

- 1. Aromatic compounds (C=C, ~1600 cm⁻¹): An intense band in the range of 1580-1620 cm⁻¹ was recorded, indicating the presence of aromatic structures typical of graphitized carbon. This indicates a high degree of aromatization of the product.
- 2. Hydroxyl groups (O–H, 3200–3600 cm⁻¹): A wide absorption band in this region indicates the presence of hydroxyl or phenolic groups. This may be due to both residual moisture and incomplete removal of oxygen-containing compounds during coking.
- 3. Carbonyl compounds (C=O, 1700–1750 cm⁻¹): The presence of carbonyl groups (e.g. ketones or acids) is confirmed by an intense peak in this region, which may indicate residual organic compounds and oxidative processes.
- 4. Aliphatic C-H compounds (2800-3000 cm⁻1): Weak bands of C-H valence vibrations indicate a low content of aliphatic fragments, which confirms the advanced stage of carbonization.
- 5. Ether and phenolic groups (C-O, 1050-1150 cm⁻¹): The presence of the C-O signal indicates the presence of residual oxygen-containing compounds.
- 6. Mineral components (500–900 cm⁻¹): Peaks in this range indicate the presence of ash compounds (Fe₂O₃, TiO₂, CaO and Al₂O₃), characteristic of spent anode material and Ekibastuz coal.

Sample 3 has a pronounced aromatic structure, good heat resistance, and a moderate content of residual organic compounds. Despite some ash content, this sample demonstrates optimal carbonization and mechanical strength, which makes it the most preferable in terms of a set of parameters.



1 The marysis results for coke sample 4 containing 30/8 Extraostal coar, 40/8 spent anothe material, and 10/8 coar p

The analysis of FTIR spectrum of coke sample 4 showed the following characteristic features:

- 1. Aromatic compounds (C=C, ~1600 cm⁻¹): The intense absorption band indicates the presence of aromatic structures typical of graphite-like carbon, which confirms the high level of carbonization.
- 2. Hydroxyl groups (O–H, 3200–3600 cm⁻¹): A wide band in this range indicates the presence of hydroxyl and/or phenolic groups, as well as residual moisture, especially characteristic of products with a high content of spent anode material.
- 3. Carbonyl compounds (C=O, 1700–1750 cm⁻¹): A pronounced peak in this region indicates the presence of carbonyl compounds, probably quinones or oxidation products.
- 4. Aliphatic C–H compounds (2800–3000 cm⁻¹): Weak peaks indicate partial preservation of aliphatic structures, probably from coal pitch.
- 5. Ether and phenolic groups (C–O, 1050–1150 cm⁻¹): The intensity of the bands in this range confirms the presence of oxygen-containing functional groups such as esters and phenols.
- 6. Mineral components (500–900 cm⁻¹): The spectrum shows clear peaks characteristic of metal oxides (Fe₂O₃, Ti₂O, Al₂O₃), which indicates a high content of ash impurities.

Sample 4 is characterized by a high degree of aromatization, but also by an increased content of oxygen-containing groups. This can affect the reactivity of coke and lead to deterioration of the mechanical properties of coke, reducing its technological suitability for sintering production.

A comparative analysis based on the results of FTIR spectroscopy showed:

FTIR spectroscopy of all samples revealed the presence of key functional groups characteristic of carbon-containing and oxygen-containing compounds: aromatic C=C (~1600 cm⁻¹), alkyl C-H (~2850–2950 cm⁻¹), hydroxyl⁻OH (~3200–3600 cm⁻¹), carbonyl C=O (~1700 cm⁻¹), as well as ether and phenolic C-O and C-O-C (~1000–1300 cm⁻¹). The intensity

of the bands associated with oxygen-containing and aliphatic groups decreases as the spent anode material content increases and the proportion of coal pitch decreases.

This indicates a higher degree of carbonization and graphitization of coke with an increase in the proportion of spent anode material in the charge, especially in sample 4. At the same time, in samples with a lower proportion of pitch, there is a decrease in the content of functional groups involved in the formation of bonds between carbon fragments, which can potentially worsen the mechanical strength of the resulting coke.

Sample 2 (50 % Ekibastuz coal, 20 % spent anode material, 30 % pitch) and 3 (50 % Ekibastuz coal, 30 % spent anode material, 20 % pitch) demonstrated the optimal balance between a high degree of carbonization and the preservation of a sufficient number of oxygen-containing groups necessary for the formation of a solid structure. This makes it the preferred option in terms of mechanical performance and adaptability.

Despite the maximum carbonization, sample 4 (40 % spent anode material, 10 % pitch) shows signs of excessive removal of binding functional groups, which can lead to a decrease in the strength of coke during agglomeration pressing and transportation.

Thus, samples 2 and 3 can be considered as the best option in terms of the combination of technological, energy and structural characteristics. Based on the analysis results, these samples were used for further practical studies of the possibility of using the obtained coke in sintering production.

Figure 7 shows the appearance of the agglomerates obtained in experiments No. 1 and No. 2. The agglomerate formed using coke with 20 % spent anode material (experiment No. 1) is characterized by high density, uniformity of granules and mechanical strength, which visually confirms the integrity of the structure. The sample from experiment No. 2 (with 30 % spent anode material) retains similar morphological characteristics, however, macroscopic observation shows a slight increase in surface heterogeneity, which may be due to a local uneven distribution of graphite-like inclusions.





Experiment No. 1 Experiment No. 2

Figure 7.

Two agglomerate samples: Sample 1 – agglomerate using coke with 20% spent anode material; Sample 2 – agglomerate using coke with 30% spent anode material

Table 5 presents the results of X-ray fluorescence analysis of the chemical composition of the agglomerate obtained during laboratory agglomeration tests. Two variants of the charge were investigated, in which coke containing 20% (experiment No. 1) and 30% (experiment No. 2) spent anode material, respectively, was used as the coke component.

Table 5. Chemical composition of the agglomerate.

| Experiment No. | Fetotal | FeO | Fe ₂ O ₃ | CaO | SiO ₂ | MgO | Al ₂ O ₃ | MnO | S | P |
|----------------|---------|-------|--------------------------------|-----|------------------|-----|--------------------------------|------|------|------|
| 1 | 51.1 | 13.65 | 10.9 | 5.2 | 7.5 | 3.7 | 6.55 | 1.19 | 0.48 | 0.04 |
| 2 | 50.85 | 13.8 | 11.3 | 4.8 | 7.8 | 3.4 | 6.9 | 1.2 | 0.52 | 0.04 |

In both samples, the total iron content (Fe $_{total}$) remains above 50%, confirming the suitability of the agglomerate for use in blast furnace smelting. The FeO and Fe $_2O_3$ contents range from 13.65 to 13.80% and from 10.90 to 11.30%, respectively, indicating a balanced distribution of reducible iron phases. Calcium oxide (CaO), acting as a flux, is present in the range of 4.8–5.2%, while silica (SiO $_2$) content increases slightly with the higher proportion of spent anode material, remaining within acceptable limits (7.5–7.8%) for blast furnace-grade sinter.

The levels of MgO, Al₂O₃, and MnO remain technologically acceptable and reflect the increasing influence of coal pitch and anode additives. Sulfur (S) and phosphorus (P) contents are also within the permissible range for metallurgical

use. The sulfur content slightly increases to 0.52 % at 30 % spent anode material, while phosphorus remains stable at 0.04 %, not exceeding critical thresholds.

These results confirm that even with up to 30% addition of spent anode material, the resulting sinter meets key compositional criteria required for use in ferrous metallurgy.

Table 6 shows the results of the mechanical strength of the agglomerate.

Table 6.The results of testing the mechanical strength of the agglomerate.

| Sample | Content of spent | Weight before the test | Fraction weight >5 | Impact strength (%) |
|--------|--------------------|------------------------|--------------------|---------------------|
| | anode material (%) | (kg) | mm (kg) | |
| No. 1 | 20 | 5.00 | 3.90 | 78.0 |
| No. 2 | 30 | 5.00 | 3.60 | 72.0 |

Both agglomerate samples exhibit acceptable resistance to impact loading. The sample containing 20 % of spent anode material demonstrated higher mechanical strength (78 %) than the sample with 30 % spent anode material (72 %). The observed reduction in impact resistance at higher spent anode material is likely attributed to structural alterations in the coke matrix and a decrease in cohesive interactions between particles.

Despite the reduction, both values remain within acceptable limits for use in blast furnace processes. The yield of suitable agglomerate was 73.5 % in Experiment No. 1 (20 % spent anode material) and 71.2 % in Experiment No. 2 (30 % spent anode material), indicating that coke modified with spent anode material retains sufficient agglomeration performance.

The results confirm that integrating spent anode material into the Ekibastuz coal blend increases the carbon content of the coke and enables effective recycling of industrial aluminum smelting waste. Microstructural and spectral analyses support the conclusion that the optimal spent anode content lies within the $20-30\,\%$ range. Exceeding this threshold results in degradation of coke structure, reduced uniformity, and diminished bonding properties. Nevertheless, agglomerates produced with $20-30\,\%$ spent anode material demonstrate sufficient chemical and mechanical characteristics for application in blast furnace operations.

4. Conclusions

The experimental research confirmed the feasibility of producing coke suitable for the sintering process by modifying Ekibastuz coal with spent anode material (a byproduct of aluminum production). Four charge compositions were analyzed, containing 10–40% of spent anode material and varying amounts of coal pitch. Fourier-transform infrared spectroscopy (FTIR) was used to evaluate the structural composition of the resulting coke samples. The following key findings were established:

- Samples containing 20–30% of spent anode material demonstrated optimal structural characteristics, including a high degree of carbonization and a balanced presence of aromatic and oxygen-containing functional groups.
- Excessive increase of spent anode material up to 40% led to a noticeable reduction in binding functional groups, potentially lowering the mechanical strength of the resulting coke.
- Laboratory agglomeration tests using modified coke confirmed its suitability for sintering. The resulting sinter agglomerates demonstrated acceptable technological and chemical properties.
- The yield of usable agglomerate ranged from 72.4% to 73.5%, confirming the effectiveness of coke with 20–30% anode material content for use in sintering processes.

The integration of spent anode material into coke production not only enhances material properties but also promotes resource conservation, waste utilization, and environmental sustainability, offering a viable pathway for circular economy practices in the metallurgical industry.

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