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Petrographic composition of char from the gasification of coal from the Wieczorek Mine after combustion

Introduction

Gasification is a modern coal processing technology that can be used to replace direct coal combustion with cleaner energy. Gasification enables more environmentally friendly energy generation and is a very flexible technology in terms of the fuels used (Higman and van der Burgt 2008). The development of technology for the gasification of coal from Polish deposits was the subject of the project titled “The Development of Coal Gasification Technology for High Production of Fuels and Electricity” funded by the National Center for Research and Development within the strategic program of research and development entitled: “Advanced Energy Generation Technologies”. As a result of this project, coal, dust, and ash samples were obtained. The presented work includes a petrographic analysis of coal from

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the Wieczorek mine, the char formed in the gasification process, and bottom and fly ashes from the combustion of coal and char.

The petrographic analysis of ash from char combustion may be helpful in the determination of possible ways of its utilization. Some of the possible applications of ash, including the use as backfilling material, which were presented by Pomykała (Pomykała 2015). Mazurkiewicz et al. (Mazurkiewicz et al. 2012) show the following possibilities of ash management: the production of solidified ash slurries used for the hardening (stabilization) of embankments, and dumping grounds; local and other roads; production of cementless binders; production of Portland clinker; as an active cement additive; concrete production; production of light aggregates; road construction; use in agriculture and horticulture; for self-solidifying backfill in mining.

The analysis of ashes, in particular residues of coal particles, are important due to their impact on the quality of concrete (Galos and Uliasz-Bocheńczyk 2005; Brzozowski 2011) and road binders (Gawlicki and Wons 2011). The petrographic composition of fly ash from coal combustion was described, among others, by Misiak (Misiak 2015), Fenelonov et al. (Fenelonov et al. 2010), Sokol et al. (Sokol et al. 2002), Ratajczak et al. (Ratajczak et al. 1999), and Hower et al. (Hower et al. 1996). The petrographic analysis of bottom ash from the combustion process was presented, among others, by Bartoňová et al. (Bartoňová et al. 2007), Demir et al. (Demir et al. 2008), and Predeanu et al. (Predeanu et al. 2016). Until now, a comprehensive petrographic characteristic of fly ash and bottom ash generated from combustion of char has not yet been presented. This paper fills this gap.

1. Research methodology

The starting material, which was subjected to the gasification process, was bituminous coal from the Wieczorek mine. The coal was subjected to gasification carried out in the pilot plant of the Institute for Chemical Processing of Coal in Zabrze (IChPW). The scheme of the plant is presented in Figure 1. The aforementioned plant is a circulating fluidized bed reactor. The gasifying agents during the process were CO₂ and O₂. Process parameters are presented in Table 1. During the gasification in the pilot plant, coal does not fully convert to syngas – from some of it, combustible char forms. The installation and the gasification process were already described in detail in the work by Chmielniak et al. (Chmielniak et al. 2015).

Then, the analysis of the circulating fluidized bed combustion of coal from the Wieczorek mine and the combustion process of chars produced from this coal was carried out at the Częstochowa University of Technology. The coal combustion process was carried out at atmospheric pressure, at temperatures in the range of 750–900°C with a fuel stream of 3 to 7 kg/h. The combustion of chars was carried out for a fuel stream ranging from 2.5 to 3 kg/h and at a temperature of 750–830°C.

The coal, char, and ash samples were collected before and after the gasification and combustion processes, respectively. The discussed samples were ground to a grain size

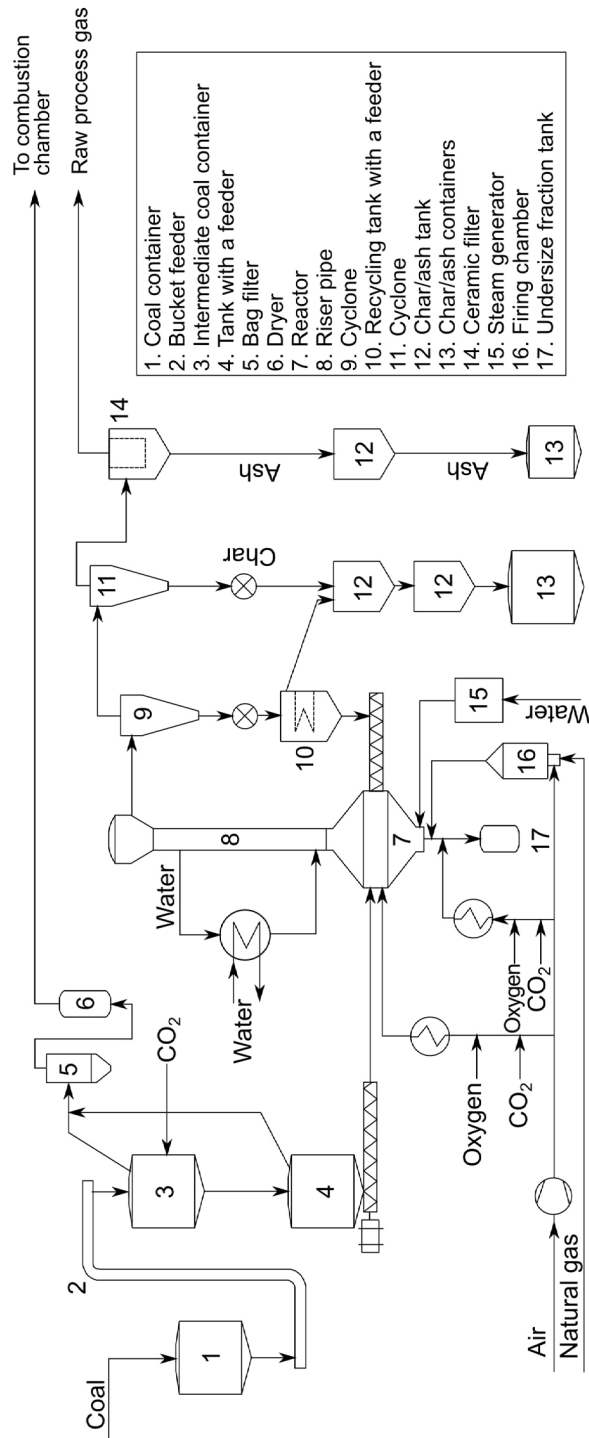


Fig. 1. Scheme of the pilot gasification plant in IChPW, after Chmielniak et al. (Chmielniak et al. 2015), modified

Rys. 1 Schemat pilotowej instalacji zgazowania w IChPW

Table 1. Parameters of technological tests and the content of combustible components in process gases (Chmielniak et al. 2015)

Tabela 1. Parametry technologiczne testu i skład gazów procesowych

Parameter	Value
Gasification temperature (°C)	794–1 091
Coal flow rate (kg/h)	125–186
Air flow rate (m ³ /h)	38–131
CO ₂ flow rate (m ³ /h)	0–61
O ₂ flow rate (m ³ /h)	0–24
Air/coal ratio (kg/kg)	2.2–11.4
	1.5–4.1
	6.5–17.0
Air/CO ₂ ratio (kg/kg)	794–1 091

of < 1 mm and used as material for polished sections, which were made according to the PN-ISO 7404-2:2005 standard. The petrographic examination was carried out in both reflected white and blue light with the use of a Zeiss Opton microscope and in accordance to the PN-ISO 7404-3:2001 standard. Maceral nomenclature for the vitrinite and inertinite maceral groups was made according to the guidelines of the International Committee for Coal and Organic Petrology (Stach et al. 1982; Taylor et al. 1998). Maceral nomenclature for the liptinite group was developed according to Pickel et al. (Pickel et al. 2017).

The classification scheme developed for combustion chars – Commission III Combustion Working Group of the International Committee for Coal and Organic Petrology (Lester et al. 2010) has also been used. The mentioned classification was adopted for gasification chars. The classification was based on the porosity and, to a lesser extent, on the percentage and structure of unburned organic matter. The identification of objects was carried out based on the evaluation of morphology. The assessed components included: porosity, the degree of conversion, and, in some cases, the shape of the cells and the thickness of their walls.

The petrographic classification of fly ashes and bottom ashes was based on Suárez-Ruiz et al. (Suárez-Ruiz et al. 2017).

The maceral group, char, and ash content analyses were carried out on 500 equally spaced points on the surface of the polished sections.

Also, a proximate and ultimate analysis of coal and char was performed at the Institute for the Chemical Processing of Coal in Zabrze, in accordance with ISO standards. The proximate analysis included: moisture content, ash content, volatile matter content, and the Gross Calorific Value. The proximate analysis included carbon, hydrogen, nitrogen, and total sulphur content.

2. Proximate, ultimate, and petrographic analysis of bituminous coal and char

The results of proximate and ultimate analysis of bituminous coal from Wieczorek mine and the char produced during its gasification are presented in Table 2.

Table 2. Results of the proximate and ultimate analyses of coal from the Wieczorek mine and the char from its gasification

Tabela 2. Wyniki analiz technologicznych i chemicznych węgla z kopalni Wieczorek oraz karbonizatu z jego zgazowania

Parameter	Symbol	Unit	Value – coal	Value – char
Moisture	M ^a	%	3.60	0.60
Ash	A ^{db}	%	8.20	38.10
Volatile matter	V ^{daf}	%	35.30	2.20
Gross calorific value	GCV ^{daf}	MJ/kg	33.26	33.961
Carbon content	C _t ^{daf}	%	81.00	96.42
Hydrogen content	H _t ^{daf}	%	4.50	1.14
Nitrogen content	N ^{daf}	%	1.20	1.67
Total sulphur	S _t ^d	%	0.42	1.09

In the case of bituminous coal, the petrographic composition was dominated by macerals of the vitrinite group (54.8% vol.) followed by macerals of the inertinite group (23.4%). The liptinite group constituted 16.2% of the examined coal volume, while the mineral matter – 5.6% (Table 3).

The most commonly observed maceral of the vitrinite group was collodetrinite (35.6% of the whole sample volume), followed by collotelinite (17.0%) (Fig. 2F). Collodetrinite was commonly observed with collotelinite, semifusinite, fusinite, inertodetrinite, and sporinite (Fig. 2A, B). Telinite, gelinite, and corpogelinite (Fig. 2D) were less commonly observed, while there was no vitrodetrinite in the examined coal.

In the case of macerals of the inertinite group, the dominant share of semifusinite (8.5%), followed by fusinite (6.1%), has been confirmed (Fig. 2E). Inertodetrinite, macrinite, micrinite, and funginite were also observed – they usually occurred within collodetrinite.

The most common maceral of the liptinite group was sporinite (14.6% of the entire sample volume), represented mainly by microsporinite (12.2%), which frequently formed large aggregations within collodetrinite. Macrosporinite was less common (2.4%). Resinite (Fig. 2C) and cutinite were rarely observed.

Table 3. Petrographic composition of bituminous coal from the Wieczorek coal mine

Tabela 3. Skład petrograficzny węgla kamiennego z kopalni Wieczorek

Group	Group content (%)	Maceral/inorganic component	Maceral/inorganic component content (%)
Vitrinite	54.8	Telinite	1.1
		Collotelinite	17.0
		Collodetrinite	35.5
		Vitrodetrinite	0.0
		Corpogelinite	0.3
		Gelinite	1.1
Inertinite	23.4	Fusinite	6.1
		Semifusinite	8.5
		Funginite	0.5
		Macrinite	2.9
		Micrinite	1.9
		Secretinite	0.0
		Inertodetrinite	3.4
Liptinite	16.2	Macrosporinite	2.4
		Microsporinite	12.2
		Cutinite	0.3
		Resinite	1.3
		Liptodetrinite	0.0
Mineral matter	5.6	Sulfides	0.5
		Carbonates	0.0
		Clay minerals	5.0

The mineral matter was represented by clay minerals (5.0% of the sample volume) and sulfides (0.5%).

The composition of the char sample was dominated by inertoids (41.2% of the whole sample volume) (Fig. 3B, C) and solid/fusinoid-type chars (11.3%) (Fig. 3E, F). The solids and fusinoids were represented by altered macerals of the vitrinite group (mostly collodetrinite) and semifusinite and fusinite relics. Mixed dense and mixed porous chars constituted 10.9% and 13.5% of the sample volume (Figs. 3A, C, and D). The strongly altered chars were represented mostly by the crassinetwork type (8.7%) (Fig. 3A); tenuinetwork, crassisphere, and

tenuisphere types were less commonly observed – they accounted for 1.6%, 4.8%, and 2.9%, respectively (Table 4).

In the examined sample, honeycomb structures were observed. Depending on their porosity, they were classified as mixed porous or crassinetwork types.

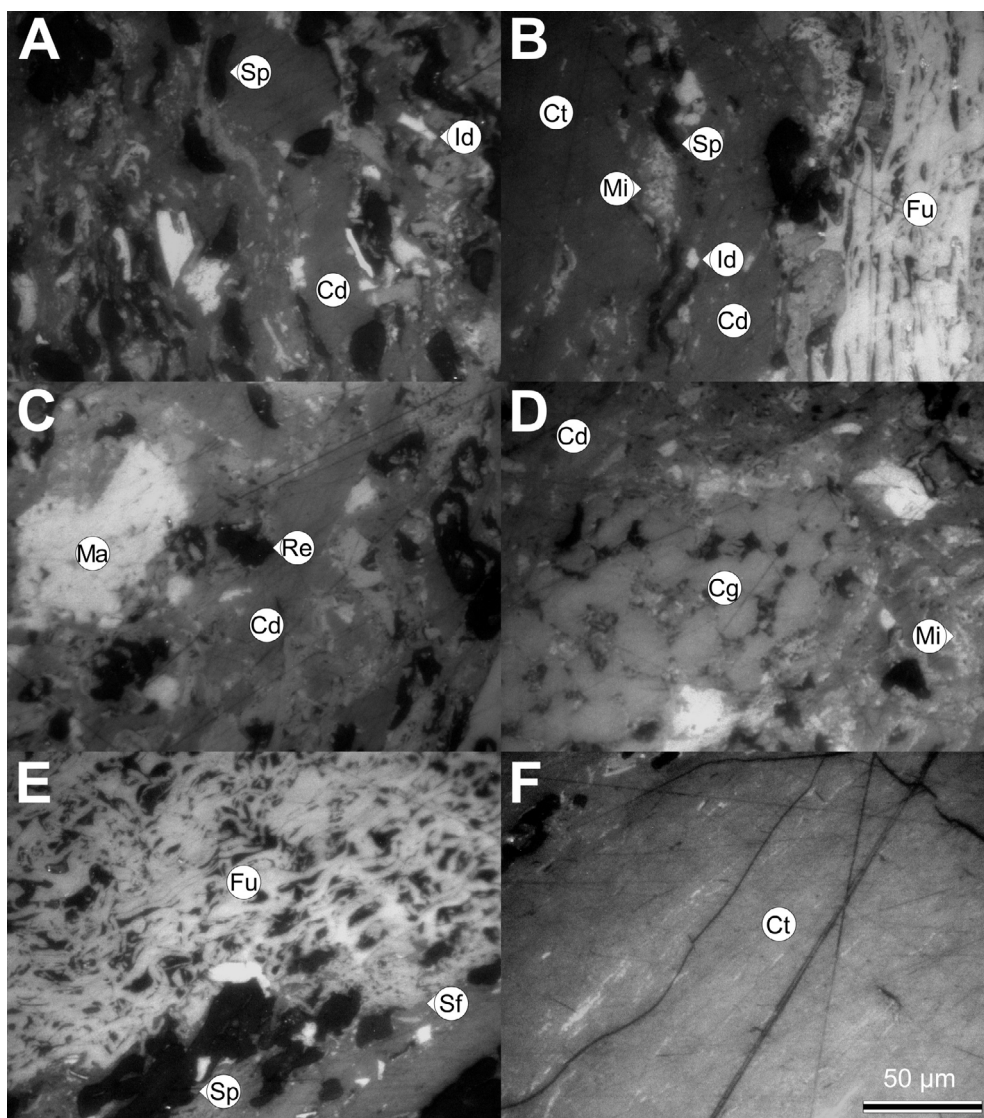


Fig. 2. Micrographs of bituminous coal from the Wieczorek coal mine
Cd – collodetrinite, Ct – collotelinite, Cg – corpogelinite, Fu – fusinite, Sf – semifusinite,
Id – inertodetrinite, Mi – micrinite, Ma – macrinite, Sp – sporinite, Re – resinite

Rys. 2. Mikrofotografie węgla kamiennego z kopalni Wieczorek

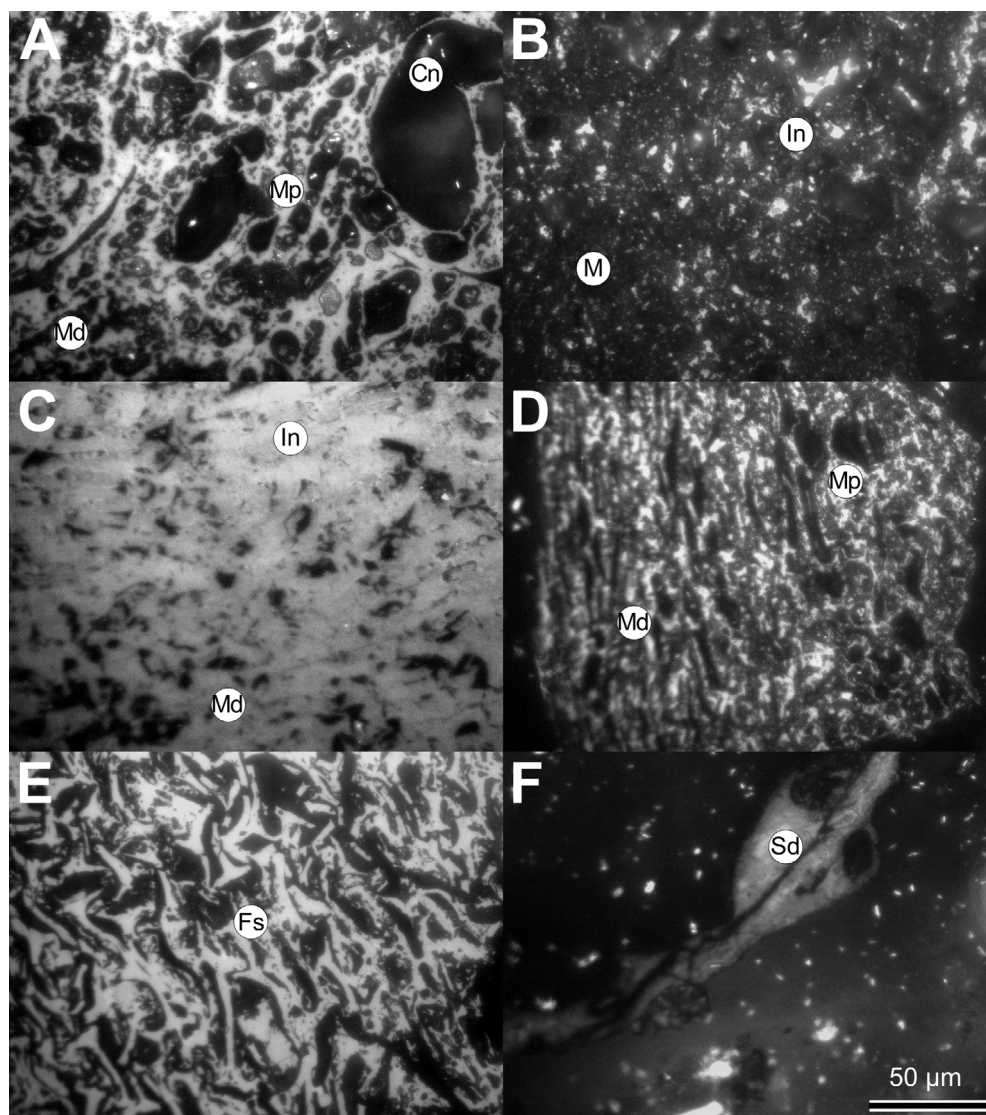


Fig. 3. Char micrographs
In – inertoid, Sd – solid, Fs – fusinoid, Md – mixed dense, Mp – mixed porous,
Cn – crassinetwork, M – mineroid

Rys. 3. Mikrofotografie karbonizatu

Table 4. Petrographic composition of the char

Tabela 4. Skład petrograficzny karbonizatu

Char type	Content (%)
Mineroid	5.1
Solid/fusinoid	11.3
Inertoid	41.2
Mixed dense	10.9
Mixed porous	13.5
Crassinetwork	8.7
Tenuinetwork	1.6
Crassisphere	4.8
Tenuisphere	2.9

3. Petrography of bottom ash from the combustion of bituminous coal and char

The bottom ash sample was dominated by mineroid-type chars (82.5% of the ash volume) (Fig. 4A, B, C, and D), followed by inertoid chars (7.1%) (Fig. 4B, C, and D). Solid and

Table 5. Petrographic composition of bottom ashes from the combustion of bituminous coal and char

Tabela 5. Skład petrograficzny popiołów dennych ze spalania węgla kamiennego i karbonizatu

Bottom ash from the combustion of bituminous coal		Bottom ash from the combustion of char	
char type	content (%)	char type	content (%)
Mineroid	82.5	Mineroid	81.9
Solid/fusinoid	1.6	Solid/fusinoid	3.3
Inertoid	7.1	Inertoid	5.5
Mixed dense	4.5	Mixed dense	4.1
Mixed porous	3.2	Mixed porous	4.1
Crassinetwork	1.0	Crassinetwork	1.1
Tenuinetwork	0.0	Tenuinetwork	0.0
Crassisphere	0.0	Crassisphere	0.0
Tenuisphere	0.0	Tenuisphere	0.0

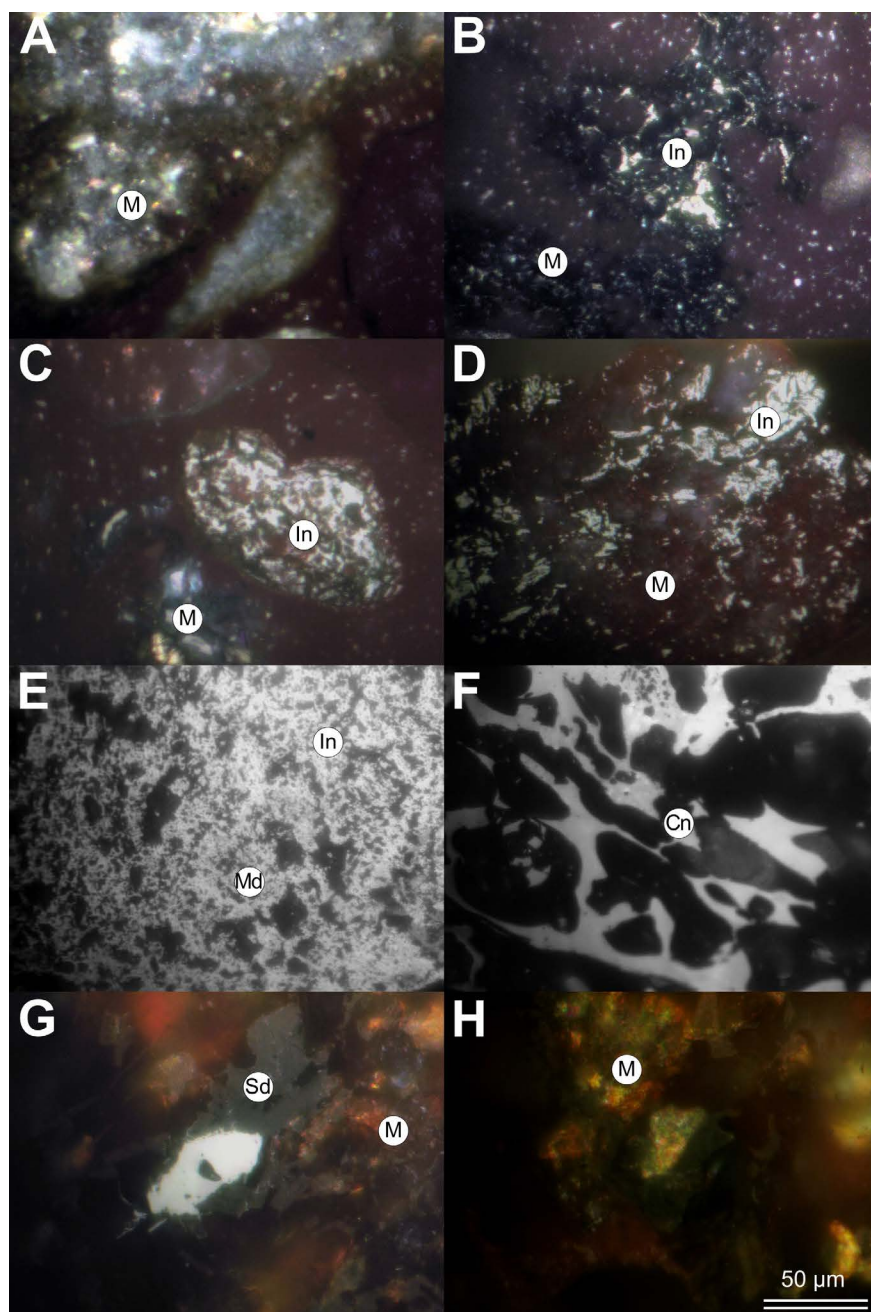


Fig. 4. Micrographs of bottom ash from coal combustion (A, B, C, and D) and from char combustion (E, F, G, and H)
In – inertoid, Sd – solid, Fs – fusinoid, Md – mixed dense, Cn – crassinetwork, M – mineroid

Rys. 4. Mikrofotografie popiołów dennych ze spalania węgla (A, B, C i D) oraz ze spalania karbonizatu (E, F, G i H)

fusinoid chars (1.6%) were also reported. Mixed dense and mixed porous chars accounted for 4.5% and 3.2%, respectively. The strongly altered chars were represented only by the crass-network type (Table 5). The bottom ash from the combustion of char was composed mostly of mineroid-type chars accounting for 81.9% of the entire sample volume (Fig. 4G, H). The second most common char type was inertoid (5.5%) (Fig. 3E), followed by mixed dense (Fig. 4E) and mixed porous chars (4.1% each). Solid-type (3.3%) (Fig. 4G) and crassnetwork (1.1%) (Fig. 3F) were also observed (Table 5).

4. Petrography of fly ash from bituminous coal and char combustion

The fly ash from coal combustion was very fine grained and contained no solid and fusinoid chars. The main components of the fly ash were the inertoid-type chars (42.1%) and mineroids (36.0%) (Fig. 5B, D). The inertoids were represented mostly by fine grains (Fig. 5A, B), which occasionally formed larger aggregates (Fig. 5C). The sample was also characterized by a relatively high tenuispheres content – 6.3% of the entire sample (Fig. 5A, B, C, and D).

The fly ash from char combustion was very fine grained and contained no solid and fusinoid chars. It was mostly composed of inertoid chars of different shapes and sizes (50.3%) (Fig. 5E, F, G and, H) and mineroids (44.0%) (Fig. 5E, H). Other char types – mostly crassisphere, crassnetwork (Fig. 5G), and tenuinetwork (Fig. 5E, G, and H) were less commonly observed.

Table 6. Petrographic composition of fly ashes from bituminous coal and char combustion

Tabela 6. Skład petrograficzny popiołów lotnych ze spalania węgla kamiennego i karbonizatu

Fly ash from bituminous coal combustion		Fly ash from char combustion	
char type	content (%)	char type	content (%)
Mineroid	36.0	Mineroid	44.0
Solid/fusinoid	0.0	Solid/fusinoid	0.0
Inertoid	42.1	Inertoid	50.3
Mixed dense	2.7	Mixed dense	0.9
Mixed porous	4.0	Mixed porous	0.7
Crassnetwork	2.7	Crassnetwork	1.1
Tenuinetwork	2.2	Tenuinetwork	0.2
Crassisphere	4.0	Crassisphere	1.6
Tenuisphere	6.3	Tenuisphere	1.1

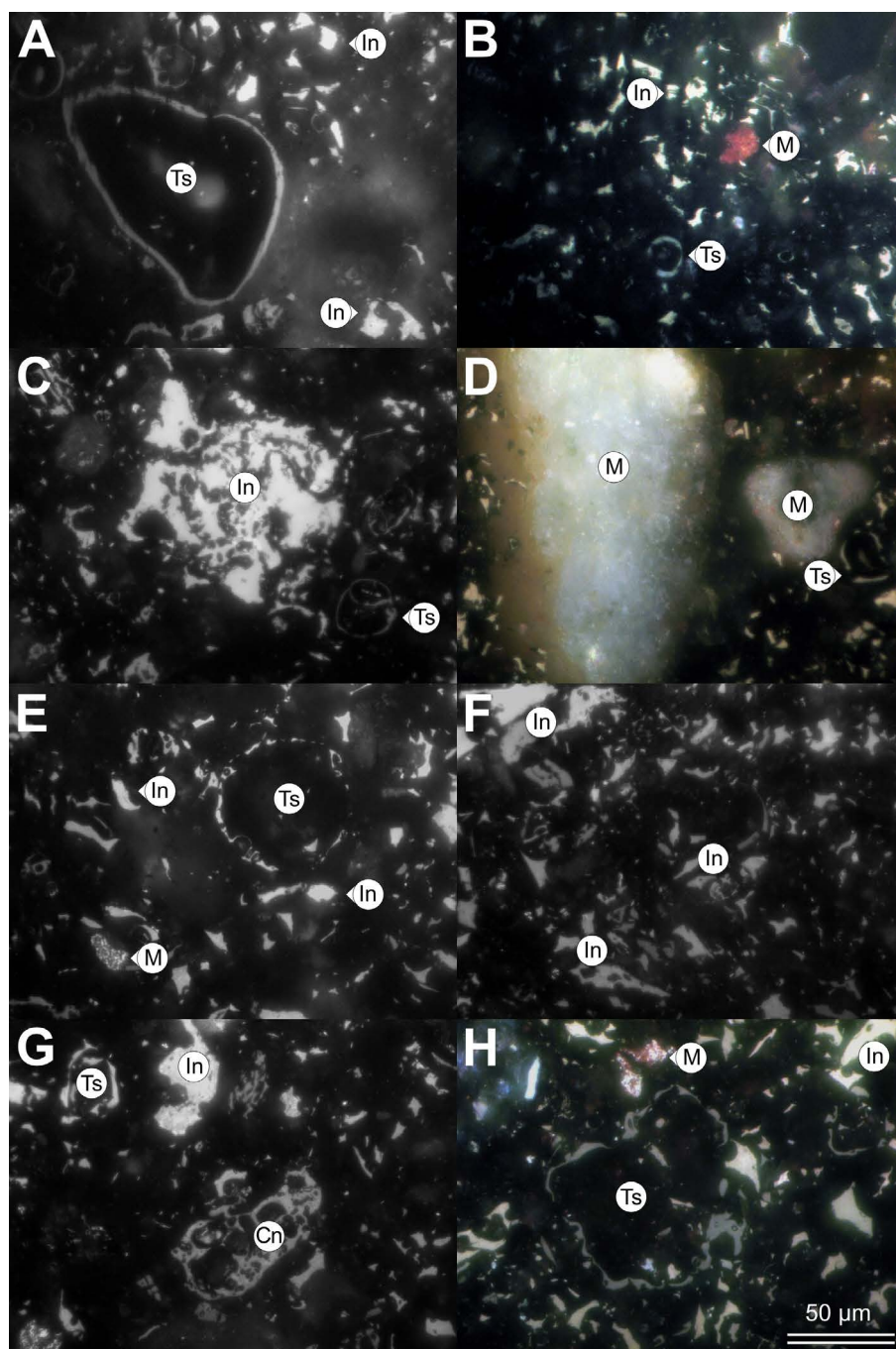


Fig. 5. Micrographs of fly ash from coal combustion (A, B, C, and D) and char combustion (E, F, G, and H)
In – inertoid, Cn – crassinetwork, Ts – tenuisphere, M – mineroid

Rys. 5. Mikrofotografie popiołów lotnych ze spalania węgla (A, B, C i D)

Both fly ash samples have a less variable composition and lower mineroid content than the bottom ashes (Table 6).

5. Results discussion

The examined coal petrographic composition, mainly vitrinite content exceeding 50%, and also the coal's chemical composition – high carbon content and low sulphur content – make it suitable for fluidized bed gasification process. The results of the experimental gasification and the gasification char properties support this observation:

- ◆ the carbon conversion during the process, calculated with the use of formula by Ammosov et al. (Ammosov et al. 1959) was rather high – 74.4%,
- ◆ the char morphologies show strong connection to the parent coal maceral composition, and also are typical for the gasification in the given temperatures (as explained below).

The gasification char composition is strongly dominated by inertoid-type chars. As vitrinite-group macerals were the most abundant in the parent coal, it can be suggested that vitrinite-group macerals are more prone to transformation into inertoid-type chars during the gasification experiment. The limited transformation of vitrinite and a relatively low content of porous chars (crassinetwork, crassisphere, tenuinetwork, and tenuisphere) is probably related to lower temperatures during char formation; Everson et al. observed (Everson et al. 2008) that vitrinite does not change its morphology during carbonization in temperatures of about 700°C while its reflectance increases. In the case of low-rank coals, ulminite is the maceral which most probably transforms into inertoids (Bielowicz 2016), which may suggest that in the case of medium-rank coals collotelinite is the maceral which takes the form of inertoids during fluidized bed gasification.

A relatively high content of solid/fusoid and mixed-type chars is probably related to the inertinite content in the parent coal. The correlation between high inertinite and inertinite-rich microlithotypes content and the increase of mixed-type and different types of dense chars was observed by Oboirien and Oboirien et al. (Oboirien 2011; Oboirien et al. 2012), who examined chars from the fluidized bed gasification of South African bituminous coals, containing 12, 59, and 71% of inertinite group macerals. The coal, discussed in this article contained 23.4% of inertinite group macerals; those macerals were frequently observed within collodetrinite, which may have caused the formation of mixed-type chars during gasification.

Compared to the char from the similar experiment, performed on coal from the Janina mine (Bielowicz 2019), the char described in this work contains significantly less crassinetwork and tenuinetwork morphotypes, and a higher content of inertoid and solid/fusoid chars. Those differences are probably connected to the difference in the petrographic composition and coal rank. The lesser content of cellular telinite in coal from Wieczorek mine leads to a lower content of network-type char morphotypes. Also, the coal from the

Wieczorek mine has a higher rank than the coal from the Janina mine – with the rank increase, the coal reactivity decreases (Speight 2014), which is probably the reason for a higher inertoid and solid content.

Inertoid chars were the most abundant in the bottom and fly ash from bituminous coal combustion. The temperatures during combustion were in the range of 750–900°C; however, the inertoid-dominated char composition suggests that low-temperature (around 750°C) conditions had the largest impact on char morphology. Aforementioned work by Everson et al. (Everson et al. 2008) has shown that vitrinite forms porous chars in temperatures around 900°C. This observation is also supported by experiments performed by Rosenberg et al. (Rosenberg et al. 1996) – chars from high-vitrinite coal combustion in temperatures around 800–900°C generally have a tenuisphere or crassisphere morphology, while the inertoid content does not exceed 15% (mineral matter-free basis).

The bottom and fly ashes from char combustion are significantly less abundant in mixed, network, and spherical chars, than in the parent char. This suggests the burnout of chars with those morphologies during char combustion. The high content of inertoids may suggest that those chars do not undergo further changes during combustion in given conditions. The drop in solid content is probably related to the transformation of remaining vitrinite relics to inertoids – the temperature during char combustion was between 751 and 836°, which favors the formation of inertoids.

Bottom ashes from the combustion of bituminous coal and char have a similar petrographic composition. More differences were observed in the composition of fly ash samples. When it comes to the fly ash from char combustion, the crassi- and tenui-types were significantly less common, while the mineroid content was higher. This suggests a better burnout of carbon during char combustion.

Generally, chars with a higher porosity and fine-grained inertoids are more prone to accumulation in fly ash. Bottom ash composition is dominated by mineral matter and larger char particles. Light chars are carried away in fly ash, while the heavier particles fall down to form the bottom ash.

Conclusions

The conducted experiment has shown that coal from the Wieczorek mine is suitable for fluidized bed gasification in a carbon dioxide atmosphere. The inertinite group accounts for almost 23%, of which around 8% is semifusinite. The share of non-reactive macerals is relatively high (around 20%). When it comes to the petrographic composition of char, only a small part did not react and took the form of solid/fusinoid. The char, dominated by inertoids, was formed during the fluidized bed gasification of bituminous coal from the Wieczorek mine. There are very few porous particles. Such a composition is associated with a fairly low temperature of the gasification process and the petrographic composition of the gasified coal. The combustion of bituminous coal and gasification char led to the formation

of bottom and fly ashes with a similar petrographic composition. However, it has been found that fly ash from the combustion of char contained more mineroids and less porous chars, which suggest a better burnout. This may, in turn, suggest that the combustion of char remaining after gasification may be economically justified.

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**PETROGRAPHIC COMPOSITION OF CHAR FROM THE GASIFICATION
OF COAL FROM THE WIECZOREK MINE AFTER COMBUSTION**

Key words

coal gasification, char petrography, fluidized bed gasification,
bottom and fly ash, fluidized bed combustion

Abstract

The aim of the paper is the petrographic characterization of coal from the Wieczorek mine and the residues after its gasification. The coal was subjected to gasification in a fluidized bed reactor at a temperature of about 900°C and in an atmosphere of oxygen and CO₂. The petrographic, proximate, and ultimate analysis of coal and char was performed. The petrographic composition of bituminous coal is dominated by macerals of the vitrinite group (55% by volume); macerals of inertinite and liptinite groups account for 23% and 16.0%, respectively. In the examined char, the dominant component is inertoid (41% vol.). Mixed dense and mixed porous account for 10.9% and 13.5% vol., respectively. In addition, the examined char also contained unreacted particles such as fusinoids, solids (11.3% vol.), and mineroids (5.1% vol.). The char contains around 65% vol. of low porosity components, which indicates a low degree of carbon conversion and is associated with a low gasification temperature. The char was burned and the resulting bottom and fly ashes were subjected to petrographic analysis. Their composition was compared with the composition of ashes from the combustion of bituminous coal from the Wieczorek mine. Bottom ashes resulting from the combustion of bituminous coal and char did not differ significantly in the petrographic composition. The dominant component was mineroid, which accounted for over 80% vol. When it comes to fly ash, a larger amount of particles with high porosity is observed in fly ash from bituminous coal combustion.

**SKŁAD PETROGRAFICZNY KARBONIZATU ZE ZGAZOWANIA WĘGLA
Z KOPALNI WIECZOREK I POPIOLÓW PO JEGO SPALENIU**

Słowa kluczowe

zgazowanie węgla, petrografia karbonizatu, zgazowanie w reaktorze fluidalnym,
popiół denny i lotny, spalanie w złożu fluidalnym

Streszczenie

Celem postawionym w poniższej pracy była charakterystyka petrograficzna węgla z kopalni Wieczorek oraz pozostałości po jego zgazowaniu. Węgiel poddano zgazowaniu w reaktorze fluidalnym w temperaturze około 900°C oraz w atmosferze tlenu i CO₂. Wykonano analizy petrograficzne, technologiczne i chemiczne węgla i karbonizatu. W składzie petrograficznym badanego węgla kamiennego przeważały macerały z grupy witrinitu (55% obj.), z kolei macerały z grupy inertynitu i liptynitów stanowiły odpowiednio 23 i 16,0% obj. W badanym karbonizacie dominującym składnikiem był

inertoid (41% obj.). Składniki zaliczane do typów *mixed dense* i *mixed porous* stanowiły odpowiednio 10,9 i 13,5% obj. Ponadto, badany karbonizat zawierał cząstki nieprzereagowane, takie jak *fusinoid* i *solid* (11,3% obj.) oraz *mineroid* (5,1% obj.). W karbonizacie znajdowało się około 65% obj. składników o niskiej porowatości, co wskazuje na niski stopień konwersji węgla i jest związane z niską temperaturą zgazowania. Następnie karbonizat poddano spalaniu, a powstałe popioły denne i lotne były przedmiotem badań petrograficznych. Porównano ich skład ze składem popiołów ze spalania węgla kamiennego z kopalni Wieczorek. Popioły denne ze spalania karbonizatu i węgla kamiennego nie różniły się znacząco pod względem składu petrograficznego. Ich dominującym składnikiem był *mineroid*, który stanowił ponad 80% obj. W przypadku popiołów lotnych, większa liczba cząstek o dużej porowatości była obserwowana w popiołach pochodzących ze spalania węgla kamiennego.