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New technology for hydrogen and geopolymer composites production from post-mining waste

Deliverable 2.2

Analysis results of testing raw mine wastes

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1. Introduction

The legal basis for the work entitled: “New technology for hydrogen and geopolymer composites production from post-mining waste” (acronym H2GEO) is a grant agreement No: 101112386 (RFCS-2022). H2GEO project consortium is composed of seven partners: Instytut Techniki Górniczej Komag (KOMAG), Poland; Główny Instytut Górnictwa (GIG), Poland; Institute of Construction and Architecture Slovak Academy of Sciences (USTARCH), Slovakia; Instytut Technologii Paliw i Energii (ITPE), Poland; VSB - Technical University of Ostrava (VSB), Czechia; Politechnika Wrocławska (PWR), Poland, and Haldex S.A. (HDX), Poland.

2. Goal of the work

Goal of the H2GEO project is the development of a comprehensive technology for the management of mine waste dumps. The main idea of the project is to use the separated mineral fractions and fly ash to produce geopolymer composites. It is planned to use CO_2 as a process carrier in the production of composites. Another important aspect of the project is to determine a possibility of obtaining hydrogen from the gasification of energy fractions of mine waste. High-quality raw materials for the production of geopolymers and hydrogen will be ensured by the use of an innovative mobile separator for processing of mine waste.

As part of Task 2.2 (titled “Testing the physical and chemical properties of mine wastes”), key physicochemical and mechanical parameters of selected mining waste form three heaps were determined, which can significantly affect the quality of the products obtained and the possibility of their use. Technical and elemental analyses of mining waste samples were carried out, the ash composition was analyzed, and the characteristic melting points for ash were determined. Analysis of trace elements classified as "critical" to the global economy was carried out, which are considered crucial to the development of new technologies and whose deficiency could have a tragic impact on world economies. Density and sieve analysis of mine waste samples regarding the share of a combustible substance were performed.

Microscopic evaluation using polarizing microscope of the landfill material was conducted to determine the deterioration of organic matter and to assist in the selection of materials. The samples were also analyzed using a scanning electron microscope (SEM-EDS) to determine the porosity and structure of the material.

Mechanical properties (resistance to fragmentation, abrasion resistance, absorbability, frost resistance) were tested to determine the quality of the aggregate and its suitability for specific applications. The properties of mine deposits were also analyzed in terms of TGA technique.

In addition, the biological activity of waste from the closed landfill was assessed in the context of planned reclamation activities. Scenarios and guidelines for the preparation of a mixture of bio-waste and plant seeds have been developed.

This report summarises the results of analyses of raw mining waste obtained by the consortium partners in the range of Task 2.2.

3. Raw material waste analyses

3.1. Raw mine waste materials for research

As part of the H2GEO project, in WP 2., samples from three mine heaps were analyzed: one Polish - Panewnicka heap (Haldex S.A.) and two from the Czech Republic: Jan Karel (Karvina) and Paskov D (Paskov) heap. Samples for testing from the Panewnicka heap were collected mechanically (using excavator). In the case of Jan Karel and Paskov heap samples were collected manually. Two samples of raw material were taken from each heap (from two different locations), which were then divided and delivered to the H2GEO Project Partners for testing.

Fig. 1. Map with sampling locations

The samples further in the KOMAG test report were named as follows:

- Haldex1 and Haldex2 (tests from the Panewnicka heap),
- Karvina1 and Karvina2 (tests from the Jan Karel heap),
- Paskov1 and Paskov2 (tests from the Paskov D heap).

Panewnicka heap, Haldex S.A. (Poland)

Mine waste from the Halemba and Bielszowice hard coal mines located in Ruda Śląska was stored on the Panewnicka heap.

Fig. 2. Sample collection site no.1 (Haldex1) - 50.216813N, 18.886416E (red dot)

Fig. 3. Sample collection site no.2 (Haldex2) - 50.215263N, 18.886639E (red dot)

Jan Karel Heap (Czech Republic)

Mine waste from the Jan Karel hard coal mine located in Karvina was stored on the heap. Mining in the above-mentioned mine ended in 2021.

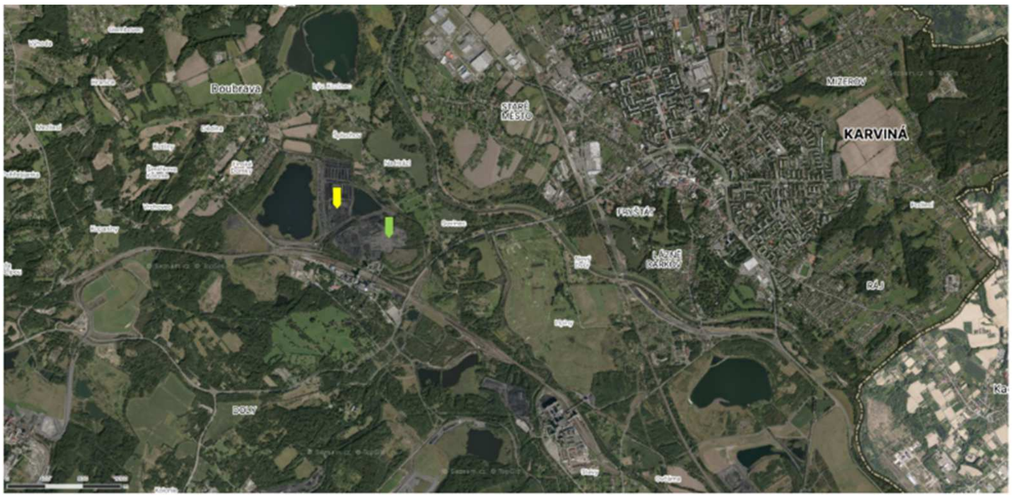


Fig. 4. Samples collection site
no.1 (Karvina1) - 49.8486647N, 18.5003019E (green mark)
no.2 (Karvina2) - 49.8509372N, 18.4916761E (yellow mark)

Paskov D Heap (Czech Republic)

Mine waste from the closed Paskov mining complex belonging to the OKD company was stored on the heap. It consisted of the Staríč, Chlebovice and Frenštát mines. Mining in the above-mentioned the mine ended in 2017.

Fig. 5. Samples collection site
no.1 (Paskov1) - 49.7462847N, 18.3024081E (yellow mark)
no.2 (Paskov2) - 49.7427194N, 18.3003856E (blue mark)

3.2. Proximate and ultimate analysis of raw material waste

Elemental and technical analysis of mining waste samples together with the analysis of ash composition, critical elements, characteristic temperatures for ash were carried out in Laboratory of Analytical Chemistry in Institute of Energy and Fuel Processing Technology in Zabrze (ITPE).

Moisture, volatile matter and ash content in analytical mine waste dumps samples were determined by use fully automatic thermogravimetric analyzer TGA-701, Leco. The ultimate analysis of the samples (carbon, hydrogen, nitrogen and sulphur content) was carried out using the Leco CHNS 628 and CS 632 analyzer, which uses IR and TC detection. Determination of calorific value of analytical samples was carried out in ITPE using automated calorimeters.

Results of proximate and ultimate analysis (including LHV analysis and chlorine content) of raw mine waste samples: Haldex1, Haldex2, Karvina1, Karvina2, Paskov1, and Paskov2 are presented in Table 1.

Table 1. Proximate and ultimate analysis of raw mine waste samples.

Parameter	Symb.	Unit	Haldex1	Haldex2	Karvina1	Karvina2	Paskov1	Paskov2
Total water content PN-ISO 589:2006 met.B1	M_{ar}	%	**3.0**	**2.3**	**1.7**	**2.0**	**1.2**	**1.2**
Water content (analit.) PN-ISO 11722:2009	M_{ad}	%	**1.4**	**1.1**	**1.3**	**1.5**	**1.0**	**1.1**
Ash content (analit.) PN-ISO 1171:2002	A_{ad}	%	**72.3**	**78.3**	**75.5**	**74.8**	**85.0**	**85.1**
Ash content (as received) PN-ISO 1171:2002	A_{ar}	%	**71.1**	**77.3**	**75.2**	**74.4**	**84.8**	**85.0**
Ash content (dry/waterless) PN-ISO 1171:2002	A_d	%	**73.3**	**79.2**	**76.5**	**75.9**	**85.9**	**86.0**
Volatile content (analit.) ISO 562:2010	V_{ad}	%	**13.04**	**11.43**	**11.92**	**12.09**	**7.20**	**7.33**
Volatile content (dry/waterless) ISO 562:2010	V_d	%	**13.23**	**11.56**	**12.08**	**12.27**	**7.27**	**7.41**
Volatile content (dry and ashless condition) ISO 562:2010	V_{daf}	%	**49.58**	**55.49**	**51.38**	**51.01**	**51.43**	**53.12**
Heat of combustion (analit.) PN-ISO 1928:2020-05	$Q_{v, gr, ad}$	kJ/kg	**6 630**	**4 440**	**5 170**	**5 330**	**2 640**	**2 630**
Heat of combustion (dry/waterless) PN-ISO 1928:2020-05	$Q_{v, gr, d}$	kJ/kg	**6 720**	**4 490**	**5 240**	**5 410**	**2 670**	**2 660**
Calorific value (analit.) PN-ISO 1928:2020-05	$Q_{p, net, ad}$	kJ/kg	**6 250**	**4 130**	**4 850**	**5 000**	**2 440**	**2 420**
Calorific value (as received)	$Q_{p, net, ar}$	kJ/kg	**6 110**	**4 050**	**4 820**	**4 960**	**2 430**	**2 420**

PN-ISO 1928:2020-05								
Total surphur content (analit.) ISO 19579:2006	$S_{T,\ ad}$	%	**0.49**	**0.49**	**0.20**	**0.22**	**0.13**	**0.07**
Total sulphur content (as received) ISO 19579:2006	$S_{T,\ ar}$	%	**0.48**	**0.48**	**0.20**	**0.22**	**0.13**	**0.07**
Total sulphur content (dry/waterless) ISO 19579:2006	$S_{T,\ d}$	%	**0.50**	**0.50**	**0.20**	**0.22**	**0.13**	**0.07**
Total carbon content (analit.) ISO 29541:2010	C_{ad}	%	**18.0**	**12.9**	**14.8**	**15.4**	**8.8**	**8.7**
Total carbon content (as received) ISO 29541:2010	C_{ar}	%	**17.7**	**12.7**	**14.7**	**15.3**	**8.8**	**8.7**
Total carbon content (dry/waterless) ISO 29541:2010	C_{d}	%	**18.3**	**13.0**	**15.0**	**15.6**	**8.9**	**8.8**
Total hydrogen content (analit.) ISO 29541:2010	H_{ad}	%	**1.75**	**1.42**	**1.51**	**1.54**	**0.94**	**0.95**
Total hydrogen content (dry/waterless) ISO 29541:2010	H_{d}	%	**1.62**	**1.31**	**1.38**	**1.39**	**0.84**	**0.84**
Nitrogen content (analit.) ISO 29541:2010	N_{ad}	%	**0.33**	**0.24**	**0.28**	**0.29**	**0.23**	**0.22**
Nitrogen content (dry/waterless) ISO 29541:2010	N_{d}	%	**0.33**	**0.24**	**0.28**	**0.29**	**0.23**	**0.22**
Oxygen content (analit. calculated) PN-ISO 1928:2020-05	O_d^a	%	**5.95**	**5.75**	**6.64**	**6.60**	**4.00**	**4.07**
Chlorine content (analit.) PN-G-04534:1999	Cl^a	%	**0.036**	**0.018**	**0.018**	**0.014**	**0.009**	**0.009**
Chlorine content (dry/waterless) PN-G-04534:1999[1)]	Cl^d	%	**0.037**	**0.018**	**0.018**	**0.014**	**0.009**	**0.009**
Phosphorus content (analit.) PN-82/G-04543[2)]	P^a	%	**0.027**	**0.031**	**0.032**	**0.037**	**0.064**	**0.074**
Phosphorus content (dry/waterless) PN-82/G-04543[2)]	P^d	%	**0.027**	**0.031**	**0.032**	**0.038**	**0.065**	**0.075**

1) Standard withdrawn and replaced by **PN-G-04534:2024-01**
2) Standard withdrawn

The presented results of the analysis of physicochemical properties presented above concern six types of materials obtained from mining heaps in Poland and the Czech Republic. These materials, called: Haldex1, Haldex2, Karvina1, Karvina2, Paskov1, Paskov2, can be classified into three main groups called Haldex, Karvina and Paskov due to their origin (i.e. place of collection) and in some respects similarity to selected parameters.

When describing the parameters chronologically, the first distinction can be noticed when taking into account the total moisture content (Table 1). Although all values of this parameter indicate that these are "dry" materials (water content below 5%), the water content in the Haldex1 sample (i.e. 3.0% by weight) differs from the value of this parameter in the case of other samples (range 1.2–2.3% by weight).

Another parameter that divides the analyzed samples into two main groups is the ash content. In order to refer to a certain base or "common denominator", the dry values were compared. The first group includes samples called Haldex1, Haldex2, Karvina1, Karvina2. In these samples, the ash content (in the range of 73.3–79.2% w/w) differs clearly, although not radically, from the ash content in Paskov1 and Paskov2 samples (i.e. 85.9–86.0% by weight). Due to the goals set in the project (including checking to what extent the analyzed materials can be used for the production of geopolymers), the ash content is a key parameter.

Other parameters that are important due to the possibility of waste heaps use are the content of volatile matter, carbon, hydrogen, and sulphur, as well as parameters indicating the calorific value (i.e. heat of combustion and calorific value in appropriate reference states). Haldex1 sample is characterized by the highest content of volatile matter in the dry state (i.e. 13.23% by weight), samples Haldex2, Karvina1 and Karvina2 show similar, although slightly lower values (i.e. in the range of 11.56–12.27% by weight). Samples: Paskov1 and Paskov2 are characterized by clearly lower values of volatile matter content in a dry state, which differ from the others (i.e. 7.27–7.41% by weight).

The same applies to the carbon content. The content of this element in the Haldex1 sample of 18.0 wt.% (in the analytical state) clearly differs from the carbon content in the Haldex2, Karvina1 and Karvina2 samples (i.e. in the range of 12.9–15.4 by weight) and even more from the Paskov1 and Paskov2 samples (8.7– 8.8% by weight). The hydrogen content for the Haldex1, Haldex2, Karvina1, Karvina2 samples is similar (1.42–1.75%) and slightly higher than that observed for the Paskov1 and Paskov2 samples (0.94–0.95%).

The sulphur content in the analytical state divides the tested samples "equally" into three groups with the highest content i.e. 0.48% by mass. (samples Haldex1 and Haldex2), moderate 0.20–0.22% (samples Karvina1 and Karvina2) and lowest 0.07–0.13% in the analytical state (Paskov1 and Paskov2 samples). It should be emphasized here that Haldex1 and Haldex2 samples may generate the most SO_2 during the roasting process.

The list of parameters influencing the calorific value (i.e. volatile matter content, carbon, hydrogen and sulphur content) is at this point a good introduction to the discussion of the obtained values of caloric parameters. The highest calorific value in the working condition (i.e. 6110 kJ/kg) was observed for the Haldex1 sample, which is consistent with the observed highest values of volatile matter, carbon, hydrogen and sulphur content for this sample. It should be emphasized that the calorific value below 5000 kJ/kg is considered to be the limit, below which it is impossible to maintain spontaneous combustion without external energy[1]. Therefore, the Haldex1 sample is slightly above this value, the Haldex2, Karvina1 and Karvina2 samples are borderline, and the Paskov1 and Paskov2 samples will require much more external energy to roast them in order to get rid of the flammable parts and obtain the pozzolanic properties. Even considering the heat of combustion, the values obtained for Paskov1 and Paskov2 samples do not exceed 2700 kJ/kg (dry state).

The chlorine content is the highest in the case of the Haldex1 sample (i.e. 0.036%) and the lowest (i.e. 0.009%) in the case of the Paskov1 and Paskov2 samples. It should be remembered that the presence of chlorine in the material may result in the presence of HCl in the gases leaving the roasting reactor, but the observed values are not high.

The nitrogen content in the tested samples is at a very low level. The highest value was recorded for the Haldex1 sample – 0.33%, and the lowest for Paskov2 sample - 0.22% in the analytical state.

The phosphorus content in samples is very low, too. The highest value was registered for Paskov1 sample (0.74%) and the lowest for Haldex1 (0.027% in the analytical state).

3.3. Characteristic ash fusion temperatures of raw material waste

Determination of characteristic temperatures of fusibility of ash samples was carried out in ITPE using apparatus with PR-25/1750 furnace, ITR, up to 1650°C. It consist of tubular furnace equipped with linear temperature programming set, CCD camera with filters system and computer equipped with image analysis programs. Determination of characteristic temperatures of fusibility is based on image analysis of sample during its programmed thermal heating, in both oxidized and reducing atmosphere. The analysis were performed by ITPE.

Characteristic ash fusion temperatures of raw mine waste samples from Haldex, Karvina, and Paskov in oxidizing and reducing atmosphere are presented in Table 2.

[1] K. Głód, J.A. Lasek, K. Supernok, P. Pawłowski, R. Fryza, J. Zuwała, “Torrefaction as a way to increase the waste energy potential”, Energy, 2023, 285, 128606.

Table 2. Characteristic ash fusion temperatures of raw mine waste samples.

Parameter			Sym	Unit	Haldex 1	Haldex 2	Karvina 1	Karvina 2	Paskov 1	Paskov 2
Characteristic ash fusion temp. *PN-ISO 540:2001 (ISO 540:1995 IDT)*	Oxidizing atm.	deformation temp.	DT	°C	**1380**	**1390**	**1410**	**1430**	**1290**	**1290**
		ball temp.	ST	°C	**1500**	**1490**	**1520**	**1550**	**1380**	**1390**
		Hemisphere temp.	HT	°C	**1530**	**1520**	**1550**	**1570**	**1430**	**1430**
		Flowing temp.	FT	°C	**1550**	**1540**	**1570**	**1580**	**1460**	**1440**
	Reducing atm.	Deformation temp.	DT	°C	**1270**	**1270**	**1220**	**1340**	**1200**	**1220**
		Ball temp.	ST	°C	**1450**	**1420**	**1500**	**1480**	**1320**	**1280**
		Hemisphere temp.	HT	°C	**1480**	**1470**	**1530**	**1530**	**1380**	**1370**
		Flowing temp.	FT	°C	**1520**	**1510**	**1570**	**1560**	**1430**	**1430**

The characteristic ash fusion temperatures of raw mine waste samples presented in Table 2 give an insight into the behaviour of the mineral part (post-ash) in terms of its softening. The observed values (i.e. above 1250°C) are well above the predicted calcination temperature ranges (i.e. below 900°C). For this reason, the occurrence of unfavourable phenomena accompanying the transformation during the roasting process in a fluidized bed reactor (e.g. agglomeration) is not expected.

3.4. Composition of ash of raw material waste

Determination of the content of oxides in ash raw material waste samples such as: SiO_2, Al_2O_3, Fe_2O_3,CaO, MgO, P_2O_5, SO_3, Mn_3O_4, TiO_2, BaO, SrO, Na_2O, K_2O was carried out in ITPE using X-ray wave dispersive spectrometer ARL OPTIM'X, Thermo Scientific. Spectrometer is sequence analyzer equipped with 200 W X-ray tube Rh, two SC and FPC detectors, and goniometer provided with changer of three crystals: AX06, InSb, LiF200.

Chemical ash composition of mine waste dumps samples from Haldex, Karvina, Paskov is presented in Table 3.

Table 3. Chemical ash composition of mine waste dumps samples.

Parameter	Symbol	Unit	Haldex 1	Haldex 2	Karvina 1	Karvina 2	Paskov 1	Paskov 2
Ash composition [3] *Q/LCA/55/B:2022 (met. ICP-OES)*	SiO_2	%	**58.84**	**58.88**	**58.38**	**57.49**	**59.56**	**61.43**
	Al_2O_3	%	**24.91**	**23.84**	**25.63**	**26.41**	**20.38**	**20.61**
	Fe_2O_3	%	**5.06**	**5.81**	**4.56**	**4.44**	**6.17**	**6.32**
	CaO	%	**0.71**	**0.55**	**0.81**	**0.87**	**0.78**	**0.94**
	MgO	%	**1.92**	**1.77**	**1.61**	**1.51**	**1.79**	**1.98**
	Na_2O	%	**0.30**	**0.32**	**0.37**	**0.38**	**0.98**	**1.00**
	K_2O	%	**3.43**	**3.37**	**3.84**	**3.92**	**3.96**	**4.09**
	TiO_2	%	**1.02**	**0.99**	**1.00**	**1.00**	**0.81**	**0.84**
	Mn_3O_4	%	**0.06**	**0.07**	**0.08**	**0.06**	**0.09**	**0.10**
	P_2O_5	%	**0.09**	**0.09**	**0.10**	**0.11**	**0.17**	**0.20**
	SO_3	%	**0.53**	**0.37**	**0.42**	**0.42**	**0.29**	**0.27**
	BaO	%	**0.05**	**0.04**	**0.05**	**0.05**	**0.06**	**0.05**
	SrO	%	**0.01**	**0.01**	**0.02**	**0.02**	**0.01**	**0.01**

[3)] sample ashed at 815°C

Results of ash composition analysis (Table 3) indicated that all raw mine waste samples are silicon-aluminium rich materials, which clearly indicates the possibility of using them as a raw material in the geopolymerization process. The SiO_2 content is very similar in all samples (i.e. in the range of 59.38–61.43%), while the Al_2O_3 content shows some distinction between the Haldex and Karvina samples (values in the range of 23.84–26.61%), and Paskov samples (20.38–20.61%). Literature data showed that, coal gangue containing similar amounts of silica and alumina were prone to geopolymerization and successfully used in the production of geopolymer mortars[2] or fire-resistant materials[3] so it can assumed that they are suitable starting material for the fabrication of geopolymer filtration membranes as well.

The Si/Al molar ratio equalled 1.85, 1.93, 2.00, 2.10, 2.48 and 2.53 for Karvina2, Karvina1, Haldex1, Haldex2, Paskov1 and Paskov2, respectively. Differences in Si/Al molar ratio

[2] Huang G., Ji Y., Jun Li, Hou Z., Dong Z. "Improving strength of calcinated coal gangue geopolymer mortars via increasing calcium content", Construction and Building Materials, 2018, 166, 760-768.
[3] Sitarz M., Figiela B., Łach M., Korniejenko K., Mróz K., Castro-Gomes J., Hager I. „Mechanical Response of Geopolymer Foams to Heating—Managing Coal Gangue in Fire-Resistant Materials Technology.

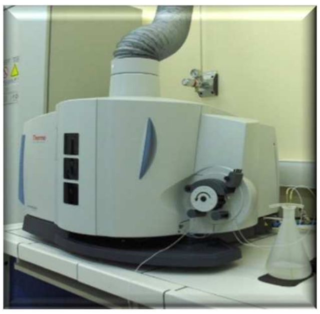
may affect the water absorption[4] of prepared geopolymers and translate into some differences in membranes transport properties.

In the context of geopolymerization of the tested materials, the calcium content is also important. All samples are characterized by relatively low calcium content. Among the analyzed materials, the highest content of CaO was observed for the Paskov2 sample (i.e. 0.94% by mass, 0.67% - converting into the content of the calcium element), and the lowest for the Haldex2 sample (i.e. 0.55% by mass, 0.39% - converting into the content of the Ca element). It should be emphasized that low starting calcium contents are advantageous taking into account the application of such materials for low-pressure filtration membranes production. Low content of Ca should have a positive effect on the acidic resistance and chemical stability of membranes that will be tested in the purification of acidic solutions (resulting from SO_4^{2-} ion presence) in Task.5.2.

Literature data showed that geopolymer obtained from materials characterized by high calcium content are generally more prone to acidic degradation due to the calcium "leaching". On the other hand, coal gangue geopolymers doped with calcium-containing slags exhibited improved compressive strength in comparison with pure coal gangue geopolymers. Also, data regarding acid mine drainage treatment using red-mud/Portland cement geopolymers suggest that under favourable pH conditions, leaching of calcium ions from geopolymer matrix may enhance heavy metals removal. Nevertheless, the effect of calcium presence/addition to geopolymer starting materials remains somewhat contentious since calcium leaching leads to geopolymer structure deterioration while "calcium overdosing" may be counterproductive and result in the decrease in the mechanical strength.

3.5. Trace elements analysis of raw material wastes

Trace analysis and ash composition was determined in ITPE using Emission Spectrometer with conductively coupled plasma iCAP 6500 Duo, Thermo Scientific (Fig. 6). The content of elements such as: As, Cd, Co, Mn, Cu, Cr, Ni, Pb, Eu, Ga, Be, Sm, Sc, W, Y, Yb Mo, Sb, Zn, V and Si, Al, Fe, Ca, Mg, P, S, Mn, Ti, Ba, Sr, Na, K was analysed.



[4] Mustofa, Pintowantoro S. "The effect of Si/Al ratio to compressive strength and water absorption of ferronickel slag-based geopolymer", The 2nd International Seminar on Science and Technology, 2016, 167-172.

Fig. 6 Apparatus for testing the content of trace elements

Table 4. Trace element analysis of raw material waste.

Research method		Symbol	Unit	Haldex 1	Haldex 2	Karvina 1	Karvina 2	Paskov 1	Paskov 2
Content of trace elements in a dry state Q/LCA/57/B:2022	Beryllium	Be^d	mg/kg	3.36	3.22	2.97	3.04	3.23	3.36
	Cobalt	Co^d	mg/kg	19.3	19.8	18.0	16.3	17.1	16.9
	Europium	Eu^d	mg/kg	0.426	0.453	0.654	0.647	1.12	1.15
	Gal	Ga^d	mg/kg	21.1	21.5	23.7	23.3	22.5	22.7
	Antimony	Sb^d	mg/kg	3.04	3.10	3.76	3.26	3.23	3.04
	Molybdenium	Mo^d	mg/kg	3.55	3.28	0.721	0.681	0.483	0.460
	Nickel	Ni^d	mg/kg	46.4	45.4	48.3	41.8	47.3	47.1
	Scandium	Sc^d	mg/kg	23.7	23.9	18.1	18.8	18.7	19.3
	Samarium	Sm^d	mg/kg	3.12	2.98	4.79	4.59	6.78	6.49
	Tungsten	W^d	mg/kg	3.20	2.84	2.60	3.10	4.21	4.04
	Yttrium	Y^d	mg/kg	5.16	5.03	1.14	1.02	1.64	1.72
	Ytterbium	Yb^d	mg/kg	1.01	0.988	0.295	0.284	0.464	0.504
	Lithium	Li^d	mg/kg	140	138	116	116	101	100
	Titanium*	Ti^d	mg/kg	4480	4720	4590	4540	4170	4320
	Magnesium*	Mg^d	mg/kg	850	844	742	691	928	1027
	Strontium*	Sr^d	mg/kg	90	90	110	110	50	50

*converted from % mass to mg/kg

The most valuable trace elements (and with the highest yield) in the tested samples were titanium (approx. 4200-4700 mg/kg of sample) and magnesium (700-1000 mg/kg of sample). Elements such as lithium, strontium, nickel, scandium and cobalt were identified in the samples in amounts not exceeding 110 mg/kg, and usually not more than several dozen mg of a given element per kg of mine waste sample.

Analyzing the results of laboratory tests, it can be concluded that the content of individual trace elements in all tested samples was similar. Samples Paskov1 and Paskov2 had significantly higher contents of europium, samarium, tungsten and magnesium compared to other samples of mine heaps. The Haldex1 and Haldex2 mine heaps contained much more molybdenum, yttrium, ytterbium and lithium than the others.

Based on literature, we can find data on the content of individual trace elements, including: beryllium: 4-5 mg/kg[5], cobalt: 2.3-5.8 mg/kg[5]; 2.5 mg/kg[6]; 32-442.01 mg/kg[7], europium: 6.50 mg/kg[8]; 1.3 mg/kg[9]; 0.09 mg/kg[10]; 0.3-1.6 mg/kg[11]; gal: 27.4-32.2 mg/kg[5]; 23.8 mg/kg[10]; antimony: 2.4-14.4 mg/kg[5]; 2.5 mg/kg[6]; 0.001-5650 mg/kg[12]; molybdenium: 2.2-3.9 mg/kg[5]; nickel: 19.1-27.2 mg/kg[5]; 35 mg/kg[6]; scandium: 12.4-15.5 mg/kg[5]; 1.90-24.2 mg/kg[7]; 20.96 mg/kg[8]; 14.4 mg/kg[9]; 41.9 mg/kg[11]; samarium: 1.29-26.24 mg/kg[7]; 13.54 mg/kg[8]; 4.9 mg/kg[9]; 0.29 mg/kg[10]; 5.5-8.2 mg/kg[11]; tungsten: 1.9-2.8 mg/kg[5]; yttrium: 6.3-12.4 mg/kg[5]; 7.87-34.02 mg/kg[7]; <1 mg/kg[8]; 34 mg/kg[9]; 1.7 mg/kg[10]; 18.6-34.2 mg/kg[11]; lithium: 174-324 mg/kg[5]; 177.9 mg/kg[10]; <1 mg/kg[7]; magnesium: 0.1-0.2 mg/kg[5]; 17 mg/kg[7]; strontium: 123-177 mg/kg[5].

By comparing the literature data with the trace element analysis results obtained for samples from the Haldex, Karvina and Paskov heaps, it can be concluded that the range of the content of the elements in the tested samples is within the range of their content in the studies of other scientists.

However, it should be noted that the content of trace elements in individual seams varies greatly, even within one area of mine heaps (the content of trace elements may vary by up to several hundred percent). For this reason, it is important to carefully examine the possibilities of obtaining trace elements potentially occurring in a given area. Each source of trace elements should be analyzed separately, as their content may often exceed the limits for economic extraction.

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3.6. Granulometric-qualitative and density-qualitative analyses of raw material waste

Within Task T.2.2, KOMAG carried out a granulometric-qualitative and density-qualitative analysis of the six samples obtained from three locations: Panewnicka heap (Haldex1 and Haldex2), Jan Karel heap (Karvina1, Karvina2), and Paskov D heap (Paskov1 and Paskov2). The studies include:

- sieve analysis,
- density analysis in heavy liquids,
- analysis of physicochemical properties (in the analytical state) in terms of determining: water content, ash content, sulphur content, heat of combustion (higher heating value, HHV) along with calculation of the net calorific value, NCV.

In sieve analysis of each material, the sieves with hole sizes 30; 10; 3; and 1 mm were used, obtaining grain classes >30 mm, 30-10 mm, 10-3 mm, 3-1 mm and 1-0 mm.

Then, the 30-10 mm and 10-3 mm grain classes were subjected to density analysis in heavy liquids (aqueous solution of zinc chloride) with densities of 1.5 and 1.8 g/cm^3, obtaining density fractions <1.5; 1.5-1.8 and >1.8 g/cm^3.

In each density fraction, water, ash, sulphur content and heat of combustion were determined, along with calculated calorific value. Additionally, the same was determined for all grain classes >30 mm, 3-1 mm and 1-0 mm. Based on the results, the average content of the parameters were calculated for the analysed grain classes and for the entire material. The "average" means the weighted arithmetic averages of the analysed materials.

All analyses and determinations were made in accordance with the following applicable standards:

- grain analysis according to PN-ISO 1953:1999,
- density analysis according to PN-G-04559:1997,
- determination of water content according to PN-ISO 589:2006,
- determination of ash content according to PN-ISO 1171:2002,
- determination of total sulphur content according to PN-G-04584:2001,
- determination of the heat of combustion according to PN-ISO 1928:2020-05.

Laboratory tests were carried out in accordance with the requirements of the PN-EN ISO/IEC 17025:2018-02 standard using the measuring equipment ensuring measurement consistency.

The research used supervised measurement equipment in the form of:

- SML 48/250 laboratory dryer,
- chamber furnace for determining ash content PM-6/1100A,
- SC 132 sulphur analyser,
- AC350 calorimeter,
- electronic laboratory scales B200B, WPT 15H2 and HR 120.

Additionally, a stand for density analysis and a laboratory screen with a set of sieves for grain analysis were used.

The results of sample analyses are presented in the following subsections, in a tabular form, with division for each sample into the results of granulometric-qualitative and density-qualitative analyses.

3.6.1.1. Results of granulometric-qualitative analyses in 30-0 mm class of Haldex1

In the grain classes obtained from the granulometric analysis of the sample, the moisture content, ash content, heat of combustion were determined and the calorific value was calculated (in the analytical state). The results of the analyses are presented in Table 5.

Table 5. Granulometric-qualitative composition

Grain class mm	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
>30	17.00	0.97	80.69	0.42	3218	2978
30-10	44.62	0.54	76.17	1.08	5616	5328
10-3	27.03	0.65	70.81	0.52	7408	7055
3-1	9.21	1.42	65.24	0.59	9021	8593
1-0	2.15	2.17	61.95	1.25	9955	9479
Sum	100.00					
Average		0.76	74.18	0.78	6 099	5 785

In the tested material, the largest share, equal to 44.62%, were grains in the class 30-10mm. In turn, the class 1-0 mm had the smallest share, which was 2.15%. Grains >30 mm had share in the sample equal to 17.00%.

The ash content was inversely proportional to the grain size and ranged from 80.69% (class>30mm) to 61.95% (class 1-0mm). As the ash content decreased, the calorific value increased from 2 978 kJ/kg to 9 479 kJ/kg. The average values of the above parameters were 74.18% and 5 785 kJ/kg. The average sulphur content in the tested sample was 0.78%, and the highest content was found in the 30-10 mm class, equal to 1.08%.

3.6.1.2. Results of density and quality analyses of 30-3 mm class of Haldex1

Tables below present the results of density and quality analyses of selected grain classes. Table 6 and Table 7 include the results of analyses of the 30-10 mm and 10-3 mm grain classes, and Table 8 contains the results for the combined 30-3 mm class.

Table 6. Density and quality composition of the material in 30-10 mm class

Density fraction g/cm³	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	7.02	0.96	11.34	0.75	30225	29167
1.5-1.8	6.36	0.99	37.83	1.16	19363	18617
>1.8	86.62	0.47	84.24	1.10	2612	2420
Sum	100.00					
Average		0.54	76.17	1.08	5 616	5 328

Table 7. Density and quality composition of the material in 10-3 mm class

Density fraction g/cm³	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	14.24	1.04	8.48	0.80	30840	29747
1.5-1.8	7.26	1.08	39.89	0.88	18139	17416
>1.8	78.50	0.54	84.98	0.44	2165	1981
Sum	100.00					
Average		0.65	70.81	0.52	7 408	7 055

Table 8. Density and quality composition of the material in 30-3 mm class

Density fraction g/cm³	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	9.74	0.99	10.26	0.77	30457	29386
1.5-1.8	6.70	1.02	38.61	0.38	18901	18164
>1.8	83.56	0.50	84.52	0.85	2443	2254
Sum	100.00					
Average		0.58	74.21	0.81	6 275	5 964

A very high share of combustible fractions was found in the tested samples. The shares of grains with a density <1.8 g/cm³ were 13.38% for the 30-10 mm class and 21.50% for the 10-

3 mm class. As a result, in the 30-3 mm class, the share of the above-mentioned grains was equal to 16.44%.

Both ash and sulphur contents were lower in the 10-3 mm class and amounted to 70.81% and 0.52%, respectively. For comparison, in the 30-10 mm class the above values were 76.17% and 1.08%. The lower ash content in the 10-3 mm class resulted in a more favourable, higher calorific value, equal to 7055 kJ/kg (in the analytical state), as opposed to the value obtained for the 30-10 mm grain class, which was equal to 5328 kJ/kg (in the analytical state).

3.6.2. Haldex2 sample results

3.6.2.1. Results of granulometric-qualitative analyses in 30-0 mm class of Haldex2 sample

In the grain classes obtained from the granulometric analysis of the sample, the moisture content, ash content, heat of combustion were determined and the calorific value was calculated (in the analytical state). The results of the analyses are presented in Table 9.

Table 9. Granulometric-qualitative composition

Grain class mm	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
>30	20.42	0.93	81.84	0.61	3061	2835
30-10	58.71	0.80	80.09	0.36	4155	3909
10-3	16.80	0.70	73.52	0.47	6181	5860
3-1	3.02	1.37	65.46	0.74	8769	8344
1-0	1.06	1.90	63.25	1.06	9733	9275
Sum	100.00					
Average		0.84	78.76	0.45	4470	4208

In the tested material, the largest share, exceeding half of the total material and equal to 58.71%, were grains in the 30-10 mm grain class. In turn, the 1-0 mm class had the smallest share, which was 1.06%. Grains >30 mm had a significant share in the sample and their share was 20.42%.

The ash content, as in the Haldex 2 sample, was inversely proportional to the grain size and ranged from 81.84% (class >30 mm) to 63.25% (class 1-0 mm). As the ash content decreased, the calorific value increased from 2835 kJ/kg to 9275 kJ/kg. The average values of the above parameters were 78.76% and 4208 kJ/kg. The average sulphur content in the tested sample was 0.45%, and the highest content was found in the 1-0 mm class, equal to 1.06%.

3.6.2.2. Results of density and quality analyses of 30-3 mm class of Haldex2 sample

Tables below present the results of density and quality analyses of selected grain classes. Table 10 and Table 11 include the results of analyses of the 30-10 mm and 10-3 mm grain classes, and Table 12 contains the results for the combined 30-3 mm class.

Table 10. Density and quality composition of the material in 30-10 mm class

Density fraction g/cm^3	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	5.61	1.03	9.72	0.86	30749	29671
1.5-1.8	4.41	1.01	41.79	0.77	18219	17519
>1.8	89.97	0.78	86.36	0.31	1805	1634
Sum	100.00					
Average		0.80	80.09	0.36	4155	3909

Table 11. Density and quality composition of the material in 10-3 mm class

Density fraction g/cm^3	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	11.11	1.23	8.49	0.84	30729	29634
1.5-1.8	5.97	1.12	38.21	0.88	18873	18130
>1.8	82.91	0.60	84.78	0.39	1977	1790
Sum	100.00					
Average		0.70	73.74	0.47	6181	5860

Table 12. Density and quality composition of the material in 30-3 mm class

Density fraction g/cm^3	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	6.84	1.07	9.45	0.86	30745	29662
1.5-1.8	4.76	1.03	40.99	0.79	18364	17655
>1.8	88.40	0.74	86.01	0.33	1843	1669
Sum	100.00					
Average		0.78	78.67	0.39	4606	4344

The analyses showed a significant share of combustible fractions in the tested samples. Share of grains with a density <1.8 g/cm^3 was 10.02% for the 30-10 mm class and 17.08% for the

10-3 mm class. As a result, in the 30-3 mm class, share of the above-mentioned grains was equal to 11.60%.

Ash content was lower in the 10-3 mm class and amounted to 73.74%. For comparison, in the 30-10 mm class the analysed parameter was 80.09. The sulphur content for classes 30-10 mm and 10-3 mm was 0.36% and 0.47%, respectively. Average calorific values or the above-mentioned classes were 3909 kJ/kg and 5860 kJ/kg, and for the combined 30-3 mm class it was equal to 4344 kJ/kg.

3.6.3. Karvina1 sample results

3.6.3.1. Results of granulometric-qualitative analyses in 30-0 mm class of Karvina1

In the grain classes obtained from the granulometric analysis of the sample, the moisture content, ash content, heat of combustion were determined and the calorific value was calculated (in the analytical state). The results of the analyses are presented in Table 13.

Table 13. Granulometric-qualitative composition

Grain class mm	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
>30	0.65	0.64	85.05	0.13	2585	2401
30-10	33.66	0.79	82.71	0.14	2969	2755
10-3	33.72	0.87	78.93	0.18	4582	4323
3-1	18.17	1.25	73.72	0.26	5276	4950
1-0	13.79	1.07	68.02	0.39	8036	7645
Sum	100.00					
Average		0.94	77.79	0.21	4629	4355

In the tested material, the largest shares were of grain classes 30-100 mm and 10-3 mm, which were approximately 33%. Traces of grains >30 mm were found, amounting to 0.65%.

As in the previous samples, the ash content was inversely proportional, and the calorific value was directly proportional to the grain size, and the average values of the above-mentioned parameters were 77.79% and 4355 kJ/kg. The most favourable parameters, regarding the share of combustible fractions, were recorded in the finest grains (1-0 mm), which had an ash content of 68.02% and a calorific value of 7645 kJ/kg. The sample had a very low sulphur content of 0.21%, and its highest content was found in the 1-0 mm class - 0.39%.

3.6.3.2. Results of density and quality analyses of 30-3 mm class of Karvina1

Tables below present the results of density and quality analyses of selected grain classes. Table 14 and Table 15 include the results of analyses of the 30-10 mm and 10-3 mm grain classes, and Table 16 contains the results for the combined 30-3 mm class.

Table 14. Density and quality composition of the material in 30-10 mm class

Density fraction	Share	Moisture content, W^a	Ash content, A^a	Sulfur content, S^a	Heat of combustion, Q_s^a	Calorific value, Q_i^a
g/cm^3	%	%	%	%	kJ/kg	kJ/kg
<1.5	1.05	1.20	13.18	0.74	29594	28554
1.5-1.8	5.32	1.34	42.55	0.55	17624	16929
>1.8	93.63	0.75	85.77	0.11	1838	1661
Sum	100.00					
Average		0.79	82.71	0.14	2969	2755

Table 15. Density and quality composition of the material in 10-3 mm class

Density fraction	Share	Moisture content, W^a	Ash content, A^a	Sulfur content, S^a	Heat of combustion, Q_s^a	Calorific value, Q_i^a
g/cm^3	%	%	%	%	kJ/kg	kJ/kg
<1.5	3.18	1.04	11.43	0.58	30312	29254
1.5-1.8	5.94	1.05	42.37	0.60	17988	17295
>1.8	90.88	0.85	83.68	0.14	2806	2603
Sum	100.00					
Average		0.87	78.93	0.18	4582	4323

Table 16. Density and quality composition of the material in 30-3 mm class

Density fraction	Share	Moisture content, W^a	Ash content, A^a	Sulfur content, S^a	Heat of combustion, Q_s^a	Calorific value, Q_i^a
g/cm^3	%	%	%	%	kJ/kg	kJ/kg
<1.5	2.12	1.12	12.30	0.66	29953	28904
1.5-1.8	5.63	1.19	42.46	0.58	17806	17112
>1.8	92.26	0.80	84.72	0.13	2322	2132
Sum	100.00					
Average		0.83	80.81	0.16	3779	3542

Lower shares of combustible fractions were found in the tested samples compared to samples from the Panewnicka heap. The shares of grains with a density of <1.8 g/cm^3 were for the classes 30-10 mm and 10-3 mm, 6.37% and 9.12%, respectively, and the result for the entire analysed material (30-3 mm) – 7.75%.

The samples had low sulphur content, which was 0.14% in the 30-10 mm class and 0.18% in the 10-3 mm class. The ash content was lower in finer grains, equal to 78.93%, and the corresponding calorific value was equal to 4323 kJ/kg. The above values for the 30-10 mm class were equal to 82.71% and 2755 kJ/kg.

3.6.4. Karvina2 sample results

3.6.4.1. Results of granulometric-qualitative analyses in 30-0 mm class of Karvina2

In the grain classes obtained from the granulometric analysis of the sample, the moisture content, ash content, heat of combustion were determined and the calorific value was calculated (in the analytical state). The results of the analyses are presented in Table 17.

Table 17. Granulometric-qualitative composition

Grain class mm	**Share** %	**Moisture content, W^a** %	**Ash content, A^a** %	**Sulfur content, S^a** %	**Heat of combustion, Q_s^a** kJ/kg	**Calorific value, Q_i^a** kJ/kg
>30	-	-	-	-	-	-
30-10	25.04	1.01	79.05	0.19	4063	3803
10-3	38.63	0.95	77.11	0.31	4682	4400
3-1	19.85	1.43	72.81	0.33	6653	6314
1-0	16.48	1.81	67.45	0.43	8297	7890
Sum	100.00					
Average		1.20	75.11	0.30	5514	5206

No grains >30 mm in size were found in the tested sample. The largest share, equal to 38.63%, were grains in the 10-3 mm class. The average ash content was 75.11% and ranged from 67.45% (class 1-0 mm) to 79.05% (class 30-10 mm). In turn, the average calorific value was 5206 kJ/kg and ranged from 7890 kJ/kg (class 1-0 mm) to 3803 kJ/kg (class 30-10 mm).

The sample had a low sulphur content, equal to 0.30%, and the highest content was recorded in the 1-0 mm class. The average ash content and calorific value were 75.11% and 5206 kJ/kg.

3.6.4.2. Results of density and quality analyses of 30-3 mm class of Karvina2

Tables below present the results of density and quality analyses of selected grain classes. Table 18 and Table 19 include the results of analyses of the 30-10 mm and 10-3 mm grain classes. Table 20 contains the results for the combined 30-3 mm class.

Table 18. Density and quality composition of the material in 30-10 mm class

Density fraction g/cm³	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	1.58	1.18	12.16	0.68	30341	29290
1.5-1.8	5.37	1.15	40.66	0.55	19285	18570
>1.8	93.04	1.00	82.41	0.16	2737	2517
Sum	100.00					
Average		1.01	79.05	0.19	4063	3803

Table 19. Density and quality composition of the material in 10-3 mm class

Density fraction g/cm³	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	3.37	1.49	9.80	0.64	30714	29631
1.5-1.8	6.27	1.29	42.24	0.68	17747	17049
>1.8	90.37	0.91	82.04	0.27	2806	2583
Sum	100.00					
Average		0.95	77.00	0.31	4682	4400

Table 20. Density and quality composition of the material in 30-3 mm class

Density fraction g/cm³	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
<1.5	2.66	1.37	10.73	0.66	30567	29497
1.5-1.8	5.92	1.23	41.62	0.63	18352	17647
>1.8	91.42	0.95	82.19	0.23	2779	2557
Sum	100.00					
Average		0.97	77.81	0.26	4441	4167

The sample had similar shares of the combustible fraction as in the Karvina1 sample. The shares of grains with a density <1.8 g/cm^3 were 6.95% (30-10 mm class) and 9.64% (class 10-3 mm). The sulphur content was low and amounted to abovementioned classes 0.31% and 0.26%.

The average ash content was lower than in the case of the Karvina 1 sample and amounted to 79.05%, 77.00% and 77.81%, respectively to the analysed classes. At the same time, the average calorific values were higher and were equal to 3803 kJ/kg, 4400 kJ/kg and 4167 kJ/kg.

3.6.5. Paskov1 sample results

3.6.5.1. Results of granulometric-qualitative analyses in 30-0 mm class of Paskov1

In the grain classes obtained from the granulometric analysis of the sample, the moisture content, ash content, heat of combustion were determined and the calorific value was calculated (in the analytical state). The results of the analyses are presented in Table 21.

Table 21. Granulometric-qualitative composition

Grain class mm	Share %	Moisture content, W^a %	Ash content, A^a %	Sulfur content, S^a %	Heat of combustion, Q_s^a kJ/kg	Calorific value, Q_i^a kJ/kg
>30	5.84	0.64	89.36	0.04	1295	1161
30-10	43.67	0.68	87.93	0.09	1890	1739
10-3	28.04	0.60	85.39	0.18	2843	2663
3-1	13.47	1.07	85.23	0.09	2497	2309
1-0	8.99	0.97	78.66	0.19	5441	5177
Sum	100.00					
Average		0.74	86.10	0.12	2524	2350

Grains 30-10 mm class, which amounted to 43.87% had the largest share in the tested material. Grains 10-3 mm also had a significant share in the sample, accounting to 28.04%. In turn, the class >30 mm had the smallest share equal to 5.84%.

Samples from the Paskov heap had the highest ash content and, as a result, the lowest calorific value of all analysed samples. The ash content was inversely proportional to the grain size and ranged from 89.36% (class >30 mm) to 78.66% (class 1-0 mm). As the ash content decreased, the calorific value increased from 1161 kJ/kg to 5177 kJ/kg. The average values of the above parameters were 86.10% and 2350 kJ/kg. The sulphur content in the tested sample was low, and its average content was 0.12%.

3.6.5.2. Results of density and quality analyses of 30-3 mm class of Paskov1

Tables below present the results of density and quality analyses of selected grain classes. Table 22 and Table 23 include the results of analyses of the 30-10 mm and 10-3 mm grain classes, and Table 24 contains the results for the combined 30-3 mm class.

Table 22. Density and quality composition of the material in 30-10 mm class

Density fraction g/cm³	**Share %**	**Moisture content, W^a %**	**Ash content, A^a %**	**Sulfur content, S^a %**	**Heat of combustion, Q_s^a kJ/kg**	**Calorific value, Q_i^a kJ/kg**
<1.5	0.33	1.66	13.39	0.56	32101	31058
1.5-1.8	1.15	0.70	42.58	0.41	18504	17818
>1.8	98.52	0.68	88.71	0.08	1594	1452
Sum	100.00					
Average		0.68	87.93	0.09	1890	1739

Table 23. Density and quality composition of the material in 10-3 mm class

Density fraction g/cm³	**Share %**	**Moisture content, W^a %**	**Ash content, A^a %**	**Sulfur content, S^a %**	**Heat of combustion, Q_s^a kJ/kg**	**Calorific value, Q_i^a kJ/kg**
<1.5	1.70	0.62	14.17	0.57	30256	29235
1.5-1.8	4.67	0.64	40.71	0.46	19169	18461
>1.8	93.62	0.60	88.92	0.16	1529	1391
Sum	100.00					
Average		0.60	85.39	0.18	2843	2663

Table 24. Density and quality composition of the material in 30-3 mm class

Density fraction g/cm³	**Share %**	**Moisture content, W^a %**	**Ash content, A^a %**	**Sulfur content, S^a %**	**Heat of combustion, Q_s^a kJ/kg**	**Calorific value, Q_i^a kJ/kg**
<1.5	0.87	1.25	13.70	0.56	31380	30345
1.5-1.8	2.53	0.68	41.85	0.43	18764	18069
>1.8	96.60	0.65	88.79	0.09	1569	1428
Sum	100.00					
Average		0.65	86.95	0.11	2262	2100

The analyses showed a small share of combustible fractions in the tested samples. In the 30-10 mm class, the share of grains with a density of <1.8 g/cm^3 was 1.48%, of which grains <1.5 g/cm^3 were only 0.33%. In the 10-3 mm class, the share of the above-mentioned grains was higher and amounted to 6.37% and 1.70%, respectively. Total share of combustible fraction grains (<1.8 g/cm^3) in the 30-2 mm class was 3.40%.

Lower share of combustible fraction grains in the 30-10 mm class resulted in higher ash content and lower calorific value (87.93%; 1739 kJ/kg) compared to the class 10-3 mm (85.39%; 2663 kJ/kg). Higher sulphur content, equal to 0.18%, was found in the 10-3 mm class.

3.6.6. Paskov2 sample results

3.6.6.1. Results of granulometric-qualitative analyses in 30-0 mm class of Paskov2

In the grain classes obtained from the granulometric analysis of the sample, the moisture content, ash content, heat of combustion were determined and the calorific value was calculated (in the analytical state). The results of the analyses are presented in Table 25.

Table 25. Granulometric-qualitative composition

Grain class	Share	Moisture content, W^a	Ash content, A^a	Sulfur content, S^a	Heat of combustion, Q_s^a	Calorific value, Q_i^a
mm	%	%	%	%	kJ/kg	kJ/kg
>30	5.90	0.64	89.62	0.03	1295	1164
30-10	33.75	0.65	87.58	0.05	1828	1673
10-3	29.66	0.90	84.61	0.10	3066	2873
3-1	18.51	1.23	85.30	0.08	2627	2438
1-0	12.18	0.88	80.17	0.16	4847	4602
Sum	100.00					
Average		0.86	85.37	0.08	2679	2497

In the tested material, grains in the 30-10 mm and 10-3 mm grain classes, had largest shares, which amounted to 33.75% and 29.66%, respectively. In turn, the class >30 mm had the smallest share, which was 5.90%.

Ash content, similarly to the Paskov 1 sample, was very high and ranged from 89.62% (class >30 mm) to 80.17% (class 1-0 mm). As the ash content decreased, the calorific value increased from 1164 kJ/kg to 4602 kJ/kg. The average values of the above parameters were 85.37% and 2497 kJ/kg. The average sulphur content in the tested sample was the lowest of all the analysed materials and amounted to 0.08%.

3.6.6.2. Results of density and quality analyses of 30-3 mm class of Paskov2

Tables below present the results of density and quality analyses of selected grain classes. Table 26 and Table 27 include the results of analyses of the 30-10 mm and 10-3 mm grain classes, and Table 28 contains the results for the combined 30-3 mm class.

Table 26. Density and quality composition of the material in 30-10 mm class

Density fraction **g/cm³**	**Share** **%**	**Moisture content, W^a** **%**	**Ash content, A^a** **%**	**Sulfur content, S^a** **%**	**Heat of combustion, Q_s^a** **kJ/kg**	**Calorific value, Q_i^a** **kJ/kg**
<1.5	0.29	0.78	15.97	0.60	29858	28857
1.5-1.8	1.36	0.71	44.74	0.41	18428	17767
>1.8	98.35	0.65	88.39	0.04	1515	1370
Sum	100.00					
Average		0.65	87.58	0.05	1828	1673

Table 27. Density and quality composition of the material in 10-3 mm class

Density fraction **g/cm³**	**Share** **%**	**Moisture content, W^a** **%**	**Ash content, A^a** **%**	**Sulfur content, S^a** **%**	**Heat of combustion, Q_s^a** **kJ/kg**	**Calorific value, Q_i^a** **kJ/kg**
<1.5	1.94	0.87	14.21	0.61	30342	29319
1.5-1.8	4.65	0.82	39.85	0.47	20136	19416
>1.8	93.41	0.90	88.30	0.07	1650	1501
Sum	100.00					
Average		0.90	84.61	0.10	3066	2873

Table 28. Density and quality composition of the material in 30-3 mm class

Density fraction **g/cm³**	**Share** **%**	**Moisture content, W^a** **%**	**Ash content, A^a** **%**	**Sulfur content, S^a** **%**	**Heat of combustion, Q_s^a** **kJ/kg**	**Calorific value, Q_i^a** **kJ/kg**
<1.5	1.06	0.82	15.15	0.60	30084	29073
1.5-1.8	2.90	0.76	42.45	0.44	19227	18538
>1.8	96.04	0.77	88.35	0.05	1578	1431
Sum	100.00					
Average		0.77	86.24	0.07	2393	2221

Small share of combustible fractions was found in the tested samples. In the 30-10 mm class, the share of grains with a density <1.8 g/cm^3 was 1.65%, of which the grains <1.5 g/cm^3 were only 0.29%. In the 10-3 mm class, the share of the above-mentioned grains was higher and amounted to 6.59% and 1.94%, respectively. Total share of combustible fraction (grains <1.8 g/cm^3) in the 30-2 mm class was 3.96%.

The average ash content in the analysed classes 30-10 mm, 10-3 mm and 30-3 mm were 87.58%, 84.61% and 86.24%, respectively. In turn, the calorific values for the above-mentioned classes were 1673 kJ/kg, 2873 kJ/kg and 2221 kJ/kg.

The highest sulphur content was found in fractions with a density of <1.5 g/cm^3, which were 0.60% and 0.61% for the 30-10 mm and 10-3 mm classes. The average value of the above parameter in the 30-3 mm class was low and amounted to 0.07%. This was due to the predominant share of grains in the material with a density > 1.8 g/cm^3, which had a very low sulphur content.

Conclusions

Analyses of mine waste samples collected from heaps located in Poland and the Czech Republic showed significant differences regarding the tested quality parameters. The diversity concerned both the grain composition of the supplied samples and the density composition, as well as the content of sulphur, ash and the related calorific value.

Share of the combustible fraction, i.e. the sum of coal grains (<1.5 g/cm^3 and hypertrophic grains (1.5-1.8 g/cm^3) was the parameter which differed significantly among the samples.

In the samples taken from the Panewnicka Dump (Haldex1, Haldex2), a very high share of the above-mentioned fractions was found, the share of which in the 30-3 mm class was: 16.44% and 11.60%, respectively. Samples from the Czech Republic contained smaller amount of combustible grains, the share of which in the 30-3 mm grain class did not exceed 10%. Samples taken from the Paskov heap had a particularly low content of these grains. In the 30-3 mm class, the fraction <1.8 g/cm^3 for the two tested samples was 3.40% and 3.96%, respectively.

Density analyses of grain classes 30-10 mm and 10-3 mm showed a higher share of combustible fractions (<1.8 g/cm^3) in the class consisting of smaller grains. The above relationship occurred for each analysed sample.

All samples had high ash content, the value of which in each case exceeded 70% and ranged from 75.18% (Haldex1) to 86.10% (Paskov1). The lowest ash content was found for each sample in the 1-0 mm grain class. As the ash content decreased, the calorific value increased and ranged from 2350 kJ/kg (Paskov1) to 5785 kJ/kg (Haldex1) in analytical state.

The analysed samples also differed in sulphur content, larger amounts of which (0.78%; 0.45%) were found in the Haldex samples. Its lower content was found was in the samples from the Jan Karel heap (0.21%; 0.30%) and from the Paskov heap, in which the sulphur content did not exceed 0.12%.

Summarizing, the raw material can be divided into a two main parts, i.e. combustible matter rich and mineral matter rich. Within the first group, the materials of lower size and lower density are observed. It is interesting that the separation methods can base on the size and density analysis. Within the first mentioned property (i.e. differences in size distribution) the following methods can be taken into account: sieving, method using inertial and centrifugal forces. Within the second property (i.e. differences in density) the methods basing on Archimedes' law can be considered.

3.7. Thermal analysis of raw mine waste

The averaged samples of raw mining waste from Haldex, Paskov, and Karvina were subjected to thermal analysis in USTARCH. Samples were dried at 105°C for 1 hour to remove the humidity. Then, the samples were calcinated at 600°C, 700°C, and 800°C to follow the weight loss due to the oxidation of coal. The procedure was used to determine the burned (coal) and unburned parts (ash). The thermal analysis results are presented in Table 29.

Table 29. Results of thermal analysis of raw waste

Sample	Weight loss at 600°C	Weight loss at 700°C	Weight loss at 800°C
Haldex	30.1	34.9	35.4
Karvina	23.8	24.1	24.3
Paskov	20.1	20.4	20.6

Considering the results depicted in table above, it is evident that the differences in the weight loss between 700 and 800°C is negligible. The weight loss of 35.4, 24.3, and 20.6 % represents the content of solid matter (fly ash) that will be considered for geopolymer preparation. The content of solid matters is not the same, but the amount is enough to deal with. The content of combustible part in Haldex - 64.6%, Paskov - 79.4%, and Karvina - 75.7 % represents the coal in the mining wastes.

This part will be deepened in WP4 („Developme□t of thermo□hemi□□l methods of m□□□geme□t of e□ergy fr□□tio□ of w□ste from jig be□efi□i□tio□ pro□ess to hydroge□ ge□er□tio□").

Thermogravimetric analysis of mine waste

To determine the thermochemical aspect of the JIG beneficiation and characterize the endothermal and exothermal effects of thermal processing of mine waste, the DSC method was used to characterize the materials under oxidation and reducing atmosphere. The samples from Haldex, Paskov, and Karvina were subjected thermogravimetric analysis (TGA). The measurements were performed using a NETZSCH STA 449 F5 Jupiter simultaneous TG/DSC analyzer (measurement conditions: ground sample ~15 mg in an open corundum crucible; dynamic atmosphere 100 ml min−1; li□e□r he□ti□g from 40°C to 950°C □t □r□te of 10°C mi□$^{-1}$).

The results of analysis of behaviours of thermal recording of different mine waste are shown in Fig. 7. The weight loss corresponds to that depicted in Table 29. It can be noticed in Fig. 7 B that the highest peak of heat exothermal effect was observed in the case of Haldex and the lowest peak was observed in the case of Paskov. It is consistent with the analysed calorific value of these materials (see Table 1). The net calorific value of Haldex samples are the highest values, whereas the NCV of Paskov samples are the lowest values. Moreover, the heat flow peaks of Haldex and Karvina were observed at lower temperature (i.e. 470–500°C) than the peak of Paskov (~530°C). It can be noticed that volatile matter content of Karvina and Haldex is higher comparing to the Paskov values (i.e. Vd= 11.56–13.23 vs. Vd= 7.27–7.41, see Table 1), and the weight loss of Karvina and Haldex in inert atmosphere is significantly higher comparing to the weight loss of Paskov (see Fig. 7 C). The weight loss in inert atmosphere inform on the intensification of volatile matter release during combustion.

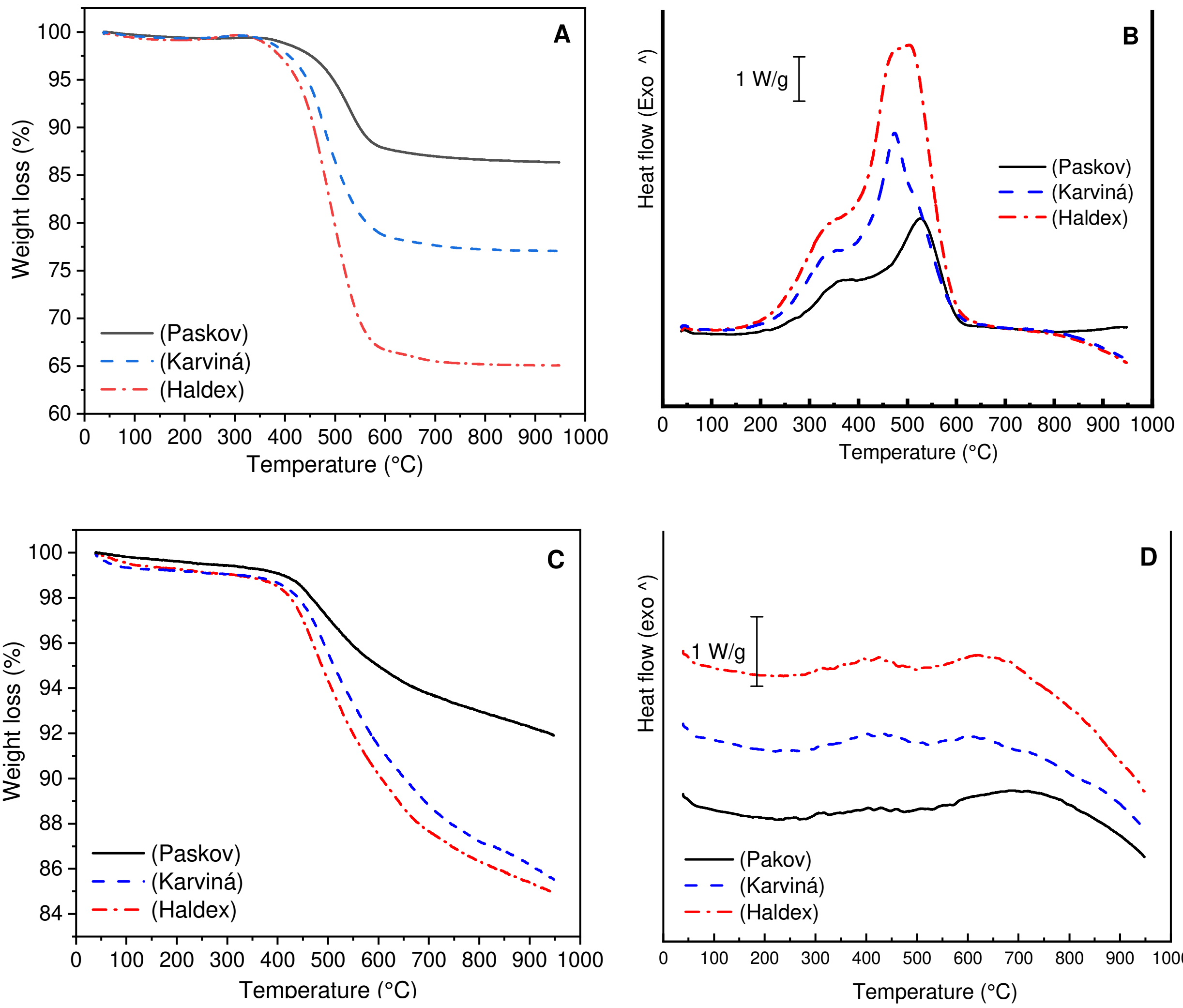


Fig. 7. Thermogravimetric (A and C) and differential scanning calorimetry (B and D) profiles of coal mine tailings in the oxidative atmosphere (A and B) and inert atmosphere (C and D; samples Paskov (–), Karvina (- -), Haldex (-· -).

3.8. Mechanical properties of raw mine waste

In the range of T.2.2 mechanical properties of raw mine waste were examined by GIG. The following tests were carried out:

- aggregate's resistance to fragmentation (Los Angeles coefficient),
- aggregate's abrasion resistance (micro-Deval),
- absorbability,
- frost resistance.

In the first stage of testing, an analysis of mine waste grain size distribution was carried out by the "wet" sieving method (Fig. 8). Square sieves with sides of 0.5m were used for the testing. The holes in the sieves were square with the following dimensions: 32; 16; 8 and 4mm.

Fig. 8. Grain size analysis by the „wet" method

The results of grain size distribution values have been presented in Fig. 9.

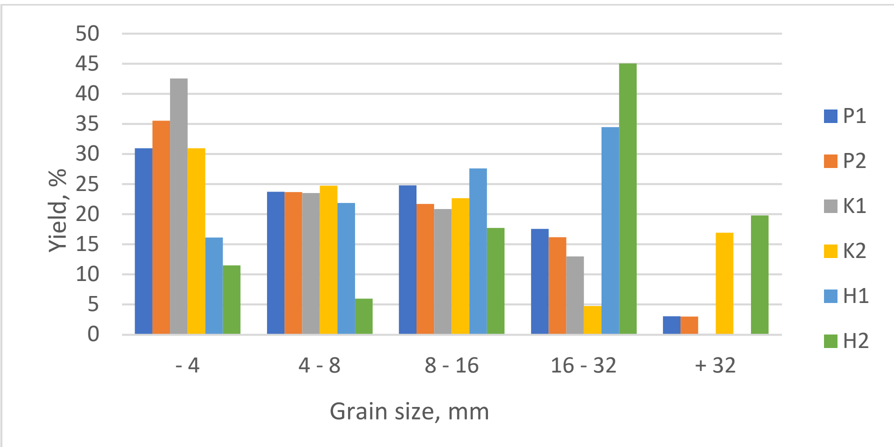


Fig. 9. Analysis of grain size distribution of 6 mine wastes from mine waste dumps

It should be noted that in all applications of rock raw materials, one of the basic requirements is their appropriate grain composition. Waste obtained directly from the heap does not meet these requirements - so the use of even simplified technology is a necessary condition for reclassifying useless waste into a product even for less demanding applications.

Most of the fine material (<4 mm) is contained in waste from Karvina and Paskov mine waste dumps. The 16-32 mm class dominates in Haldex waste. The content of grain classes 4-8 mm and 8-16 mm in all coal waste is similar and ranges from 20-25%.

3.8.1. Determination of aggregate's resistance to fragmentation by the Los Angeles method

To determine the crushing resistance of raw mine waste using the Los Angeles method in accordance with the PN-EN 1097-2 standard, the Los Angeles UTA-0600 drum from Multiserw Morek was used (Fig. 10). The research was carried out at GIG.

Fig. 10. Los Angeles UTA-0600 drum for aggregate's resistance to fragmentation test

The resistance to fragmentation was determined by the Los Angeles method, in accordance with the PN-EN 1097-2 standard. In the first stage, samples of mining waste from mine waste dumps were separated into 5 fractions: >32 mm; 32-16 mm; 16-8 mm; 8-4 mm; <4 mm. In accordance with the requirements of the standard for determining the resistance to fragmentation by the Los Angeles method, a wet fraction with a grain size of 14-10 mm was separated from the 16-8 mm fraction.

This method involves testing the fragmentation of a material in a rotating steel drum containing 11 steel balls with a diameter of 47 mm and a total mass of 4690 g – 4860 g.

Testing procedure and determination of the Los Angeles (LA) coefficient

Approximately 15 kg was taken from the prepared 14-10 mm fraction, which was reduced by the quartering method to a mass of 5000±5 g. After drying to constant mass, the collected waste was placed in the Los Angeles drum with steel balls, and the device was started. The drum rotation speed ranged from 31 to 33 rpm, the number of drum revolutions was 500. After stopping the drum, the material was removed from the drum and washed with water on a 1.6 mm sieve. The residue above 1.6 mm was dried to constant mass and weighed.

The Los Angeles (LA) coefficient was calculated with the use of the following formula:

$$LA = (5000 - m)/50$$

where m - mass of material with grain size above 1.6 mm.

Table 30 presents the LA coefficient values determined for the tested waste.

Table 30. Determination of resistance to fragmentation by the Los Angeles method

Sample	LA coefficient
Paskov1	32.0
Paskov2	34.8
Karvina1	40.0
Karvina 2	43.2
Haldex1	39.8
Haldex 2	50.2

The tables present requirements for units regarding the LA coefficient (*Table 31*).

Table 31. Requirements: Crush resistance categories - Los Angeles index

LA crushing resistance category	LA coefficient
LA_{15}	≤15
LA_{20}	≤20
LA_{25}	≤25
LA_{30}	≤30
LA_{35}	≤35
LA_{40}	≤40
LA_{50}	≤50

The LA coefficient affects the classification of a concrete mixture prepared from a given aggregate to a specific strength class. This is done based on the conversion factor given in the table below.

Table 32. Concrete strength classes depending on the LA coefficient

LA crushing resistance category	LA coefficient
C50/60 and more	≤ LA_{30}
C20/25 - C50/60	≤ LA_{35}
C16/20 - C30/37	≤ LA_{40}
C8/10 - C20/25	≤ LA_{50}

The strength of mine wastes determined by testing the LA index (resistance to crushing) ranges from 32 - 50.2, which corresponds to the LA_{35} – LA_{50} category - this means that the aggregate from such post-mining waste is classified as low-strength aggregate. Haldex mining waste has the highest durability and Paskov1 has the lowest. Based on post-mining waste, it is possible to obtain concrete of the following classes: C20/25 to C50/60, C16/20 to C30/37 and C8/10 to C20/25.

3.8.2. Determination of aggregates' abrasion resistance by the micro-Deval method

The determination of the abrasion resistance of aggregates using the micro-Deval method was performed using the wet method in accordance with the PN-EN 1097-1 standard (Fig. 11). This test was performed in a Micro-Deval U TA-0620 drum from Multiserw Morek.

Fig. 11. Micro-Deval U TA-0620 drum for aggregates' abrasion resistance test

The determination of aggregate abrasion resistance by the micro-Deval method was performed with the use of the wet method in accordance with the PN-EN 1097-1 standard. The analysis of the grain size distribution of individual waste samples revealed that there was insufficient sample mass to determine the above coefficient in the recommended grain class of 10-14 mm, therefore grain classes of 4-8 mm were used for testing. The share of grain loss through an intermediate sieve with a mesh size of 6.3 mm was checked in accordance with the standard. The samples were rinsed in water and dried to constant weight at 110°C. Samples weighing 500 g each were placed in the device container and 2.8 dm^3 of water was added. At the same time, a test on two samples of each waste was carried out. Lids were installed on each container and placed on the shafts of the device for determining the coefficient. The device rotated the containers at a speed of 100± rpm. The set number of revolutions was 12,000±10. After the device had been stopped, the containers were opened and their contents transferred to a protective sieve with a mesh size of 10 mm. The protective sieve was placed on a sieve with a mesh size of 1.6 mm. The material with steel balls was rinsed on sieves in a stream of clean water. The balls were carefully separated from the material on the protective sieve. The remaining mineral material was transferred to the 1.6 mm sieve and rinsed with water. The material remaining on the sieve was transferred onto a tray, dried at 100°C and weighed.

The micro-Deval coefficient was calculated for each sample with the use of the following formula:

$$Mde = (500-m)/5$$

where: Mde – micro-Deval coefficient (determined in a wet state),

m - mass of the fraction retained on the 1.6 mm sieve, in grams.

Based on the two results obtained for each sample, average values were calculated to the nearest integer and summarized in Table 33.

Table 33. Abrasion resistance determined by the micro-Deval method

Sample	Mass [g]	micro-Deval coefficient	Average values
Paskov1	76.7	84.66	85.0
Paskov 2	72.6	85.48	
Karvina1	65.0	87.00	88.0
Karvina2	52.8	89.44	
Haldex1	246.1	50.78	51.0
Haldex2	245.7	50.86	
Paskov1	274.8	45.04	46.0
Paskov2	268.3	46.34	
Karvina1	300.2	39.96	40.0
Karvina2	300.5	39.96	
Haldex1	242.0	51.60	55.0
Haldex2	211.4	57.72	

The Table 34 present the categories of abrasion resistance of coarse aggregate - micro-Deval coefficient.

Table 34. Categories of abrasion resistance of coarse aggregate - micro-Deval coefficient

Categories of abrasion resistance	Micro-Deval coefficient
$M_{DE}10$	≤ 10
$M_{DE}15$	≤ 15
$M_{DE}20$	≤ 20
$M_{DE}25$	≤ 25
$M_{DE}35$	≤ 35
$M_{DEDeclared}$	>30
M_{DENR}	no requirements

The micro-Deval coefficient of the post-mining waste samples ranged from 40 (Karvina) to 85 (Paskov), which corresponds to the abrasion resistance category of the aggregate $M_{DEDeclared}$. In the case of concrete exposed to abrasion, it is recommended to use aggregate with high abrasion resistance - with low micro-Deval coefficients, which limits the use of raw mining waste.

3.8.3. Absorbability of raw mine waste

Absorbability of raw mine waste was determined in GIG in accordance with the PN-EN 1097-6 standard (Fig. 12).

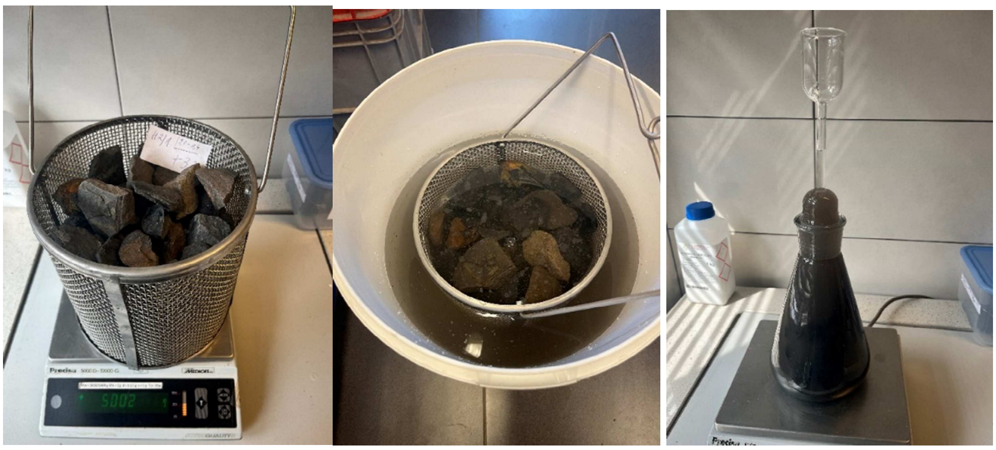


Fig. 12. Absorbability test

Test samples were prepared from a homogeneous material with a mass matched to the grain size of the tested material (Table 35).

Table 35. Weight of waste required for testing

Grain size mm	Min. mass of sample g
+ 8 mm	1000
+ 16 mm	2000
+ 32 mm	5000

The samples were placed in baskets and flooded with water to a height of approximately 5cm above the level of the waste being tested. Next, the tested material was left in water until the samples were completely saturated. After some time, the samples were removed, wiped with a damp absorbent cloth and weighed. The subsequent stage of the test involved drying the sample to constant mass by placing it in a dryer at a temperature of approximately 110°C for 24 hours. After being removed from the dryer, the samples were weighed.

The absorbability of aggregate was determined from the following formula:

$$W_A = \frac{m_1 - m}{m} \cdot 100\%$$

where:

m – mass of the sample dried to constant weight, in grams,

m1 – mass of the sample completely saturated with water, in grams.

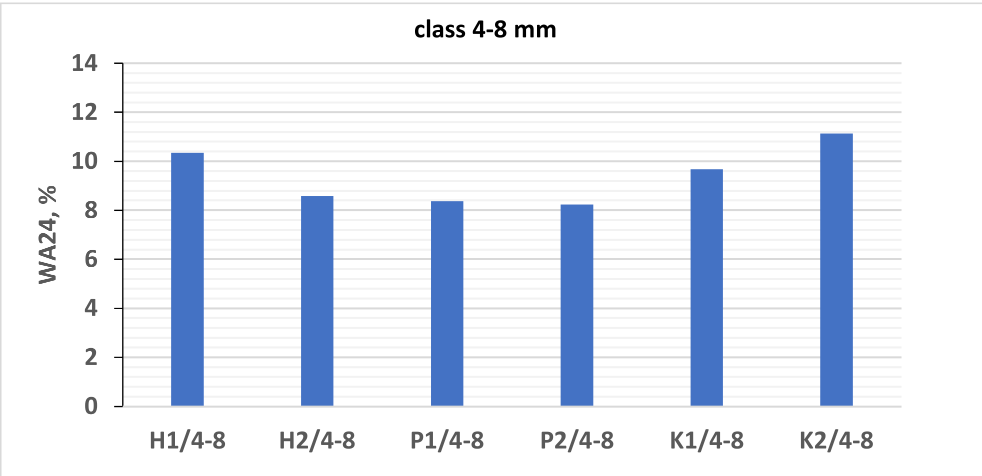
The grain analysis data (Fig. 9) for individual waste samples indicate that the mass of samples over 32 mm was insufficient for determining absorbability in this grain class. Therefore, approximately 1.5 kg of the sample was used for testing in this grain class.

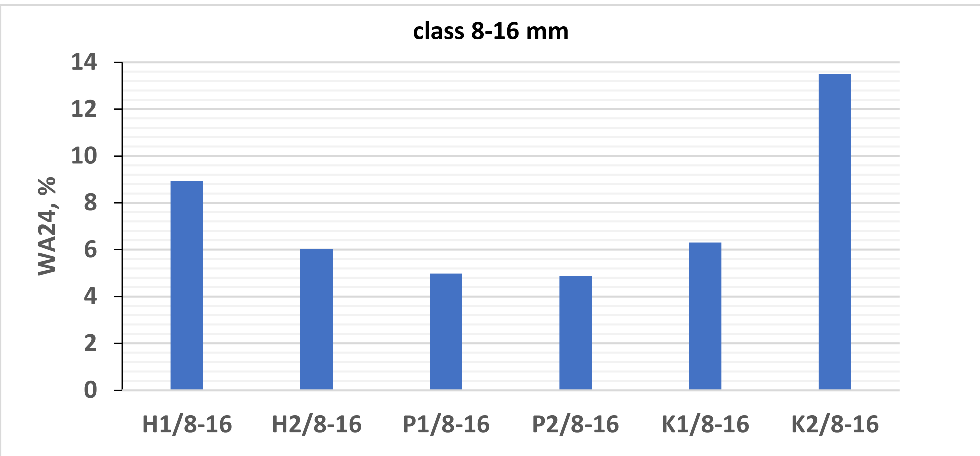
Fig. 13 and Table 36 shows the results of absorbability coefficient determination.

Table 36. Absorbability of waste in grain classes

ID sample	Soaking time [h]	Weight of dry waste M1 [g]	Weight of wet waste M0 [g]	WA24 [%wt]
H1/4-8	24	987.7	1089.9	10.3
H2/4-8	24	1115.4	1211.2	8.6
P1/4-8	24	954.3	1034.1	8.4
P2/4-8	24	971.5	1051.5	8.2
K1/4-8	24	866.4	950.2	9.7
K2/4-8	24	1047.0	1163.5	11.1
H1/8-16	24	1661.9	1810.2	8.9
H2/8-16	24	1522.5	1614.4	6.0
P1/8-16	24	1617.2	1697.7	5.0
P2/8-16	24	1495.7	1568.6	4.9

K1/8-16	24	1364.6	1450.6	6.3
K2/8-16	24	1399.0	1588.0	13.5
H1/16-32	24	1752.5	1854.1	5.8
H2/16-32	24	1954.4	2046.0	4.7
P1/16-32	24	1575.6	1624.1	3.1
P2/16-32	24	1934.1	1991.1	2.9
K1/16-32	24	1414.5	1484.4	4.9
K2/16-32	24	993.1	1036.7	4.4
H1/+32	No sample			
H2/+32	24	4947.1	5093.1	3.0
P1/+32	24	1425.8	1453.7	2.0
P2/+32	24	1650.1	1680.0	1.8
K1/+32	No Sample			
K2/+32	24	4893.0	5091.0	4.0





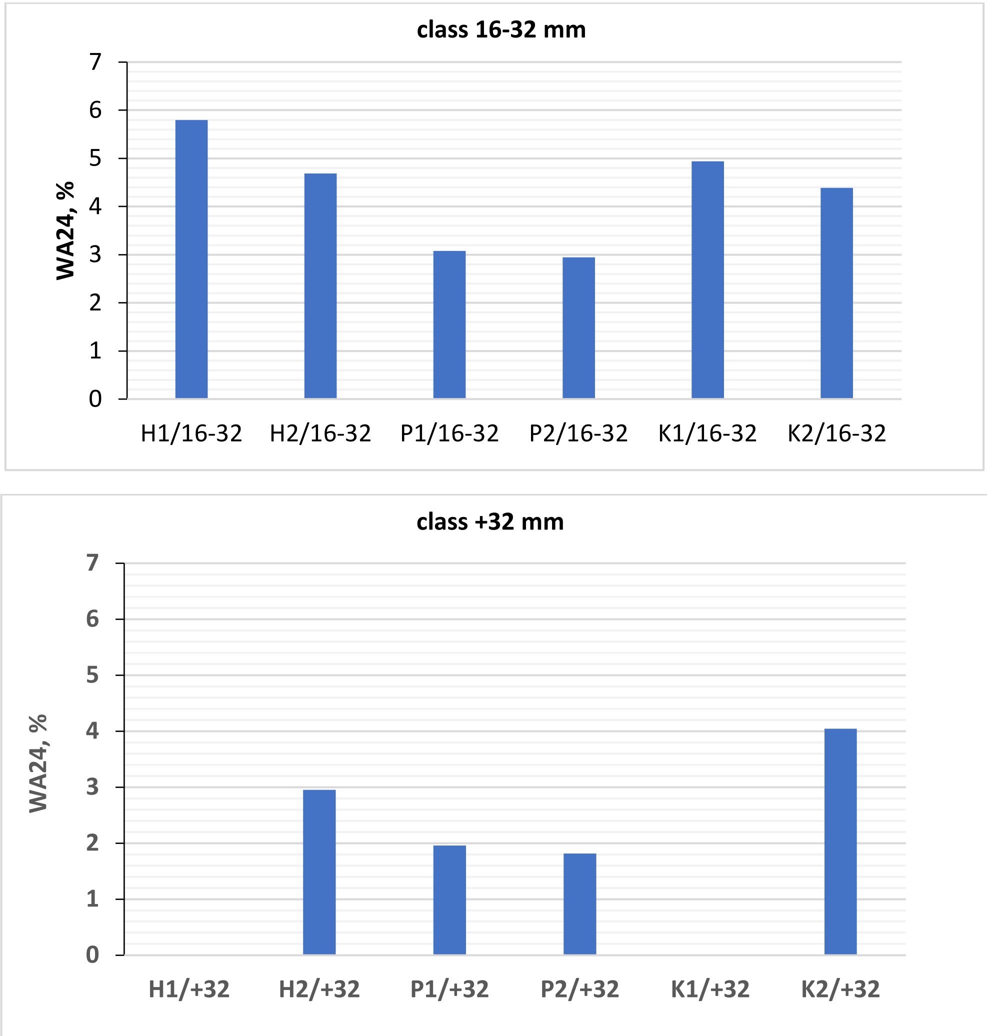


Fig. 13. Absorbability of raw mine waste in grain classes

Absorbability of raw mine waste ranges from 1.8% (Paskov 2, +32 mm) do 13.5% (Karvina 8-16 mm) which means that the standard parameter WA24 is the declared value for all products.

In accordance with the requirements of European standards, aggregate absorbability can be assumed as an indicator of frost resistance (PN-EN 1097-6). It is believed that absorbability not exceeding 1% guarantees the frost resistance of an aggregate. This parameter is met by all tested samples of post-mining waste.

3.8.4. Frost resistance of raw mine waste

SZM-700 climatic chamber and stainless steel containers with covers (producer UNI-MORS Łukasz Mnich), presented in Fig. 14, were used for testing the resistance to freezing and thawing of raw mine waste in accordance with the following standards: PN-EN 1367-1:2007 and PN-EN 1367-6:2008.

Fig. 14. SZM-700 climatic chamber for frost resistance tests

Frost resistance tests were performed according to the PN-EN 1367-1 standard. The tests were carried out for the following grain classes: 4-8 mm, 8-16 mm, 16-32 mm.

The tests consisted of the following stages:

1. The waste sample collected for testing was dried to constant mass at a temperature of (110 ± 5)℃.
2. The waste sample (M1) was placed in a metal box with the following dimensions: external diameter: 130 mm, external height: 200 mm. The minimum mass of the test sample is specified in Table 37.
3. Distilled water was poured into the waste (10 mm above the upper layer of the waste) and left for 24 h at a temperature of (20 ± 5)℃.
4. The can was covered and placed in the climatic chamber.
5. The cycle of temperature changes in time was as follows:
   the temperature was changed from (20 ± 5)℃ to (0 to -1℃) in (150 ± 60) min and the samples were kept at the set temperature for (210 ± 90) min.

the temperature was changed from (0 to -1)℃ to (-17.5 ± 2.5)℃ in (180 ± 60) min and the samples were kept at the set temperature for a minimum of 240 min
the can was removed from the chamber and thawed by immersing it in water.
the cycle lasted a total of 24 hours.

6. Points 4 – 5 were repeated 10 times.

After 10 cycles, the sample was passed through a sieve with the mesh size specified in Table 37. The residue on the sieve was dried at a temperature of (110 ± 5)℃ and weighed (M2).
The F factor, determining the percentage loss of sample mass after 10 cycles of freezing and thawing, was calculated according to the following formula:

$$F = \frac{M1 - M2}{M1} * 100$$

Table 37. Sieve size and the mass of waste required for testing

Grain mm	Min. Mass of sample g	Sieve mm
4 - 8	1000	2
8 - 16	2000	4
16 - 32	4000	8

The frost resistance test results are presented in Table 39 and in Fig. 15 shows the results of frost resistance determination.

Table 38. The results of frost resistance (F) determination

ID sample	Number of cycles	Initial mass of waste M0 [g]	Final weight of waste M1 [g]	F
H1/4-8	10	1114.6	806.6	27.6
H2/4-8	10	1071.3	640.7	40.2
P1/4-8	10	1061.7	976.1	8.1
P2/4-8	10	1094.2	1006.1	8.1
K1/4-8	10	1056.4	778.7	26.3
K2/4-8	10	1095.2	737.9	32.6
H1/8-16	10	1604.4	731.2	54.4
H2/8-16	10	1563.4	934.3	40.2
P1/8-16	10	1828.2	1598.9	12.5
P2/8-16	10	1719.4	1491.3	13.3
K1/8-16	10	1476.3	866.7	41.3
K2/8-16	10	1373.6	554.0	59.7
H1/16-32	10	2121.1	1331.3	37.2
H2/16-32	10	2127.4	1468.4	34.1
P1/16-32	10	2145.0	1758.6	16.5
P2/16-32	10	2137.3	1803.3	15.6
K1/16-32	No sample			
K2/16-32	10	2175.6	1585.1	30.5

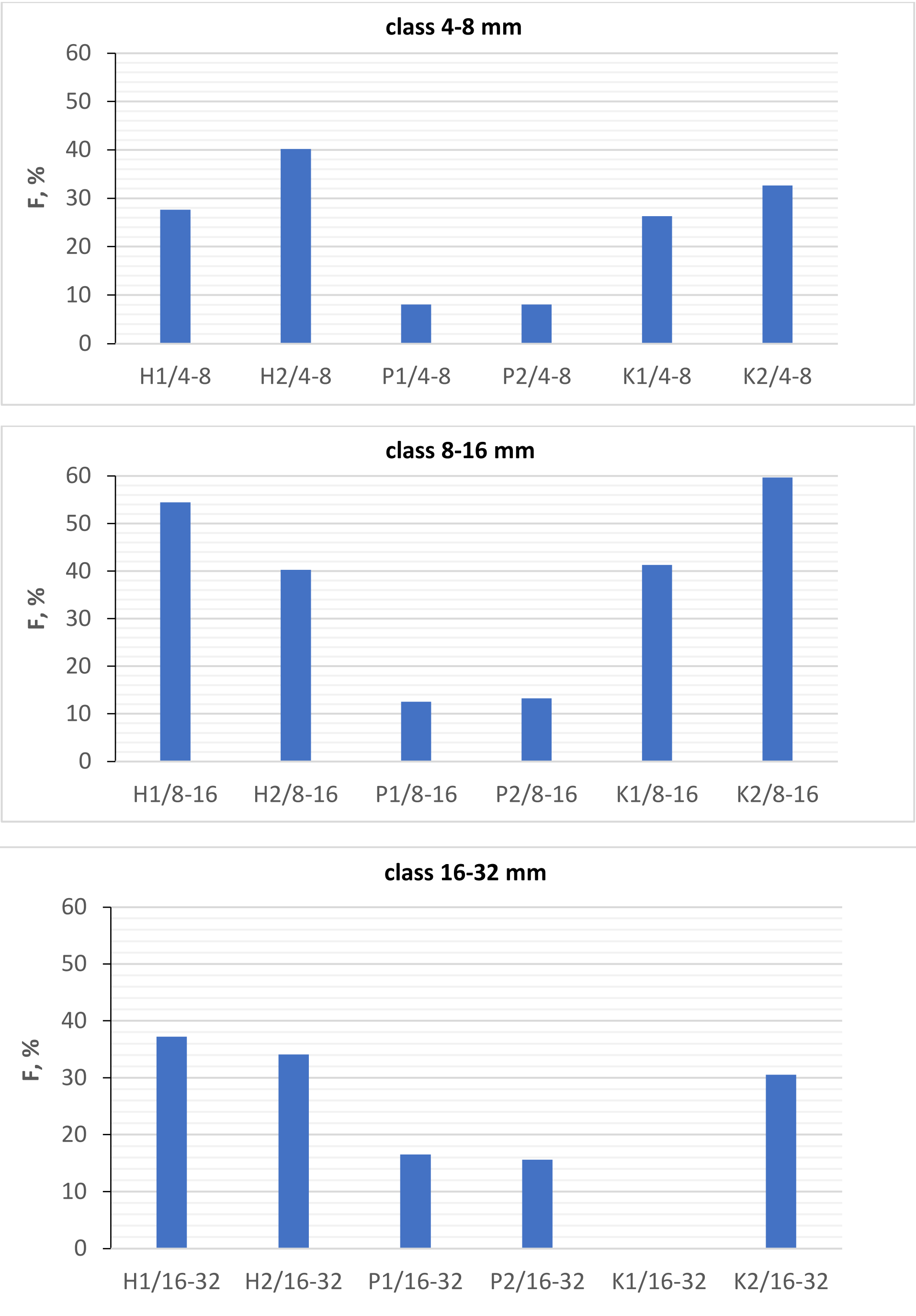


Fig. 15. Frost resistance of waste in grain classes

The frost resistance index of raw mine waste ranges from 8.1 to 59.7. The frost resistance index F for all mining waste is a declared value. For all waste, this parameter is higher than the lowest one specified in the standards, with a value of F = 4.

3.9. Analysis on an SEM/EDS Scanning Electron Microscope

A scanning electron microscope was used to observe the surface structure of the samples and for chemical identification. The scanning electron microscope (SEM) enables the observation of the surface structure of matter at the microscopic level, at a magnification of 5-300,000x. It is used to observe and characterize organic and inorganic materials at the nanometres to micrometres scale. The low observation vacuum allows the observation of non-conductive specimens and samples containing water without the need to prepare the preparation. The SEM located in GIG laboratory allows for observations using topographic contrast - Secondary Electron (SE detector) and contrast based on the difference in atomic numbers of individual elements of the structure – BSE (Backscatter Electron). The EDS (Energy Dispersive Spectroscopy) microanalyzer allows the identification of the elemental composition of the tested material for all elements with an atomic number greater than boron. Most elements are detected at concentrations of 0.1%. SEM-EDS is a test that allows the analysis of each grain and their distribution in the sample taken. It is used to identify inorganic compounds in an image. The SEM scanning electron microscope enables, among others: examining and analyzing the surface and subsurface areas of materials, analyzing the chemical composition of materials, analyzing crystallographic orientations, determining magnetic properties and electrical materials, material quality control, determining intracellular structures and processes, finding and recognizing chemical substances.

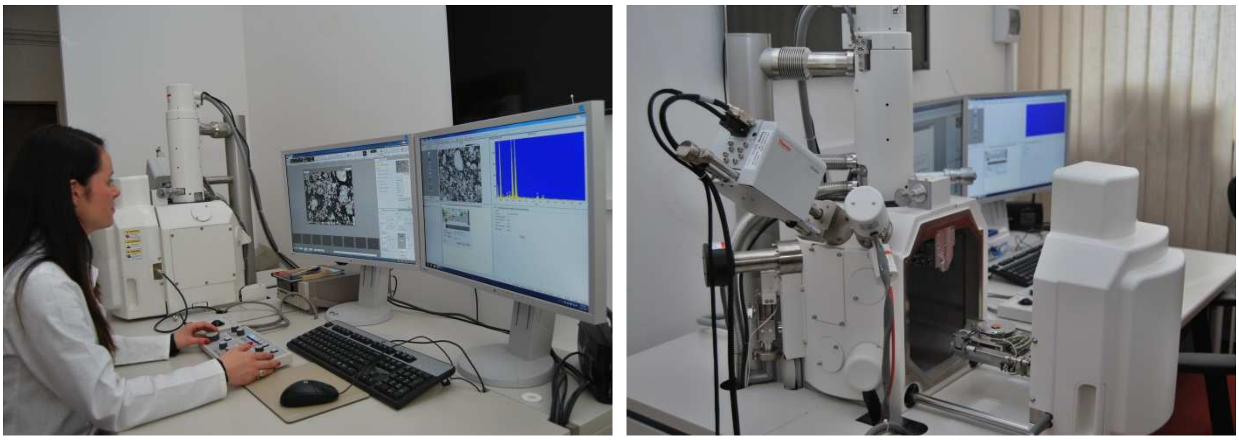
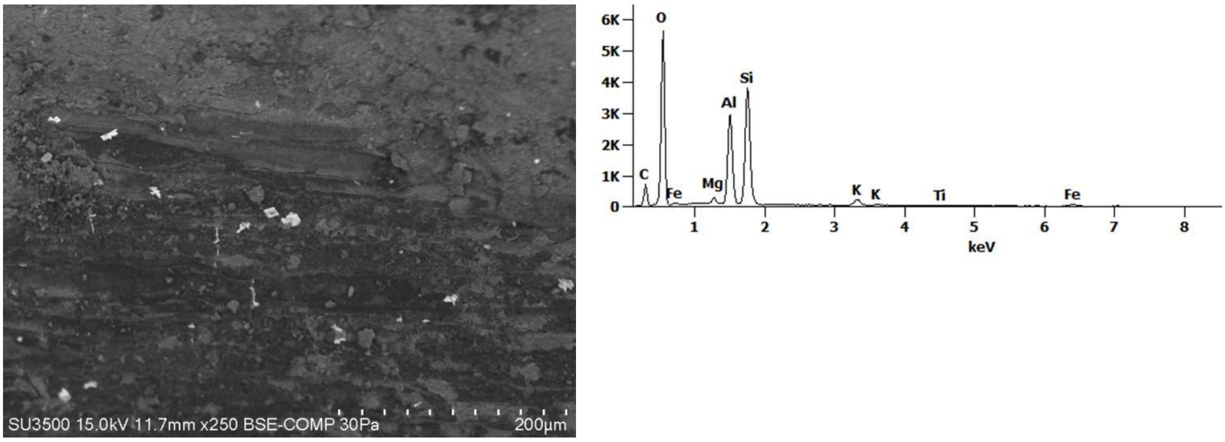


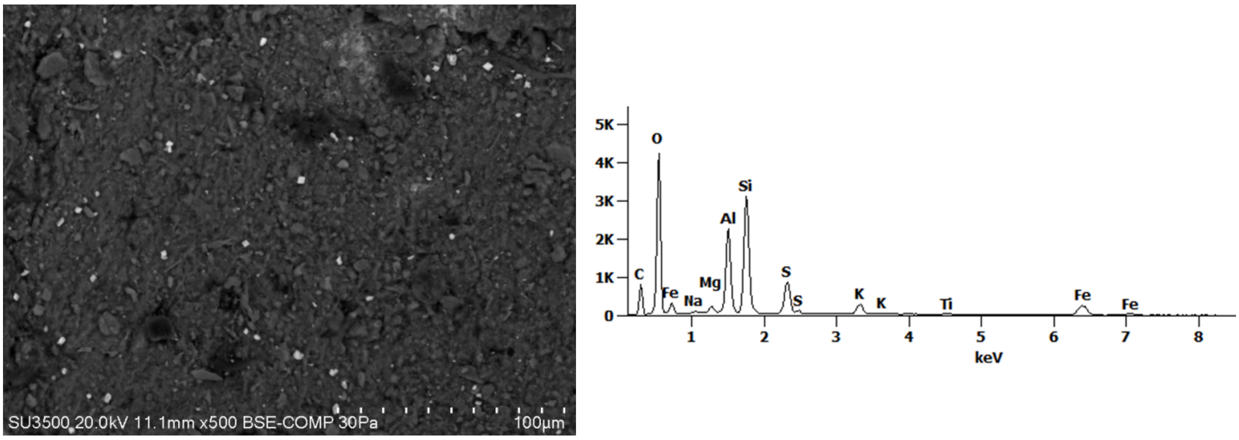
Fig. 16. Scanning Electron Microscope

The structure of the samples' surface was observed in a SEM-SU3500 variable vacuum scanning electron microscope from Hitachi, combined with an EDS UltraDry energy-dispersive X-ray spectrometer from Thermo Scientific. The tests were carried out in a variable vacuum mode at an accelerating voltage of 15 keV.

For the purposes of the preparation, several BSE (backscattered electrons) images were recorded, showing the morphology and dimensions of waste particles and the diversity of chemical composition.



a



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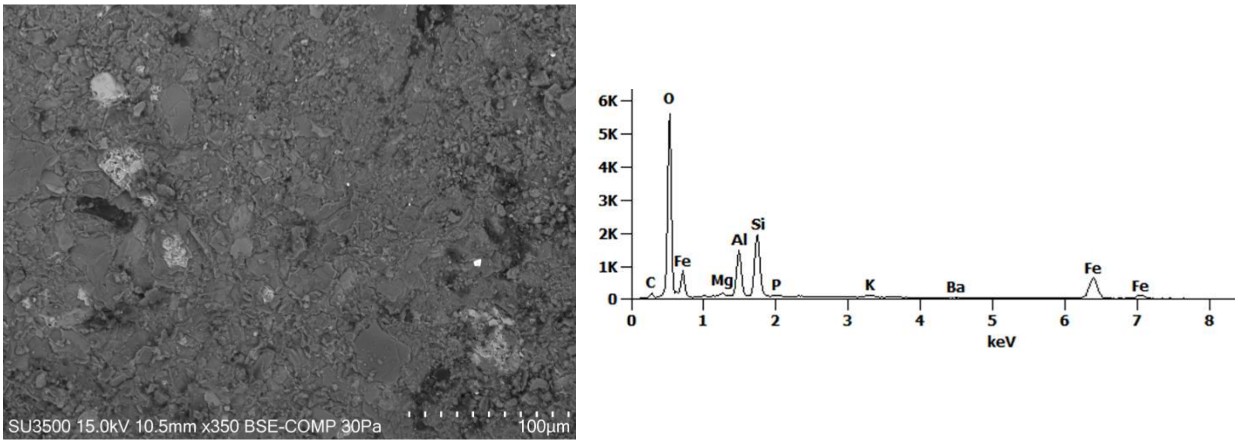
SU3500 15.0kV 10.5mm x350 BSE-COMP 30Pa
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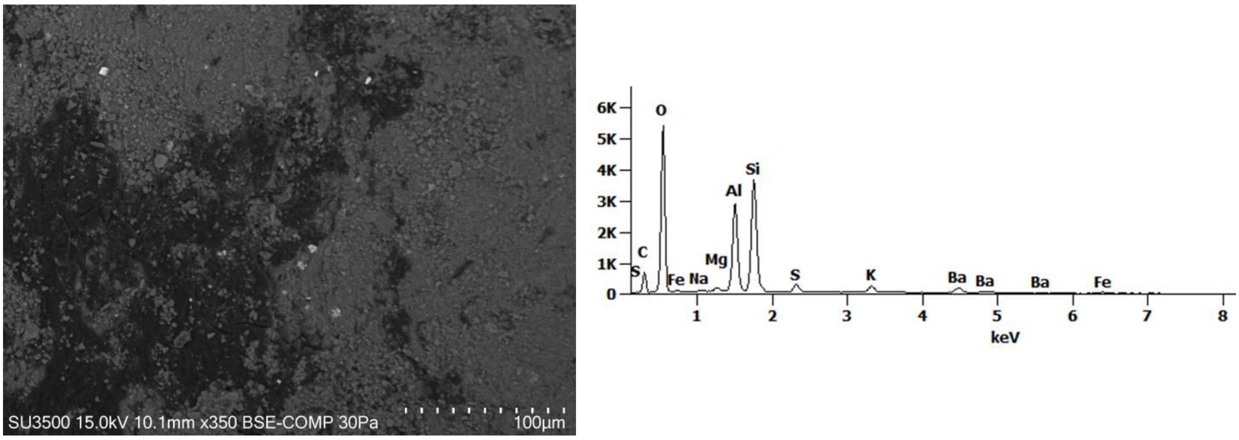
SU3500 15.0kV 10.1mm x350 BSE-COMP 30Pa
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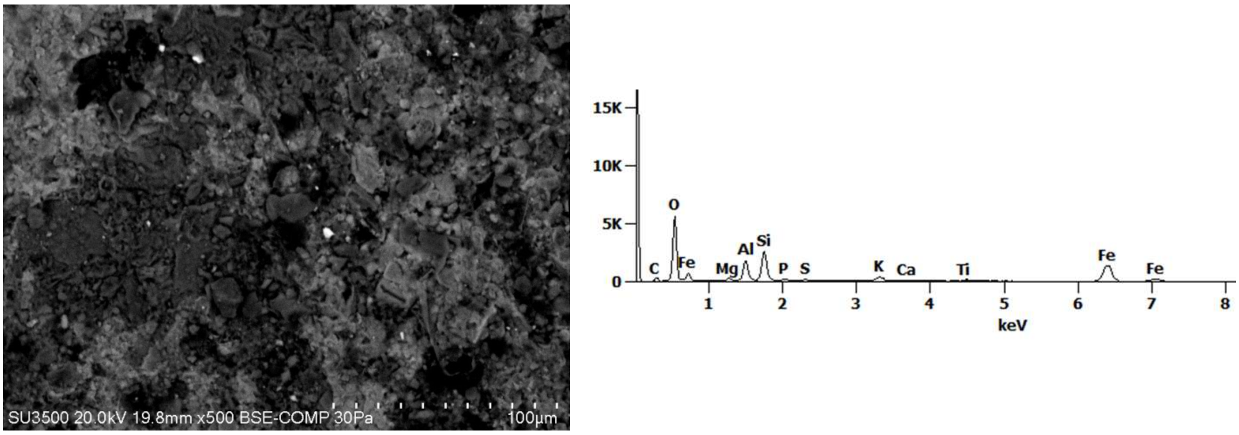
SU3500 20.0kV 19.8mm x500 BSE-COMP 30Pa
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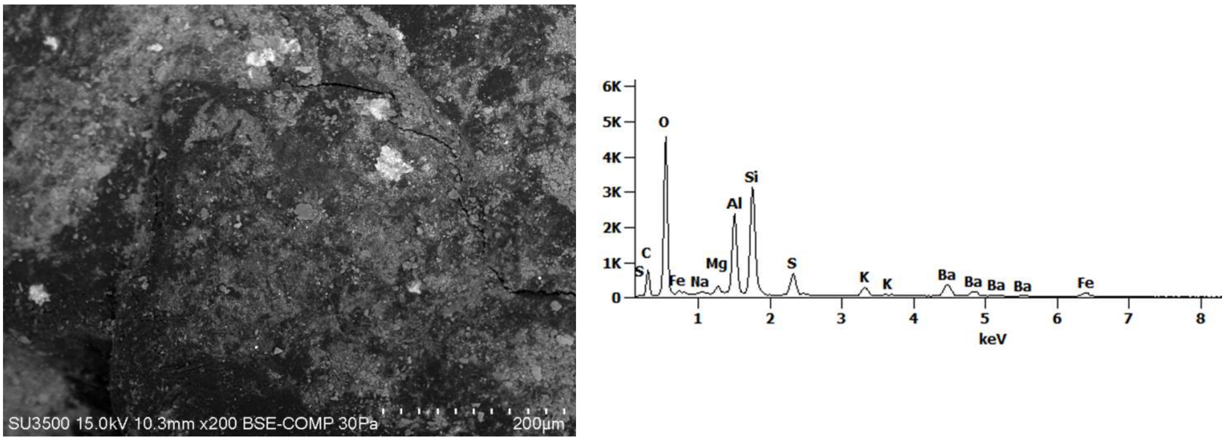


f

Fig. 17. Sample morphology and chemical composition of mine waste from mine waste dumps: a) Haldex 1 b) Haldex 2 c) Karvina 1 d) Karvina 2 e) Paskov 1 f) Paskov 2, observed in SEM EDS.

For selected molecules, EDS spectra illustrating their chemical composition were recorded. A series of micro-analyses were performed for the samples, including several to several dozen measurements of their chemical composition in order to determine the presence of individual elements. The final result is the average of the chemical composition measurements in the micro-area.

The performed analyses indicate that a dominant component in all the samples subjected to testing was Si, the average content of which ranged from 12.56 to 18.60 wt.%, as well as Al, with an average content ranging from 8.96 to 11.76 wt.%, and Fe - 12.49 to 4.38 wt.%. Ba was also found in the samples, the average content of which ranged from 3.09 to 7.24. The contents of other chemical components, such as: Na, Mg, P, S, K, Ca, Cl, Ti, Cr, Mn, Co, Ni, Zn, reached several percent. There were also occasional grains containing rare earth elements, i.e. La, Ce, Nd, Eu (Fig. 18).

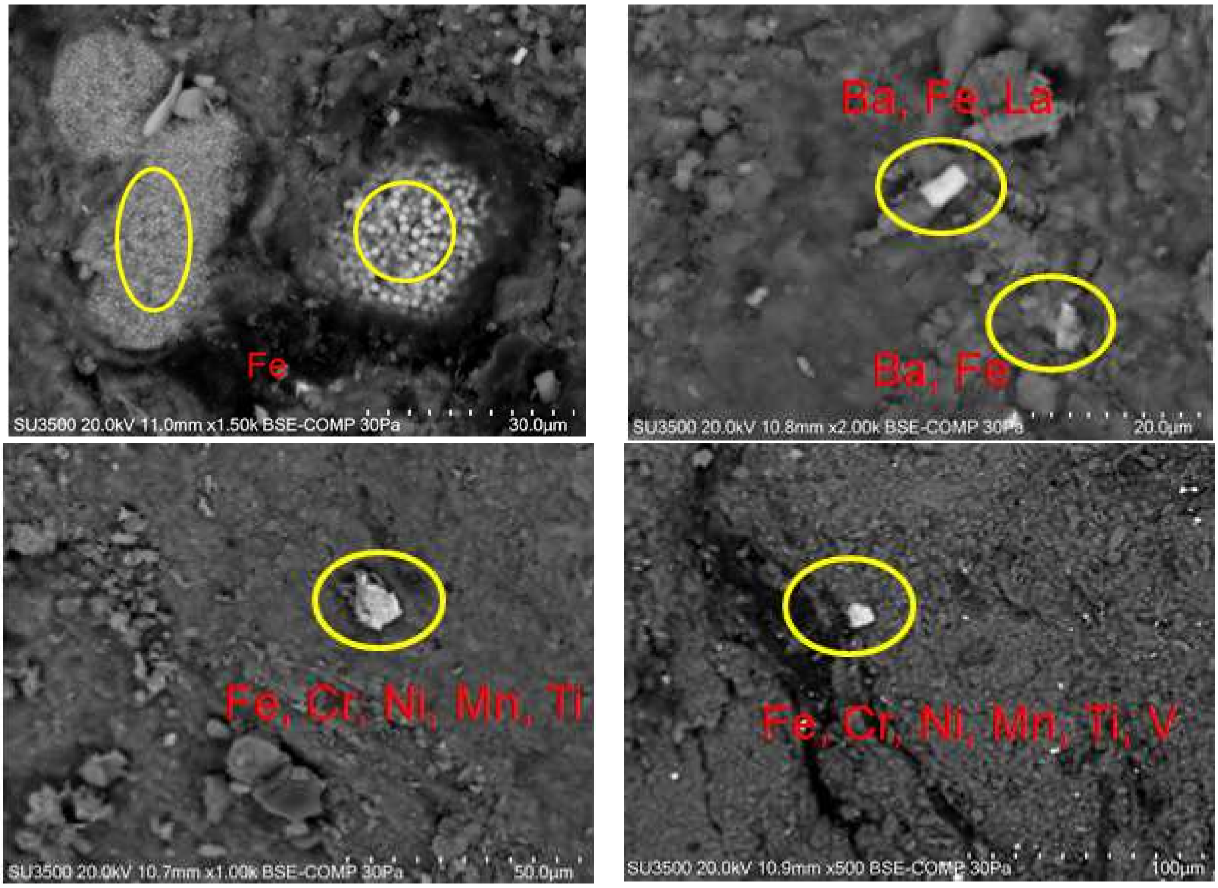


Fig. 18. Morphology and chemical composition of the grains

3.10. Morphological examination of raw mine waste using polarizing microscope

The microscopic analysis of mine waste dumps samples was done by ITPE using Microscope Axio Imager M1m (Carl Zeiss, Germany) (Fig. 19), MSP 300 Spectrophotometer (J&M Microsystems, Germany) and AxioVision programme (for image registration and analysis).

Fig. 19. Photo of Microscope Axio Imager M1M (Carl Zeiss, Germany)

The morphological characteristics of the samples were determined, consisting in defining the composition of the material and the degree of possible transformations of organic matter as a result of temperature or chemical factors. Fig. 20 – Fig. 25 show exemplary photomicrographs of raw mine waste sample from Karvina, Paskov and Haldex.

The delivered samples were dried and averaged, and then an analytical sample weighing approximately 300 g was prepared from each waste sample. Due to the high content of inorganic matter (rock) that is difficult to crush, the sample was not prepared for petrographic analysis in accordance with the ISO 7404-2 standard. i.e. grain size <1 mm. Due to the possibility of grinding too deeply the coal matter accompanying the rock. it was decided to crush the sample to a grain size of <3 mm. Each sample was prepared for microscopic analysis according with the recommendations of the ISO 7404-02 standard (apart from sample grain size). Approximately 5 g was taken from each sample and prepared pellet. by embedding in acrylic resin and polishing using an automatic grinder & polisher and diamond/oxide suspensions. The prepared preparations were subjected to microscopic evaluation.

Additionally a reflectance analysis (PN-ISO 7404-5:2002) was performed for all samples in order to determine the degree of coalification of the organic matter contained in the sample and the homogeneity of the samples. The calibration was made based on standards with R values equal to 0%, 0.595%, 0.909%. and 1.722%. The examination was performed in white light at x 500 magnification in oil immersion.

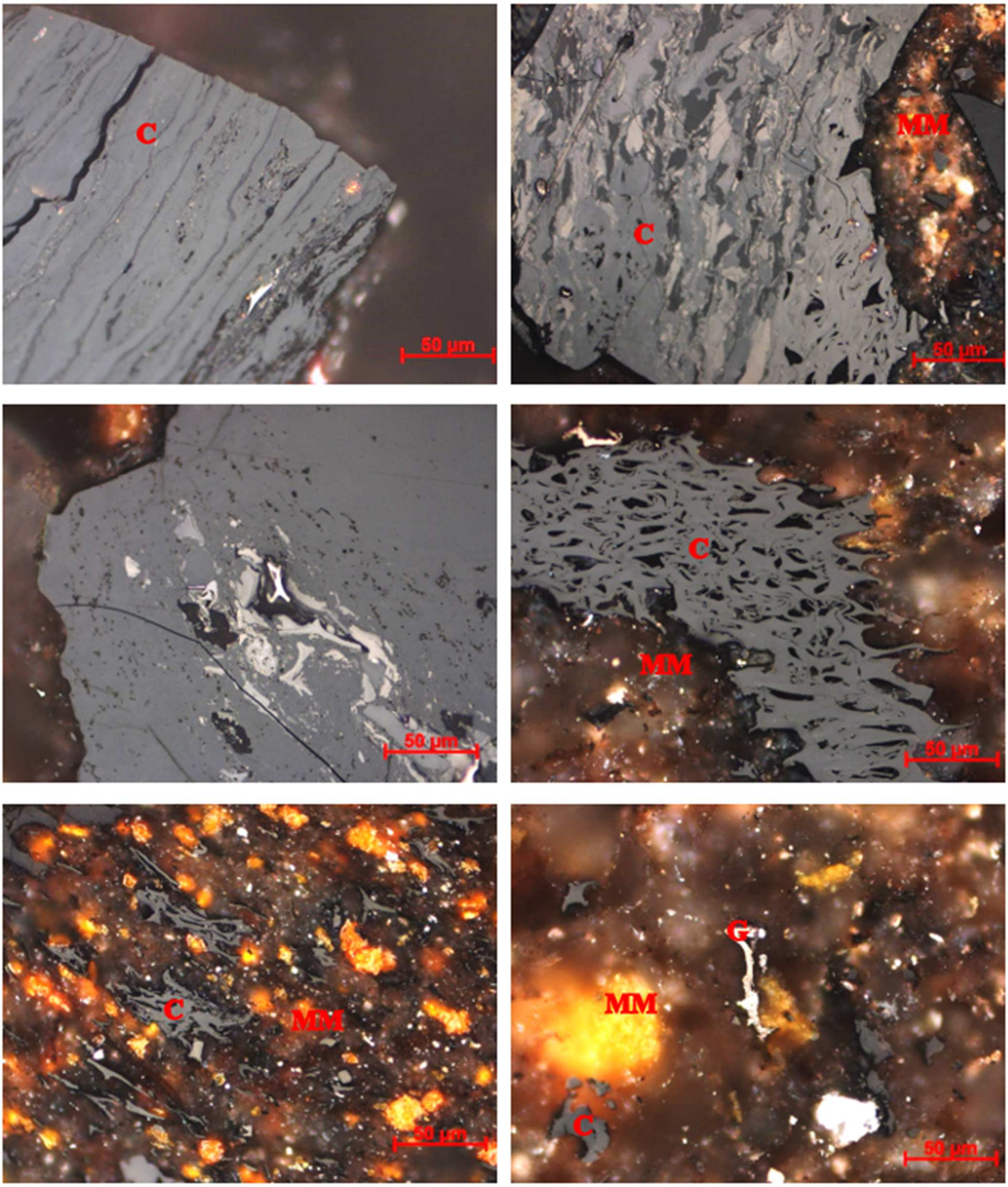


Fig. 20. Exemplary photomicrographs of the Karvina1 sample (white light, reflected, oil immersion, area ×500) C – coal substance; MM- mineral matter; G-graphite

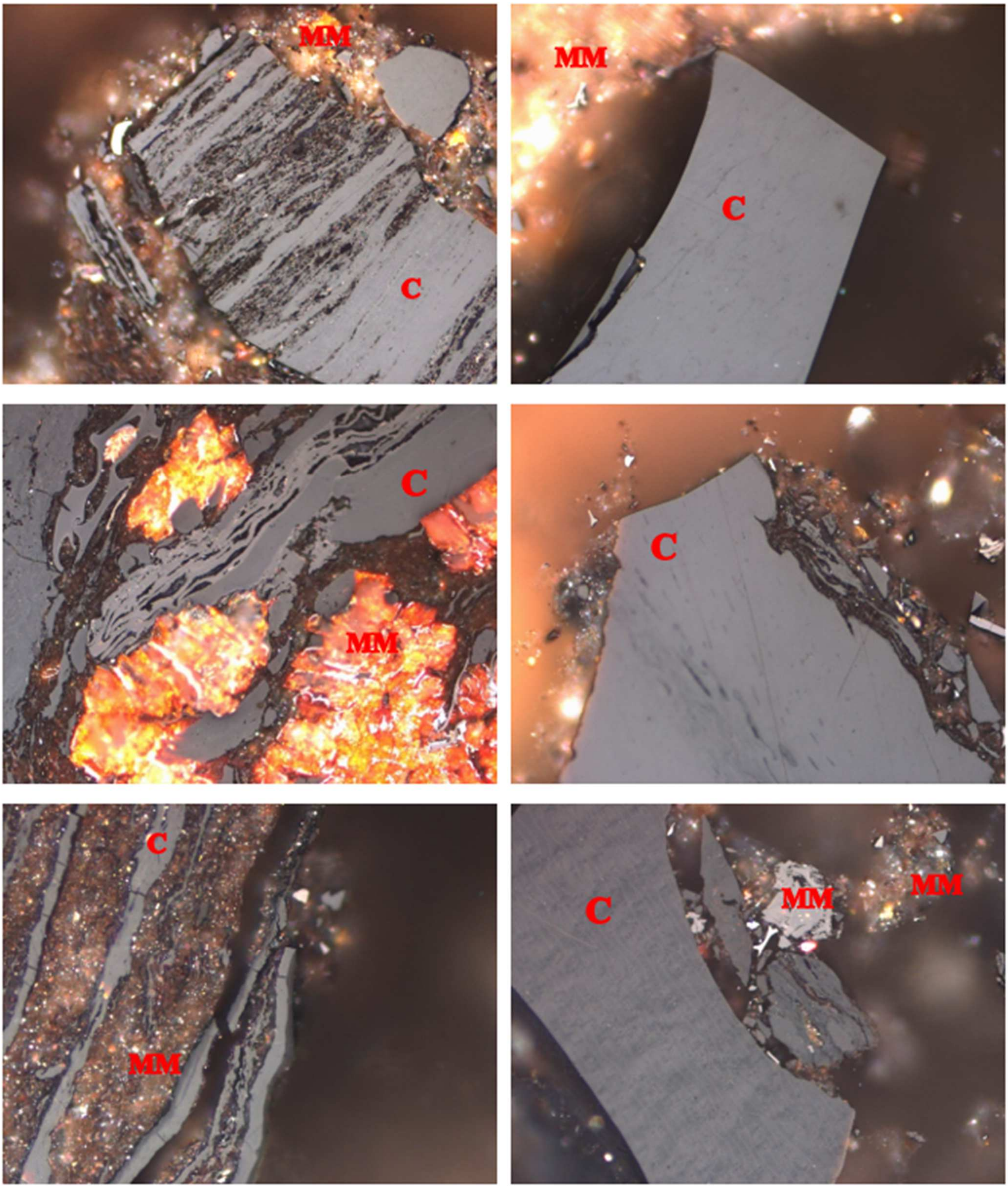


Fig. 21. Exemplary photomicrographs of the Karvina 2 sample (white light, reflected, oil immersion, area ×500) C – coal substance; MM- mineral matter

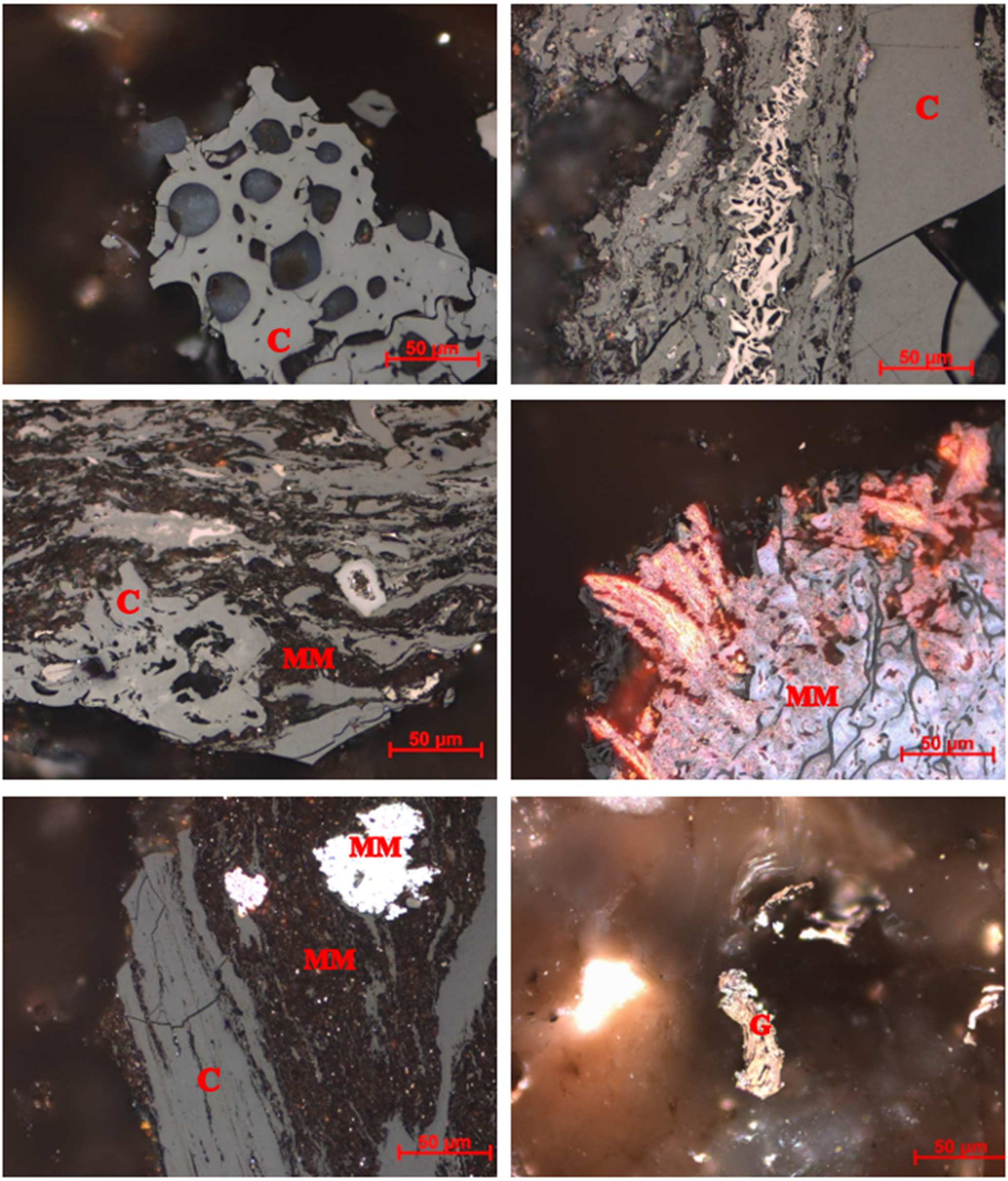


Fig. 22. Exemplary photomicrographs of Paskov1 sample (white light, reflected, oil immersion, area ×500) C – coal substance; MM- mineral matter; G-graphite

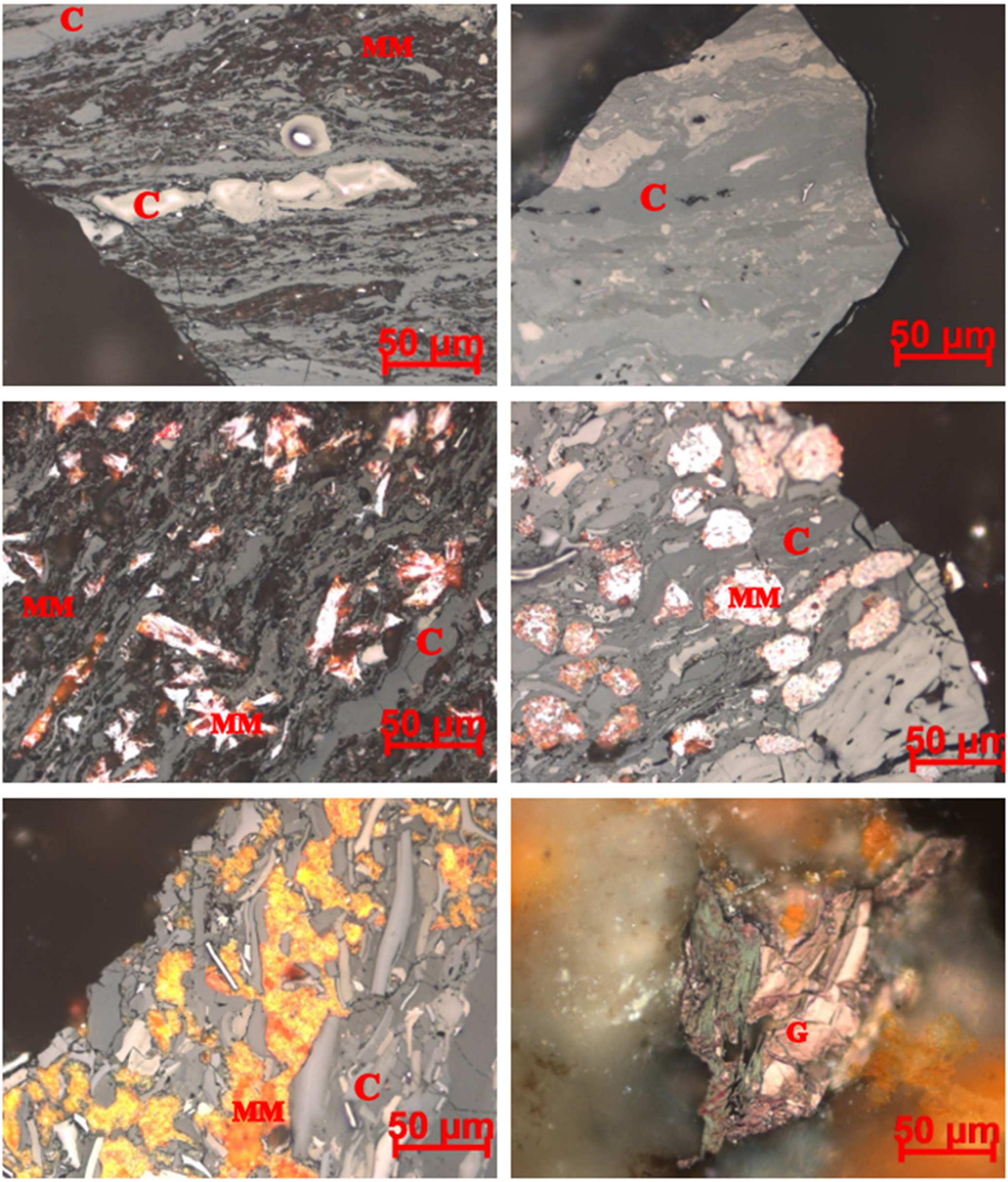


Fig. 23. Exemplary photomicrographs of Paskov2 sample (white light, reflected, oil immersion, area ×500) C – coal substance; MM- mineral matter; G-graphite

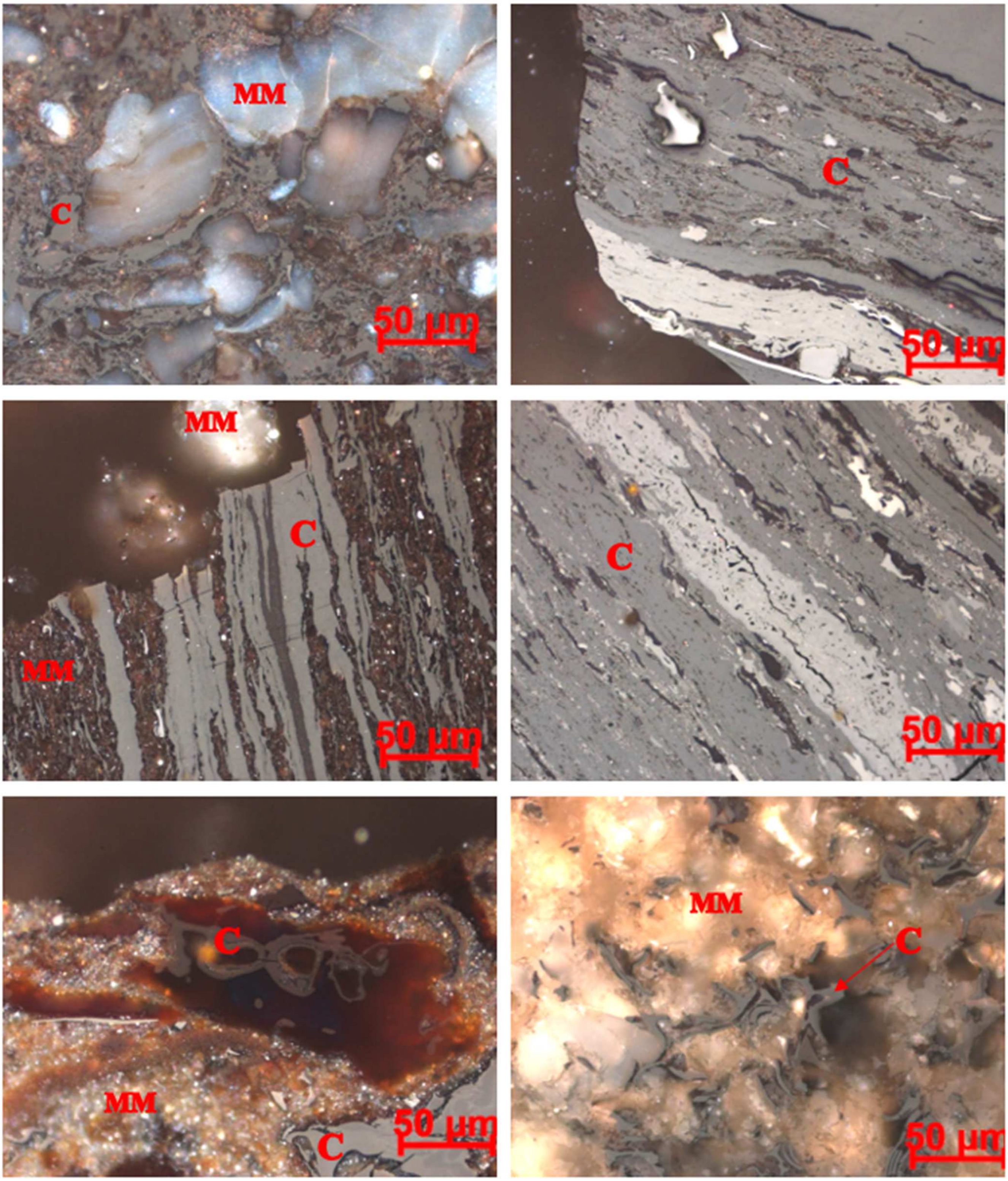


Fig. 24. Exemplary photomicrographs of Haldex1 sample (white light, reflected, oil immersion, area ×500)
C – coal substance; MM- mineral matter; G-graphite

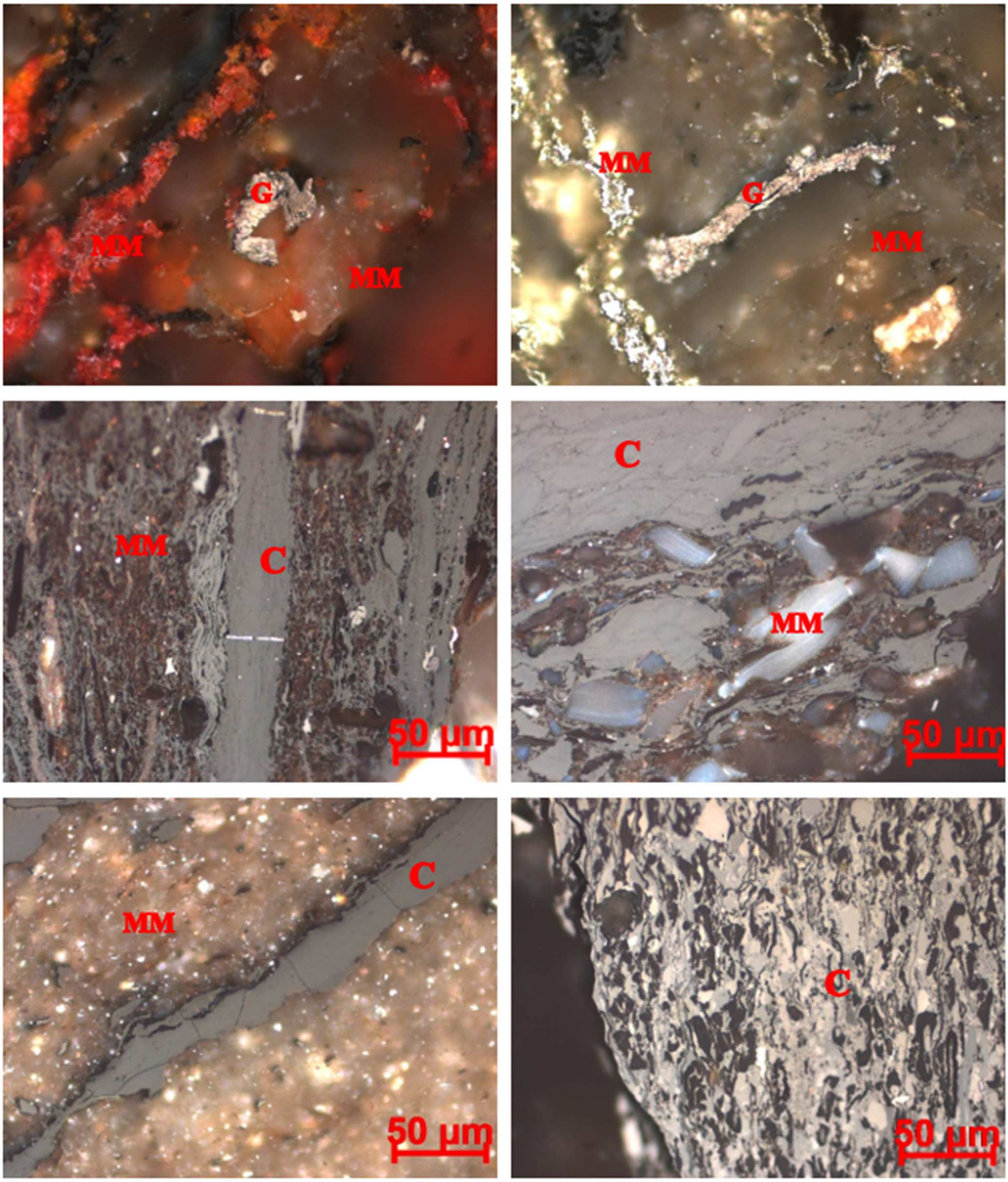
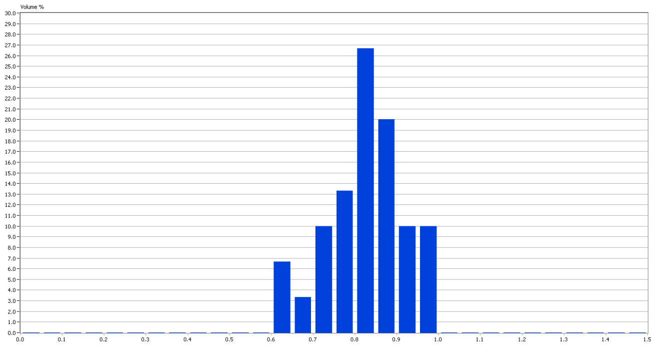
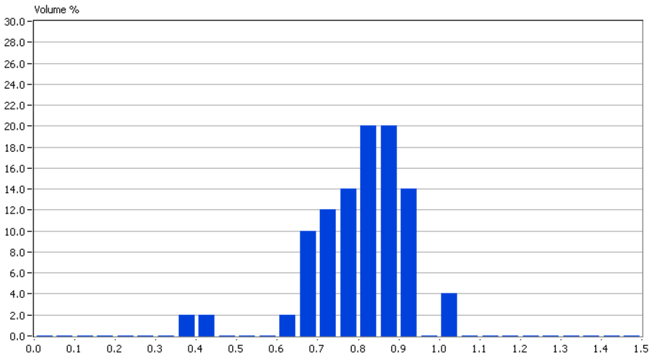
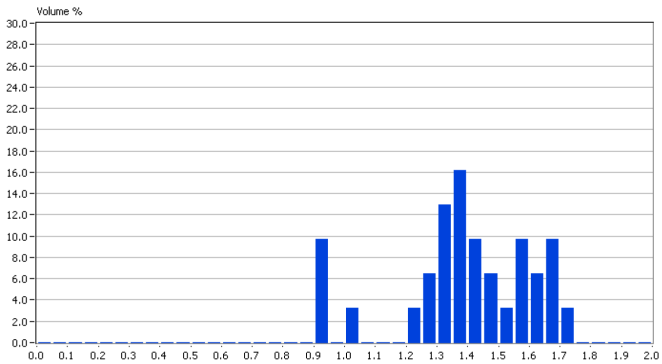
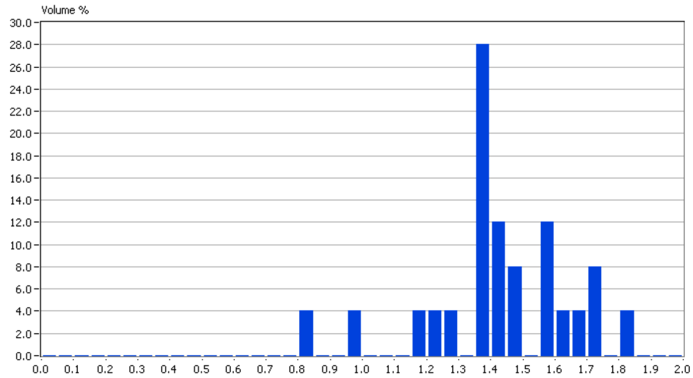
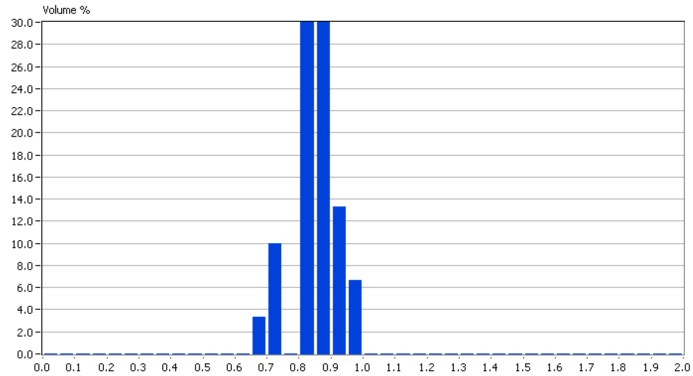
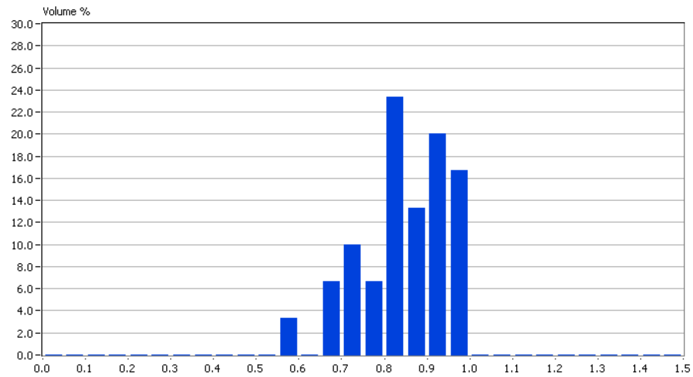


Fig. 25. Exemplary photomicrographs of Haldex2 sample (white light, reflected, oil immersion, area ×500) C – coal substance; MM- mineral matter; G-graphite

Microscopic evaluation – reflectance and homogeneity

Table 39. Mean random vitrinite reflectance values (VR0) of the post-mining waste samples

Sample	VR_0 [%]	Reflectance histogram
Karvina1	0,83	
K□rvi□□2	0,83	
Paskov1	1,40	

Paskov2	1,46	
Haldex1	0,85	
Haldex2	0,85	

The aim of the study was the optical and morphological characterization of project samples originating from post-mining waste heaps. The analysis intended to resolve microscopic textural and structural features which can be applied to detect alteration processes of organic matter (thermal and/or chemical) occurring in the heap. Additional measurement of mean reflectance of vitrinite was performed to evaluate the waste origin and homogeneity.

Morphological characterization consisted in determining the material composition and the level of possible alteration of organic matter as a result of the influence of temperature or chemical factors (oxidation/weathering, gasification).

Phenomena indicating thermal or chemical transformation of organic matter are usually:

- the presence of round pores or cracks resulting from the gasification of organic matter;
- fraying of the edges of the grains, development of the outer surface of the grains, resulting from the gasification of organic matter;
- rounding of the grain edges, development of the outer surface of the grains, resulting from the plasticization of organic matter as a result of pyrolysis;
- presence of oxidation rims in coal grains (light or dark depending on the degree of coalification of the initial organic matter);
- increase of anisotropy, appearance of a mosaic optical texture;
- delamination in line with the organic matter stratification.

The conducted microscopic morphological analysis of mining waste samples did not show any visible changes indicating that the organic matter contained in the waste had undergone any thermal or chemical transformations (Fig. 20 – Fig. 25). No features indicating oxidation, gasification or pyrolysis processes were observed. The organic matter may still have been partially weathered, the symptoms of which cannot be observed or determined without an in-depth geological analysis of the raw material in the deposit from which the waste originates.

Reflectance analysis was performed for all samples in order to determine the degree of coalification of the organic matter contained in the sample and the homogeneity of the samples (Table 39). All tested samples were characterized by a very low organic matter content, therefore the reflectance analysis was carried out according to the principles adopted for DOM (Dispersed Organic Matter). Despite the dominant content of inorganic matter, the samples showed significant homogeneity.

The test results indicated that most likely all samples came from heaps where coal from one mine or several mines with the same petrographic characteristics of the coal deposit, were stored. Paskov 1 and Paskov 2 samples are characterized by broader reflectogram bases. According to the description, the samples probably the wastes come from the Paskov mine. Coal from the Paskov mine is characterized by a high degree of coalification and a wide range of coalification, and consequently broad histogram. The results do not differ from expectations.

3.11. Degree of respiratory activity of microorganisms

For selected samples of mining waste dumps (Haldex, Karvina, Paskov) respiratory activity was determined. It is a parameter used for determining the biological stability/activity of waste. The AT4 test was performed in GIG according to the ONORM S2027-4:2011 standard. The results of testing the degree of respiratory activity of microorganisms have been presented in Table 40.

Table 40. Degree of respiratory activity of microorganisms in mine waste

Samples	AT4 [mg O2/g]
Paskov1	<4
Paskov2	<4
Karvina1	<4
Karvina 2	<4
Haldex1	<4
Haldex2	<4

Based on the tests performed, it can be concluded that all samples taken from landfills have a value lower than the statutory value (4 mg O2/g], which means that this waste can be safely stored in post-mining waste landfills.

4. Scenarios and guidelines for the preparation of a mixture of bio-waste and plant seeds

4.1. Literature analysis on reclamation of post-mining dumps

Industrialized regions with developed, either in the past or at present, mining and processing industries, are characterized by a large transformation of the land surface, as well as a significant share of devastated post-mining areas. In these areas there are landfills for various types of waste. This is especially true in areas where mining activities related to the extraction of mineral resources, including hard coal, were (and are) carried out. The production, and consequently the storage of waste rock and tailings material, consumes huge areas creating dumps, commonly called heaps[13,14]. Devoid of vegetation cover, they are particularly troublesome for the environment. This is associated with dust (PM10, PM2.5), chemical and thermal activity, as well as pollution of groundwater and surface watercourses[15,16]. The degradation of the landscape forces to take appropriate steps to manage these areas by restoring the vegetation cover.

It is assumed that restoration techniques in the form of greening devastated areas should be based on the use of plants from local resources. This vegetation spontaneously and spontaneously forms on unreclosed post-industrial dumps for many years, forming the most permanent cover[17,18].

The survey[19] showed that reclamation treatments in the form of sodding, shrubs and bushes are often used on post-industrial dumps. Most often these are common plants (shrubs and native trees), but there are also ornamental species. With the passage of time, planted plants (which often require maintenance) are displaced by plants from spontaneous succession.

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16 Romańczyk M. i inni: Prognoza oddziaływania na środowisko regionalnej polityki miejskiej województwa śląskiego. Centrum Dziedzictwa Przyrody Górnego Śląska. Katowice 2021.
17 Rostański A.: Flora spontaniczna hałd Górnego Śląska. Archiwum Ochrony Środowiska 23/1997.
18 Rostański A.: Rola lokalnych zasobów genowych w zagospodarowaniu nieużytków poprzemysłowych. Materiały Sympozjum „Warsztaty 2000 – Zagrożenia naturalne w górnictwie. Wieliczka, 29 maja-1 czerwca 2001" Druk-Pol, Kraków 2001.
19 Rostański A.: Spontaniczne kształtowanie się pokrywy roślinnej na zwałowiskach po górnictwie węgla kamiennego na Górnym Śląsku. Wydawnictwo Uniwersytetu Śląskiego. Katowice 2006.

Post-mining dumps form a peculiar type of anthropogenic refuge for the organisms that inhabit them and are characterized by their height above the surrounding terrain. They are directly exposed to wind and sun. The chemical and mechanical composition of the heap, as well as the extremely high (in the summer months) and extremely low (in the winter months) temperatures and the water deficits that occur (mainly in the surface layers) largely contribute to the size and type of vegetation cover. Thus, heaps, on the one hand, can be spontaneously "greened" by the growth of plants from wind-borne seeds (which promotes the formation of natural refuges for wildlife), while on the other hand they are undeveloped for many years.

The chemical composition of the soil on the dumps has a significant impact on the development of vegetation. The waste that occurs on the dumps varies in quality, from fresh material to completely weathered material. The rate of degradation of the material is influenced by the physicochemical properties and mechanical composition, which is heavily influenced by the way coal deposits are accessed and exploited and the technology of processing plants. The above makes the subsoil deposited on the dumps have the characteristics of regolith, i.e. poor high skeletal soil with a high proportion of stony and gravelly fractions[20].

The development of vegetation found on heaps is significantly affected by the fragmentation of the soil substrate. It occurs, among other things, in the process of weathering and, unfortunately, covers only the surface layers of the heaps. The dynamic transformations occurring on the surface of the heaps also affect the varying pH of the soil substrate. Despite its low values, there is a further decline as the weathering process continues. The chemical composition of the soil substrate, which can vary even within a single heap, can have a significant impact on the formation of conditions for plant growth and development. Moreover, the total carbon content of the substrate structure is quite high compared to typical soils in Poland, while the nitrogen content is so low that post-mining dump habitats can be classified as trophically poor and not conducive to plant growth[21].

As a result of the above, native species of flora are leading in the species composition of plants taking up residence on the heap areas. Admittedly, reclamation procedures are carried out to facilitate the development of the plant cover, no less, they require maintenance procedures to prevent degeneration and extinction of species from planting. Reclamation is a stage preceding the proper development of heaps, but it is associated with the financial costs of the ultimate development of such areas. Initially, reclamation treatments, which are a stage preceding proper

[20] Skarzyńska K. M.: Odpady powęglowe i ich zastosowanie w inżynierii lądowej i wodnej. Akademia Rolnicza. Kraków 1997.
[21] Rostański A.: Spontaniczne kształtowanie się pokrywy roślinnej na zwałowiskach po górnictwie węgla kamiennego na Górnym Śląsku. Wydawnictwo Uniwersytetu Śląskiego. Katowice 2006.

development, greatly facilitate the development of plant cover. However, it requires constant maintenance treatments. Otherwise, it will degenerate, and the planted species will slowly recede, and this will significantly delay the processes of spontaneous plant succession[21].

Several directions for the management of reclaimed post-mining dumps are currently under consideration. One of them is afforestation. This is a relatively inexpensive and widely used method. However, it requires constant care and supervision in the initial period. Another challenge is that the formation time for this type of management is long - the full effect is achieved only after several generations of the main components of the stand have been replaced and the undergrowth and undergrowth have stabilized[22]. The second direction could be agricultural development, but due to the occurrence of abandonment and fallowing of large areas of farmland, this direction seems unlikely. The third direction is recreational or communal development. Although it represents a positive public perception, it requires sizable initial outlays (planting of non-native plants) and ongoing costs to keep the land in proper condition. All of these treatments are cost-intensive and place a significant burden on the budgets of cities and municipalities in areas where heaps are located. This is the reason that often these areas remain as a land reserve for future investment[23]. Thus, for economic reasons, these sites are most often left without any development with colonization of spontaneous vegetation forming a dense and permanent cover.

With the above in mind, the target development of post-mining wastelands should be preceded by a study of the existing natural conditions and the most complete possible assessment of the possibilities and limitations associated with their planned transformation. It should be borne in mind that the specified direction of development must take into account actions extended in time, such as the strategy for stimulating the development of particular types of vegetation developing in specific environmental conditions (physicochemical parameters of the substrate)[24]. Thus, it is expedient to carry out reclamation of post-mining dumps (heaps), where the work performed should allow restoration of areas degraded by mining activities to a state in which they can be reused for natural, agricultural, recreational, residential or other purposes, in accordance with social and environmental needs. Post-mining dumps, which are landfills for waste generated by mineral extraction, often pose a major aesthetic and environmental problem.

[22] Gieburowski W.: Ocena rekultywacji zwałowiska odpadów pogórniczych „Wiesia III" w nadleśnictwie Katowice. Katedra Ekologii Lasu, Wydział Leśny, Akademia Rolnicza im. H. Kołłątaja. Kraków 1999.
[23] Rostański K. M.: Sanacja nieużytków miejskich metodą kreowania sukcesji roślin. Odnowa krajobrazu miejskiego ULAR. Politechnika Śląska. Gliwice 2005.
[24] Rostański A.: Spontaniczne kształtowanie się pokrywy roślinnej na zwałowiskach po górnictwie węgla kamiennego na Górnym Śląsku. Wydawnictwo Uniwersytetu Śląskiego. Katowice 2006.

The reclamation process, due to the many years of negative impact on the environment, and the landscape, is time-consuming. It is also cost-intensive, and must include, among other things:

- site survey - attention should be paid to the location of the dump and the chemical composition of the topsoil, soil condition, or hydrological conditions;
- determining the purpose and direction of reclamation - only green areas, use for recreation, or housing;
- if necessary, reinforce slopes and escarpments - protection against wind and water erosion, which can be associated with plantings to help hold the soil in place;
- **Neutralization of possible contaminants – liming;**
- **enrichment of the soil with nutrients - the use of a layer of fertile soil, soil substrate or compost, since heaps often consist of materials poor in the above components, which are necessary for plant growth;**
- **planting of plants adapted to growth in reclaimed conditions;**
- carry out water management works - rainwater and surface water drainage systems, creation of retention basins;
- adaptation of the site to its intended function;
- conducting ecological monitoring - the condition of the reclaimed land.

In terms of nutrient soil enrichment, it is possible to cover the topsoil with bio-waste. These bio-waste, which can be in either dry or wet form, are organic wastes of plant origin.
They come from, among other things:

- households - food scraps, cut grass, leaves, branches;
- food industry;
- agriculture - straw and other vegetable waste;
- wood industry - sawdust and uncontaminated wood waste.

This waste, which undergoes a natural process of biodegradation by microorganisms, is a full-fledged material for reuse in the process of land reclamation. Nutrient-rich compost (formed from bio-waste) will play a role in supporting the process of vegetation growth, and this in turn will contribute to strengthening the surface of post-mining dumps. In addition, the processing of bio-waste at least into compost affects the reduction of landfill waste. On them, methane is produced in the process of anaerobic decomposition of organic matter. Protection of soil and groundwater from contamination are other advantages of bio-waste processing.

4.2. Seeds and bio-waste mixtures essential for technology

Based on the analysis of post-mining heap management options, scenarios and guidelines were developed for the preparation of the mixture of bio-waste and plant seeds necessary for the soil reclamation technology under development at WP5.

4.2.1. Spoil mixture

Reclamation of post-mining dumps by introducing vegetation requires taking care of favorable conditions for their growth. In this case, the main factor is the quality of the topsoil covering the surface of the heap. Often these are particles of sand and organic material carried by the wind. However, on “young” dumps there may be largely only material from mining production. The quality of such material is poor and often not conducive to plant growth. It is a fact that over time a layer of humus can form on the surface of the heap (from plants that have managed to establish themselves in the area), but this is a long process and does not guarantee success. This may happen, for example, due to the possibility of water leaching (rainfall) or wind blowing away the forming humus layer. Therefore, measures should be taken to promote the formation of layers friendly to the development of plant cover. For this purpose, compost can be used, which is produced by companies that dispose of bio material. Compost, diluted with water, can be mixed with very “poor” soil. Such a compost mixture should be spread mechanically (by spraying) on the surface of the heap and properly hydrated, mainly to prevent it from blowing away. Another method can be the application to very “poor” soil of natural liquid material (manure, slurry) or obtained from sewage treatment plants. Of course, it must be a properly prepared material that does not contain toxic components. In this case, the liquid material is applied to the soil surface in the form of a spray. What will be important is the amount of compost or liquid material applied to create a substrate friendly to the subsequent greening of the heap area.

The technology, which is to be responsible for fertilizing post-mining dump sites, will use a spray system. It requires the use of a liquid mixture of bio-waste with a density of water or diluted compost that is sprayable. After the liquid mixture is prepared in the preparation system, it will be applied to the land surface through special nozzles.

Scenario I

In view of the above, in scenario one, it is proposed to use manure, which is a natural nitrogen and potassium fertilizer (fermented urine, almost completely devoid of phosphorus, contains on average up to 3% dry matter, 0.3-0.6% N, 0.68-0.83% K and less than 0.04% P, the amount of manure used should be 10 to 20 m^3/ha, before application should be diluted with water in a ratio of 1:1 to 1:4[25]), or slurry (liquid, fermented mixture of excrement (feces and urine) of livestock and water, possibly with an admixture of unused feed, coming from litter-free barns[26]).

Scenario II

Scenario two proposes the use of bio-waste (biodegradable waste from gardens and parks, food and kitchen waste from households, restaurants, caterers, retail units, as well as comparable waste from establishments that produce or market food). Bio-waste should be composted and mixed with water in a ratio of, for example, 1:4 (bio/water). Fig. 26 and Fig. 27 show the organic material to be composted, which, after processing, will become a valuable material to enrich the ground layer for plant seeding and an industrial compost heap.

Fig. 26. Organic material for composting[27]

Fig. 27. Industrial heap composting plant[28]

[25] https://pl.wikipedia.org/wiki/Gnoj%C3%B3wka (access: 30-04-2024)
[26] https://pl.wikipedia.org/wiki/Gnojowica (access: 30-04-2024)
[27] https://prezero.pl/kompost/ (access: 27-05-2024)
[28] http://ekodolina.pl/o-eko-dolinie/instalacje-zakladu-technologia/kompostownia-pryzmowa/ (access: 27-05-2024)

Manure and slurry are offered by agricultural producers. Their ads can be found on popular online platforms. Fig. 28 shows the fertilization of a meadow with manure / slurry.

Fig. 28. Fertilizing a meadow with manure / slurry [29]

4.2.2. Plant seeds

It was assumed that post-mining dumps would be seeded. Accordingly, on the basis of the literature analysis and by conducting our own observations, seeds of more than a dozen plant species were selected. These seeds can come from purchases at commercial outlets, as well as can be taken from existing sites located on the heaps. Seeds from purchase will be characterized by homogeneity in terms of species. Accordingly, provision should be made for the creation of seed mixtures for sowing. This approach will result in the formation of biodiversity in the area subjected to sowing.

Seeds from the harvest will be characterized by species diversity. Brought by the wind, they have already formed multi-species plant carpets, trying to establish themselves on the surface of the heap.

It is proposed (due to the habitat conditions on the slopes of the heaps) to use seeds of plants with low and medium moisture requirements.

These may include:

- various species of grasses, such as:
    - carpet bentgrass,
    - tall oatgrass,

[29] https://www.agrofakt.pl/gnojowica-i-gnojowka-dawka/ (access: 27-05-2024)

  - hungarian brome,
  - cat grass,
  - reed fescue, red fescue, meadow fescue, sheep's fescue,
  - perennial ryegrass,
  - reed canary grass,
  - timothy,
  - meadowgrass,
- birdsfoot trefoil,
- lucerne,
- white melilot,
- various species of clover,
- sainfoin,
- white mustard.

In accordance with the above, the following scenarios are proposed for the use of seeds in the process of sowing post-mining dumps.

Scenario I

In scenario one, it is proposed to use only grass seeds (mentioned above), which tolerate well the conditions of strong sunlight and little water. The following photo (Fig. 29) shows grass seed, while the next one (Fig. 30) shows the popular meadow timothy.

Fig. 29. Grass seed[30]

[30] https://sprzedajemy.pl/nasiona-trawy-wieloletnej-kupkowka-2023-wysylka-janow-lubelski-4-e30984-nr59628143 (access: 27-05-2024)

Fig. 30. Meadow timothy[31]

Scenario II

The second scenario, based on previous research work carried out by GIG (RECOVERY), would be based on the use of a mixture of grass seed and other plants, such as: lucerne, white melilot and various species of clover. A more species-diverse mix would make fuller use of the soil and environmental conditions of the sown area. The photo below (Fig. 31) shows the reclaimed - as part of the RECOVERY project - area of a part of the post-mining dump.

Fig. 31. Reclaimed area of part of the post-mining dump[32]

[31] https://ogrodowapasja.blog/2016/08/30/pelen-traw-pelen-turzyc/ (access: 27-05-2024)
[32] https://gig.eu/pl/newsy/podsumowanie-projektu-recovery (access: 27-05-2024)

Scenario III

Scenario three proposes the use of seeds of roadside flowers and perennials that form so-called flower meadows, adapted to grow in very difficult conditions (the longest droughts, the greatest weed pressure, the poorest soil and the weakest care possible). Plants that should do well in this case include: cornflowers, toadflax, chamomile, plantain, tansy, poppy (flowers), common yarrow, common daisy, borage, purple coneflower, St. John's wort, oregano, lovage, lemon balm, blue phacelia, sage, thyme (perennials).

The proposed roadside flowers and perennials are a strong pro-environmental element, allowing to use for the environment areas degraded by man. Many of them repel mosquitoes or nematodes, while providing an invaluable benefit for bees, for example. In the years to come, the above plants will spread extensively, as well as reproduce through roots and sundry plants. This will allow rapid colonization of the reclaimed area..

The recommended sowing density, in each scenario, is 2 to 3 g/m^2.

Grasses, flowers and perennials are offered by many online retailers, while the photos (Fig. 32 and Fig. 33) show single and perennial plants for sowing in dry and very dry areas.

Fig. 32. Flower meadow of roadside plants[33]

[33] https://semini.pl/produkt/kwiaty-przydrozne-nasiona/ (access: 27-05-2024)

Fig. 33. Perennial plants for dry areas [34]

[34] https://lakikwietne.pl/produkty/marki/beewild/na-suche-tereny-niska-wieloletnia/ (access: 27-05-2024)

5. Summary

Mine waste dumps resulting from mining activities pose many environmental hazards, including fires and the associated emission of gases that pollute the atmosphere (carbon monoxide and dioxide), as well as contamination of surface and groundwater through the leaching of chlorides, sulphates and heavy metals from dumps. At the same time, dumps can and should be treated as secondary deposits of useful materials. The use of the materials deposited there will reduce the consumption of primary raw materials and is in line with the principle of the circular economy.

The recovery of useful materials and subsequent reclamation of dumps creates the possibility of developing areas that, as a result of the above-mentioned hazards, seemed to be lost to the environment and impossible for other use.

The results of the analyses of mine waste obtained from the selected dumps indicate that it is possible and justified to use the waste for the main purposes assumed in this project. These are the production of hydrogen from the combustible fraction and geopolymers using the mineral fraction. It was observed that the separation method between combustible matter rich and mineral matter rich can apply the observed differences in particles size distribution and density. It is worth to underscore that the highest net calorific value after separation was in range of 30 MJ/kg. Nevertheless, the combustion or/and gasification performance will be examined in the next stages of this project. The combustion stability of this fraction will be strongly influenced by the voilatile matter content. One of the possible way of heat management in this process is the thermal processing (roasting) of the mineral part before geopolymers production.

Density analyses of the samples showed that the tested materials contained a significant amount of combustible fraction grains, which after its separation should be subjected to gasification and then hydrogenation.

In turn, the high share of silicon and aluminium in the tested samples indicates the possibility of using the waste for the preparation of geopolymer materials with high-added value. Low calcium content of tested coal gangues, as well as adsorptive properties of geopolymer matrices, make geopolymer membranes suitable, low-cost and more environmental-friendly solutions for acid mine drainages treatment.

The material obtained from selected of raw mine waste is not useful directly in road construction, in mechanically stabilized base layers or in concrete (additionally, it is related to the significant carbon content causing the concrete to swell). In conditions of protection against water and frost, these wastes can be used:

- as materials for improvement of irresponsible basic and auxiliary foundations, among others pedestrian and road routes,
- as materials for the construction of embankments, as well as:
- for recultivation works,
- as spacer material used in municipal waste landfills and for biological reclamation,
- as aggregates for the sub-base, binding and wearing course of roads, for the surface layer of asphalt concrete.

It is important to determine the carbon content in the waste, which will be the subject of further work. On the basis of waste, it is also possible to produce artificial aggregates (covering the waste with fly ash), which have a wider application than mine waste without refining. The possibilities of additional management of waste from coal enrichment will be the subject of research in the next stage of the project.