

Qualitative and chemical characteristics of mineral matter in the selected lignite deposits in light of their suitability for clean coal technologies

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Abstract. An analysis of the variability of ash content and chemical composition of coal ash is crucial when assessing the possibility of using this raw material in clean coal technologies (CCS). The paper focuses on chemical, technological and quality characteristics of ash from the combustion of lignite from Polish deposits. Ash content on a dry ash-free basis for the samples from 9 deposits varies from 1 to 50%. The most commonly observed ash content in the studied samples of lignite is in the range between 10-20%. The measured ash melting temperatures from the collected lignite samples have shown that the sintering, softening, melting and flow temperatures, determined in an oxidizing atmosphere (air), are higher than those determined in a reducing atmosphere. In the majority of Polish lignite deposits, the dominant component of ash was SiO₂, followed by CaO. When evaluating the suitability of coal in energy production, with particular emphasis on the combustion and gasification processes, it is also necessary to study the behavior of ash at high temperatures and the impact of ash on the gasification and combustion facilities. Ash fouling and slagging processes, which are heavily dependent on the ash content, composition, melting point and the content of chlorine, phosphorus and sulfur in coal, have a negative effect on the combustion process.

1 Introduction

Lignite is one of the most important energy sources in the world. However, the introduction of the new EU energy and environmental policy has resulted in advances in innovative and more environmentally friendly technologies using coal, known collectively as clean coal technologies (CCT). Full implementation of the above mentioned technologies will open new applications for this energy raw material, which could strengthen its position in the market.

The direct combustion of lignite is the most common form of producing energy from this raw material (municipal and industrial heat and electricity production).

Currently, the combustion of coal is carried out mainly in fluidized bed boilers, pulverized coal-fired boilers and, to a lesser extent, in grate boilers. However, in order to meet the expected coal quality standards, related mainly to the environmental protection, particularly emissions limits for sulfur and nitrogen oxides, particulate matter and mercury [1], it is necessary to introduce increasingly better and cleaner technologies. In recent years, major advances in new coal technologies have been done. New coal technologies are being introduced while those already known are being improved. One of them is the gasification of coal.

The gasification of coal can be carried out via surface and underground gasification technologies. Coal gasification is a key technology for the production

of energy from coal while taking care of the environment. The versatility and flexibility of gasification technologies enables the production of a wide range of products, including mainly liquid fuels, chemical fuels, gas fuels and many others. As opposed to conventional combustion, this technology has a positive impact on the environment because of its ability to capture CO₂ before the gasification process. The capture of carbon dioxide allows obtaining a gas producing much less harmful substances during the combustion process when compared to the conventional combustion of coal. In this process, 99% of sulfur and other harmful substances can be removed and processed for industrial use. Currently, there are about 200 coal gasification plants in the world, while 17 are under construction and another 50 are being planned [2]. Lignite is the main raw material used in the gasification process. According to the forecasts of the US Department of Energy, it will maintain its dominant position in the coming years [2].

In Poland, there are 90 documented lignite deposits, mainly in the Paleogene and Neogene sediments in the Polish Lowlands. The total proved recoverable reserves of lignite amount to 23 510.59 million tonnes.

The proved recoverable reserves in currently exploited deposits amount to 1 482.69 million tonnes, representing 6.3% of the total proved recoverable reserves. Lignite from these deposits is extracted in 5 mines, namely: Bełchatów, Turów, Adamów, Konin

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and Sieniawa lignite mines. Given the current level of consumption, these reserves should last for another several decades. As of today, lignite is mainly used in large plants such as power stations.

2 The impact of the mineral matter on the suitability of coal for CCT

However, it should be underlined that coal is a mixture of carbonized organic matter represented by macerals and mineral matter. Therefore, the ash content (content of non-combustible mineral matter) is an extremely important parameter when assessing the possibility of using coal for combustion purposes. The term ash content applies to the residues (unburned mineral matter) produced after the complete combustion of coal under certain conditions. The content and composition of the non-combustible mineral matter in coal affect the quality of coal, mainly by reducing the gross calorific value [3], thus reducing its usefulness for energy purposes. The ash content of coal determines the possibility of its use in gasification and liquefaction processes.

The content and the type of mineral matter contained in the combusted coal also have an impact on this process. According to the current guidelines, the ash content (dry basis) of coal subjected to gasification process should be less than 40%. According to Shirazi et al. [3], the amount of the mineral and chemical matter and their composition can significantly reduce the amount of energy obtained due to the need to use part of it in order to heat the non-combustible materials. It has been observed that the energy loss was more significant in pulverized coal-fired boilers. Ash components react with each other at high temperatures and - forming new mineral phases and volatile compounds - affect the combustion process [4, 5, 6]. The most troublesome is the formation of fly ash, slag and chemical compounds inside the furnace, resulting in corrosion of boilers. The knowledge of the ash melting point is crucial when estimating the impact of mineral matter on a given combustion technology. The individual minerals have different melting points. The low ash melting point is the reason for many negative phenomena during combustion processes. The vitrified ash damages grate of the boiler and its housing material hinders the circulation of air and sticks to the unburned coal, which contributes to a decrease in thermal efficiency of the furnace. It can also - reacting with the material of the furnace walls - form low melting slag, which can lead to serious accidents. The ash melting point depends on its chemical composition. Generally, the more silicates and fewer oxide connections of iron and alkali, the higher the ash melting temperature. Both softening and melting of ash are very complex process. These reactions run parallel to the other chemical reactions, which are highly dependent on process time, fragmentation and arrangement of the ash components. Ash components react with each other and with their decomposition products both before melting and during the reaction [7]. The least troublesome in this respect is quartz, which has higher melting point (about 1800 °C) than

the temperatures occurring in furnaces [6] and therefore does it does not pass into the liquid state.

Ash fouling and slagging processes, which are heavily dependent on the ash content, composition, melting point and the content of chlorine, phosphorus and sulfur in coal, have a negative effect on the combustion process [8]. In order to determine the tendency to form unfavorable residues in the furnace, the alkalinity/acidity, slagging and SiO₂ ratios, as well as the tendency to contamination of heating surfaces (fouling), are determined [9]. According to Crossley [10], coals with chlorine content above 0.3%, phosphorus content greater than 0.03% and sulfur content over - 1.8% have the greatest tendency to slagging and fouling. According to Atakül et al. [11] this tendency is directly proportional to the Na₂O content and inversely proportional to the Al₂O₃ content. On the other hand, Róg [9] suggests that the higher the aluminum silicate content and the lower the iron and alkali content, the higher the melting point of ash and lower tendency to fouling.

Kaolinite decomposes already at 450 °C, losing OH groups in the crystal structure, while clay minerals (mainly illite and smectite) begin to decompose at about 500 °C to form new mineral phases, which can also contribute to the plastic deformation of ash and the formation of slag, which is difficult to remove.

Calcite and dolomite decompose at about 900°C, resulting in the formation of calcium oxide (CaO), which - upon reaction with water vapor - forms artificial minerals like portlandite (Ca(OH)₂) and interacts with aluminosilicates to form gehlenite (Ca₂Al₂SiO₇) and anorthite (CaAl₂Si₂O₈). Carbonates, especially calcite, can react with sulfur contained in coal and form the artificial mineral phase chemical composition typical for anhydrite, thereby permanently binding the sulphur [12]. This process has been used in one of the methods of dry flue gas desulfurization (flue gas resulting from the combustion).

Despite the fact that combustion technologies are constantly being improved, the gasification of coal is considered as one of the cleanest coal technologies. Gasification can be carried out in several types of gasifiers. The choice of the technology used depends largely on the quality of coal, defined by the following parameters: the maximum ash content, maximum moisture of fuel, ash melting temperature, reactivity, and the rank of coal. The rank of coal and reactivity of the fuel allow to determine whether the coal can be used in all types of gasifiers. The maximum moisture content below 35% or 5% depending on the type of gasifier can prevent the use of a given lignite type. In the case of fixed bed gasifiers, the maximum moisture content below 35% is good enough. Fluidized bed and entrained flow gasifiers require the complete drying of coal. The ash content and composition have significant impact on the gasification process because some minerals can act as catalysts and others as inhibitors, while the high content of mineral matter causes a decrease in system efficiency [13, 14] Therefore, the maximum permissible content of mineral matter in coal depends on the gasification technology used. Different ash melting points determine the choice of gasification technology

and the removal of residues resulting from the process. Therefore, the knowledge of this parameter for a given fuel is necessary before designing the gasifier. In this context, the ash content, oxide composition and the ash melting point are of crucial importance when it comes to determining the suitability of minerals for gasification process.

According to Sekine et al. [15] the Ca, K and Na content improves the reaction rate. On the other hand, Johnson [16] suggests that dispersed alkaline substances can act as a catalyst and the reactivity is independent from the coal rank of the gasified coal, solely depending on the type of mineral matter. On the contrary, silicon (Si) and aluminum (Al) slow down the gasification of coal and tend to suppress the aforementioned process [15].

Skodras and Sakalloropoulos [17] have correlated the alkalinity/acidity index - hereafter referred to as alkali index, characterized by Sakawa et al. [18] – to the reactivity of coal during the gasification process. They have found that there is a clear relationship between the Ca content and the reactivity of coal, unlike in the case of Mg, K, Na, Fe and the reactivity. In addition, it has been found that oxides, mainly SiO₂, CaO and iron oxide can adversely affect refractory materials of the reactor, contributing to their corrosion [19].

The ash melting point, which determines the technology used (Table 1) is another parameter crucial for the gasification process [13, 20]. This parameter can be correlated as a function of the SiO₂, Al₂O₃, TiO₂, Fe₂O₃, CaO, MgO, Na₂O and K₂O content in ash [21, 22]. The alkalinity/acidity index of ash can be used to correlate the ash melting point and its chemical composition. For example, the high alkali content in Ca, Fe, K₂O and Na₂O oxides in coal suggest low ash melting point. In the case of entrained flow gasifiers, the melting point of coal ash should be lower than their operating

temperature in the range 1400-1600 °C. In the case of fluidized bed gasifiers it is necessary to use coal with ash melting point higher than the operational temperature of the reactor (1000 °C) in order to prevent ash slagging processes, favored by high pyrite and sodium silicate content in the gasified coal.

3 Research methodology

The study included field work and laboratory work. Field work was aimed at the collection of lignite samples, which were subjected to a series of laboratory tests. In total, about 150 lignite samples have been collected and used for proximate analysis. In addition, a large database consisting of archival results from mines and scientific literature has been collected. All the results obtained were used in the statistical analysis. The studies have used the lignite samples collected from the currently exploited and perspective deposits. The distribution of these deposits is shown in Figure 1.

Research on the properties and usability of different coal types involves technical, physico-chemical and petrographic analysis of coal. Laboratory tests of the collected lignite samples included:

- a-Technical and physical analysis of coal,
- b-Chemical analysis of coal and coal ash. After the determination of free moisture content, the samples collected from lignite deposits were dried and grinded. The chemical and technical analysis used coal with a grain size of 0.02 mm. Technical analysis of coal has been carried out in accordance with the applicable standards for determining:
 - Total and analytical moisture
 - The ash content
 - The ash melting point
 - The sand content
 - The oxide composition of ash.

Table 1. The characteristics of gasification reactors [20, 23; supplemented].

Reactor type	Fixed bed gasification		Fluidized bed gasification		Entrained flow gasification
	Dry at the bottom	Slag	Dry ash	Agglomerated ash	
Ash collection conditions	Dry at the bottom	Slag	Dry ash	Agglomerated ash	Slag
Typical designs	Lurgi	BGL	Winkler, HTW, KBR, CFB, HRL	KRW, U-Gas	KT, Shell, GEE, E-Gas, Siemens, MHI, PWR
Requirements for fuel					
Preferred fuel	lignite, hard coal, anthracite, waste	hard coal, anthracite, petroleum coke, waste	lignite, hard coal, anthracite, waste	lignite, hard coal, anthracite, waste biomass, petroleum coke	lignite, hard coal, anthracite, petroleum coke
Particle size	6-50 mm	6-50 mm	6-10 mm	6-10 mm	<100um
The maximum ash content%	unlimited	<25 (preferred)	unlimited	unlimited	<25 (preferred)
The maximum moisture%	<35	<35	<5	<5	<5
Ash melting temperature °C	>1200	<1300	>1100	>1100	<1300
The preferred rank of coal	All	High	Low	All	All
Reactivity	Low	Low	High	High	High



Figure 1. Location map of lignite deposits, from which samples for testing were collected [24; completed].

In order to determine the tendency for the formation of unfavorable by-products (residues) in the furnace, the following ratios were determined:

1) The alkalinity/acidity index (*base/acid (B/A) ratio*)
 $B/A = \%(\text{Fe}_2\text{O}_3 + \text{CaO} + \text{MgO} + \text{Na}_2\text{O} + \text{K}_2\text{O}) / [\%(\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{TiO}_3)]$ (1)

$\text{SiO}_2, \text{Al}_2\text{O}_3, \text{TiO}_3, \text{Fe}_2\text{O}_3, \text{CaO}, \text{MgO}, \text{Na}_2\text{O}, \text{K}_2\text{O}$, – the percentage of individual oxides in the ash.

2) The slagging index (*Rs*)

$$R_s = B/A \cdot S_{t^d} \text{ or } R_s = B/A \cdot \text{Fe}_2\text{O}_3$$
 (2)

S_{t^d} – total sulfur content in coal (dry basis) Depending on the R_s value, the studied coal is characterized by:

- Low tendency to slagging – $R_s < 0.6$,
- Medium tendency to slagging – $0.6 < R_s < 2.0$,
- High tendency to slagging – $2.0 < R_s < 2.6$,
- Very high tendency to slagging – $R_s > 2.6$

3) The silica value, (*SV*)

$$SV = (\text{SiO}_2 \cdot 100) / (\text{SiO}_2 + \text{Fe}_2\text{O}_3 + \text{CaO} + \text{MgO})$$
 (3)

Depending on the SV value, the studied coal is characterized by:

- Low tendency to slagging – $SV > 72$,
- Medium tendency to slagging – $65 < SV < 72$,
- High tendency to slagging – $SV < 65$.

4) The tendency to fouling of the heating surfaces (*Fouling Index, Rf*)

$$R_f = B/A \cdot (\text{Na}_2\text{O} + \text{K}_2\text{O})$$
 (4)

Depending on the R_f value, the studied coal is characterized by:

- No tendency to fouling – $R_f < 0.6$,
- High tendency to fouling – $0.6 < R_f < 40.0$,
- Very high tendency to fouling – $R_f > 40.0$.

5) The alkali index *AK*

$$AK = \text{Na}_2\text{O} + 0.96559\text{K}_2\text{O} (A^d/100)$$
 (5)

A^d – the percentage of ash content in coal (dry basis) Depending on the AK value, the studied coal is characterized by:

- Low tendency to fouling with alkali components – $AK < 0.3$,
- Medium tendency to fouling with alkali components – $0.3 < AK < 0.45$,
- High tendency to fouling with alkali components – $0.45 < AK < 0.6$,
- Very high tendency to fouling with alkali components – $AK > 0.6$ [25].

4 Research results

The average results of the analysis for the individual deposits are shown in Table 2.

In the case of the examined lignite deposits, the ash content (A^d) on a dry basis is very variable parameter. For the aforementioned deposits, the average ash content on a dry basis ranges from 8.0% in the Turów deposit to 25.9% in the Szczerców deposit (Table 2). In the individual samples, this parameter is in the range from 4.9% in the Turów deposit to 68.7% in the Gubin deposit, with an average value of 20.3%. The asymmetrical distribution with a positive skewness can suggest a tendency for higher ash content in the lignite, which is clearly visible. The coefficient of variation for the ash content on a dry basis is 66.4%, which is fairly strong. The most frequently observed ash content in the studied lignite samples is in the range between 10-20%.

Sand, a granular fraction consisting of hard mineral grains, is a harmful element in the mineral matter of coal in ash resulting from the combustion of lignite. The main component of sand is quartz, followed by feldspar and mica. In the studied deposits, the sand content on a dry basis (p^d) is very variable – the highest in the Ościslówo deposit, reaching on average to 8.3%, while the lowest in the Adamów deposit – 1.4% (Table 2). The above mentioned variability is very strong also

Table 2 The average chemical and technological parameters of domestic lignite deposits.

Deposit	Ash content A^d [%]	Sand content p^d [%]	Ash melting point t_B [°C]	CaO content [%]	Alkalinity/ acidity B/A [%]	Slagging index R_s	Silica value SV	Fouling index R_f	Alkali index AK
Adamów	8.6	1.4	1375	2.91	1.69	0.43	34.53	0.07	0.03
Bełchatów	18.0	4.4	1285	3.82	0.37	0.43	67.78	0.11	0.22
Drzewce	12.7	3.2	1310	3.73	0.49	0.75	66.34	0.06	0.05
Gubin	19.2	2.7	1300	2.52	0.38	0.79	73.28	0.31	0.65
Józwin	16.8	5.0	1365	3.80	0.55	1.04	64.34	0.07	0.10
Ościslówo	25.4	8.3	>1500	2.93	0.25	0.44	79.73	0.01	0.03
Sieniawa	7.5	2.7	1390	2.03	-	-	-	-	-
Szczerców	25.9	4.4	1356	3.90	0.76	1.48	53.29	0.13	0.12
Turów	8.0	5.7	1345	0.65	0.34	0.15	76.70	2.48	6.52

in the case of individual samples of lignite, with a coefficient of variation amounting to 158% and an average sand content of 4.3%. While there is practically no sand in some of lignite samples from the Szczerców deposit, the highest sand content, reaching up to 47.7%, has been shown in the Gubin deposit. Generally, the examined samples contained up to 5.0% sand. In the case of Polish lignite deposits, a very strong variability of this parameter, with the average sand content of about 7.4%, can be observed.

The calcium content of coal, expressed by the CaO content, is associated with the mineral matter present in coal, especially - calcite (CaCO_3), partially aragonite (CaCO_3) and - occasionally occurring in coal deposits - calcium aluminum hydroxycarbonate e.g. paralumohydrocalcite ($\text{CaAl}_2(\text{CO}_3)_2(\text{OH})_4 \cdot 6\text{H}_2\text{O}$) [26]. However, it should be noted that part of the calcium, along with Fe, Mg, K and Na, reacts with the mineral matter (coal) to form forms dopplerite, a calcium salt [27]. The calcium oxide content in the tested lignite ranges from 0.65% in the Turów deposit to 3.9% in the Szczerców deposit, while the average calcium content in the lignite is determined to be 2.85%. The coefficient of variation for this parameter is 82.39%, which indicates a strong variation of this parameter and at the same case suggests that it is difficult to estimate using statistical methods. The highest CaO content in domestic lignite deposits in Poland is observed in the Szczerców deposit [28]. In other deposits, this value usually does not exceed 5.00% [29]. This finding has also been confirmed in the presented study, as the CaO content in the majority of the examined lignite is in the range of 2-4%.

The ash melting temperatures of the collected lignite samples have shown that the sintering, softening, melting and flow temperatures, determined in an oxidizing atmosphere (air) are higher than those determined in a reducing atmosphere (a mixture of CO and CO_2 in the ratio 3:2). The melting point (t_B) in an oxidizing atmosphere ranges from 1285 °C (the Bełchatów deposit) to over 1500 °C (the Oświęcimo deposit) while a reducing atmosphere can range from 1170 °C (the Szczerców deposit) to > 1500 °C. This parameter is also highly variable for individual deposits.

The average chemical composition of ash, resulting from slow incineration of lignite, in the examined deposits indicates that in all of them, with the exception of the Adamów deposit, the dominant component of ash is SiO_2 , ranging from 30.23% in the Adamów deposit to 67.7% in the Oświęcimo deposit. In the former, the dominant component of ash is CaO (40.39%), while the average content of this component in other deposits ranges from 8.9% in the Turów deposit to 25.5% in the Szczerców deposit. The share of Na_2O is usually low, in the range between 0.03% in the Adamów deposit and 6.5% in the Turów deposit. The content of magnesium oxide (MgO) is also limited, ranging from 0.20% to 5.02%. The aluminum oxide content (Al_2O_3) is highly variable and ranges from 1.01% in the Oświęcimo deposit to 17.23% in the Turów deposit. The average P_2O_5 content in ash of the analyzed raw material is below 0.31%, with the exception of one

sample from the Józwin deposit, where it has reached 1.09%. Similar low levels have been determined for potassium oxide (K_2O), with the highest value, 2.77%, in the Gubin deposit, and the lowest - 0.01% - in deposits located in the Konin and Turek area. Sulfur oxide (SO_3) in the tested lignite ash ranges from 5.7% in the Turów deposit to 14.8% in the Gubin deposit. A high content of this component can be associated with the fact that sulphates in the lignite are not decomposing in the combustion process, as opposed to sulphides and organic sulfur [30]. The iron oxide (Fe_2O_3) has a significant share in the tested lignite ash, ranging from 2% in the Bełchatów deposit to 14.1% in the Adamów deposit. The ashes also contain manganese (MnO) and titanium oxides (TiO_3), but their share in the tested samples does not exceed 1%.

When it comes to the analyzed oxides, the content of Na_2O , P_2O_5 , K_2O and MnO in the individual lignite ash samples is highly variable, which could also suggest that the estimation of their share in the ash using the ashes statistical methods will be difficult. The low variability can be observed only for the SiO_2 content in ash.

Among the tested lignite samples, the least favorable parameters regarding the tendency to slagging have been shown for the Adamów and Szczerców deposits, where a very high tendency to slagging has been observed.

The high and very high tendency to fouling of the heating surfaces with alkali components has been shown for ashes from the Gubin and Turów deposits.

5 Conclusions

The analysis has shown that the quantity and quality of mineral matter in lignite has a large impact on its suitability for clean coal technologies. According to a preliminary analysis of the selected parameters from several deposits in Poland, it can be stated that the ash content is a very variable parameter for both individual deposits and all of the tested lignite. The ash content is a parameter limiting the possibility of use of coal for the gasification processes. It is assumed that the ash content for the most promising technologies should not exceed 25%. Therefore, a significant portion of the resources has been classified as unsuitable for gasification purposes.

The analysis of ash melting temperatures allows concluding that the tested lignite can be successfully used for fluidized bed gasification. The ash melting point in all of the tested deposits is higher than 1100 °C, required for the fluidized bed gasification. The CaCO_3 content in some of the tested deposits is unfavorable for the gasification process. The aforementioned deposits occur mainly in the Bełchatów area, where large amounts of chalk lake - accompanying the lignite seams - can be observed. However, the majority of the tested lignite ash shows no tendency to fouling and slagging.

On the basis of the presented analysis it can be concluded that the Polish lignite is an excellent energy raw material for advanced coal technologies. However, due to the high variability of the key technological

and chemical parameters, its quality must be constantly monitored.

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