



Report on characterization of primary and secondary raw materials (initial)

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TABLE OF CONTENTS

1. PURPOSE	7
1.1 EXECUTIVE SUMMARY	7
2. ROM CHARACTERIZATION.....	8
2.1 CLC ORE.....	8
2.2 KGHM ORE	11
2.3 SOMINCOR ORE	13
3. CONCENTRATES CHARACTERIZATION	14
3.1 CLC POLYMETALLIC CONCENTRATE	14
3.2 BOR POLYMETALLIC MATERIAL	18
3.3 KGHM POLYMETALLIC CONCENTRATE.....	23
3.4 SOMINCOR POLYMETALLIC CONCENTRATE.....	28
3.5 BOR PYRITE CONCENTRATE	32
3.6 CLC PYRITE TAILINGS.....	35
3.7 SOMINCOR PYRITE CONCENTRATE	38
4. SUMMARY	42

LIST OF FIGURES

- Fig. 1.** The overview of CLC deposit and sample collection.
- Fig. 2.** Images of metallographic microscope of CLC ore.
- Fig. 3.** Images of metallographic microscope of KGHM low grade ore.
- Fig. 4.** XRD pattern of CLC polymetallic concentrate (IMNR).
- Fig. 5.** Images of metallographic microscope of CLC polymetallic concentrate.
- Fig. 6.** Images of metallographic microscope of CLC bulk concentrate.
- Fig. 7.** Particle size distribution of CLC polymetallic concentrate.
- Fig. 8.** The overview of Bobija mine and sampling method that was used.
- Fig. 9.** Mechanical tests of the polymetallic ore from BOR: *a* – drilling sample; *b* –press analysis.
- Fig. 10.** XRD pattern of BOR polymetallic material.
- Fig. 11.** Images of metallographic microscope of BOR polymetallic ore.
- Fig. 12.** Particle size distribution obtained using laser particle size analyzer.
- Fig. 13.** XRD pattern for KGHM low grade concentrate.
- Fig. 14.** Images of metallographic microscope of KGHM low grade concentrate.
- Fig. 15.** Images of metallographic microscope of KGHM low grade concentrate.
- Fig. 16.** Particle size distribution of KGHM low grade concentrate.
- Fig. 17.** XRD pattern of SOMINCOR polymetallic concentrate.
- Fig. 18.** Images of metallographic microscope of SOMINCOR polymetallic concentrate.
- Fig. 19.** Images of metallographic microscope of Somincor sample.
- Fig. 20.** Particle size distribution of SOMINCOR polymetallic concentrate.
- Fig. 21.** XRD pattern of BOR pyrite concentrate (BOR INST).
- Fig. 22.** Images of metallographic microscope of BOR pyrite concentrate.
- Fig. 23.** Grain size distribution obtained using laser particle analyzer.
- Fig. 24.** XRD pattern of CLC pyrite concentrate.
- Fig. 25.** Images of metallographic microscope of CLC pyrite tailings.
- Fig. 26.** Particle size distribution of CLC pyrite tailings.
- Fig. 27.** XRD pattern of Somincor pyrite tailings.
- Fig. 28.** Elements distribution within selected area using magnification 400x.
- Fig. 29.** Quantitative analysis using EDS method for previously selected points.
- Fig. 30.** Particle size distribution of Somincor pyrite tailings.

LIST OF TABLES

Table 1. ELEMENTAL COMPOSITION OF CLC ORE, [%]
Table 2. MINERALOGY OF CLC ORE, [%]
Table 3. ELEMENTAL COMPOSITION OF KGHM LOW GRADE ORE, [%]
Table 4. MINERALOGY OF KGHM LOW GRADE ORE
Table 5. MINERALOGY OF KGHM LOW GRADE ORE, [%]
Table 6. ELEMENTAL SOMINCOR POLYMETALLIC ORE, [%]
Table 7. ELEMENTAL COMPOSITION OF CLC POLYMETALLIC CONCENTRATE, [%]
Table 8. XRD RESULTS OF CLC POLYMETALLIC CONCENTRATE
Table 9. MINERALOGY OF CLC POLYMETALLIC CONCENTRATE, [%]
Table 10. GRAIN SIZE DISTRIBUTION OF CLC POLYMETALLIC CONCENTRATE
Table 11. ELEMENTAL COMPOSITION OF BOR POLYMETALLIC MATERIAL, [%]
Table 12. MECHANICAL CHARACTERISTIC OF BOR POLYMETALLIC MATERIAL
Table 13. XRD RESULTS OF BOR POLYMETALLIC MATERIAL
Table 14. MINERALOGY OF BOR POLYMETALLIC MATERIAL, [%]
Table 15. GRAIN SIZE DISTRIBUTION OF BOR POLYMETALLIC MATERIAL
Table 16. ELEMENTAL COMPOSITION OF KGHM LOW GRADE CONCENTRATE, [%]
Table 17. XRD RESULTS OF KGHM LOW GRADE CONCENTRATE
Table 18. MINERALOGY OF KGHM LOW GRADE CONCENTRATE, [%]
Table 19. GRAIN SIZE DISTRIBUTION OF KGHM LOW GRADE CONCENTRATE
Table 20. ELEMENTAL COMPOSITION OF SOMINCOR POLYMETALLIC CONCENTRATE, [%]
Table 21. XRD RESULTS OF SOMINCOR POLYMETALLIC CONCENTRATE
Table 22. MINERALOGY OF SOMINCOR POLYMETALLIC CONCENTRATE, [%]
Table 23. GRAIN SIZE DISTRIBUTION OF SOMINCOR POLYMETALLIC CONCENTRATE
Table 24. ELEMENTAL COMPOSITION OF BOR PYRITE CONCENTRATE, [%]
Table 25. XRD RESULTS OF BOR PYRITE CONCENTRATE
Table 26. MINERALOGY OF BOR PYRITE CONCENTRATE, [%]
Table 27. GRAIN SIZE DISTRIBUTION OF BOR PIRYTE CONCENTRATE
Table 28. ELEMENTAL COMPOSITION OF CLC PYRITE CONCENTRATE, [%]
Table 29. XRD RESULTS OF CLC PYRITE CONCENTRATE
Table 30. MINERALOGY OF CLC PYRITE CONCENTRATE, [%]
Table 31. GRAIN SIZE DISTRIBUTION OF CLC PYRITE TAILINGS
Table 32. ELEMENTAL COMPOSITION OF SOMINCOR PYRITE TAILINGS, [%]

ABBREVIATIONS AND ACRONYMS

XRF	X-ray Fluorescence
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrometer
ICP-MS	inductively coupled plasma – mass spectrometry
FAAS	Flame Atomic Absorption Spectroscopy
py	Pyrite
sl	Sphalerite
cp	Chalcopyrite
cv	Covellite
gn	Galena
td	Tetrahedrite
eng	Enargite
cc	Chalcocite
bn	Bornite
arp	Arsenopyrite
CRM	Critical Raw Materials
SME	Small Medium Enterprise
RIR	Reference Intensity Ratio
ROM	Run Of Mine

1. PURPOSE

1.1 EXECUTIVE SUMMARY

The WP 1 is the first part of the IntMet project with an objective to collect and characterize primary and secondary raw materials to be studied and tested. The primary raw materials include ores, concentrates and middlings and these were provided by mining partners i.e. (i) polymetallic ores and concentrates by SOMINCOR, CLC and BOR INST; (ii) complex or low-grade ores, concentrates and middlings by KGHM and BOR INST. The secondary raw materials include flotation tailings and metallurgical wastes that will be supplied by the mining partners. This work package was divided into four tasks concerning the following objectives:

- definition, collection and characterization of primary raw materials,
- definition, collection and characterization of secondary raw materials such as flotation tailings and metallurgical wastes,
- collection and characterization of materials and intermediate by-products or final products generated in the various technology developments,
- control and management of the package and delivery of primary and secondary raw materials samples to be provided for testing under WP2, 3, 4, 5 and 6.

Task 1.1 deals with ores, concentrates and middlings samples collection and characterisation". It was assumed to select, collect and characterize primary and secondary raw materials samples, provided for laboratory and pilot plant test works by mining companies. The following materials are considered:

- polymetallic ores, concentrates and tailings: SOMINCOR, CLC, BOR INST
- complex or low-grade copper ores, concentrates, middlings: KGHM, BOR INST.

Physical, chemical, mineralogical and microscopic analysis, which were chosen to fully characterize investigated materials, allowed to review the existing geological information and flotation plants databases. It was suggested in the project proposal to use the following methods analyzing the chemistry of primary materials:

chemical analysis of major and minor metals - carried out using various techniques including: XRF, ICP-OES, ICP-MS, fire-assay; XRD, optical microscopy, optical mineralogy, automated quantitative mineralogy, electron microprobe analysis (EMPA) and metallographic microscopy - used to determine ore texture and quantitatively evaluate particular minerals amount as well as to investigate mineral geochemistry and compositional maps.

2. ROM CHARACTERIZATION

2.1 CLC ORE

The contribution of CLC in defining and characterizing task was to collect polymetallic ore and performed required analysis of this material. The overview of deposit as well as view of the sample were presented in **figure 1**.



Fig. 1. The overview of CLC deposit and sample collection.

Three different methodologies were used to evaluate the elemental composition of this material using inductively coupled plasma spectrometer. Namely, in the ME-ICPORE method material was subjected to highly oxidizing conditions of HNO_3 , KClO_3 and HBr and then the solution was treated with aqua regia; in ME-ICP81x, material was fused with sodium peroxide at 700°C ; in ME-ICP41 sample decomposition was achieved by digestion in an aqua regia. These methods were complementary, as for example ME-ICPORE was not able to analyze refractory metals while ME-ICP41 was not appropriate for mercury. Results of elemental analysis, prepared by CLC, in weight % were presented in **table 1** (data with asterik were given in ppm).

Table 1. ELEMENTAL COMPOSITION OF CLC ORE, [%]

Method	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Cd	Hg	Mo	S	Ag	Sb	Co
ME-ICPORE	0.989	39.5	0.59	2.42	3.35	0.444	0.046	0.029	0.006	79*	<0.001	47.6	57*	0.086	0.031
ME-ICP81x	0.98	40.9	0.58	2.37	2.97	0.45	0.04	–	–	–	–	48.9	–	–	0.036
ME-ICP41	8970*	26.2	0.57	>10000*	>10000	2960*	289*	214*	55.6*	66*	8*	>10	49.7*	546*	301*
Method	Al	B	Ba	Be	Cr	Ga	K	La	Mg	Na	Ni	P	Sc	Se	Sr
ME-ICPORE	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
ME-ICP81x	0.14	–	–	–	–	–	–	–	–	–	–	–	–	–	–
ME-ICP41	0.01	10*	20*	<0.5*	3*	<10*	<0.01	<10*	0.01	0.02	29*	<10*	<1*	<10*	16*
Method	Th	Ti	Tl	U	V	W	–	–	–	–	–	–	–	–	–
ME-ICPORE	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
ME-ICP81x	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
ME-ICP41	<20*	<0.01	60*	10*	3*	10*									

* – values in ppm

These results showed that precision of elements detection was very high. Some rarely analyzed elements were also placed in the table to show completeness of used techniques. The mineralogical examination combined with quantitative analysis showed that material was composed mainly of pyrite and primary sulfides, present in polymineral fragments associated with each other and pyrite, while minor sulfides were observed in lesser extent. The precised data were presented in **table 2**.

Table 2. MINERALOGY OF CLC ORE, [%]

Sample	MINERAL									
	Pyrite	Sphalerite	Galena	Chalcocite	Chalcopyrite	Covellite	Bornite	Tetrahedrite	Enargite	
HPP<2mm	84.67	11.1	2.41	0	1	0.35	0	0.35	0.12	

Particular minerals were identified in samples using metallographic microscope. Images were presented in **figure 2**.

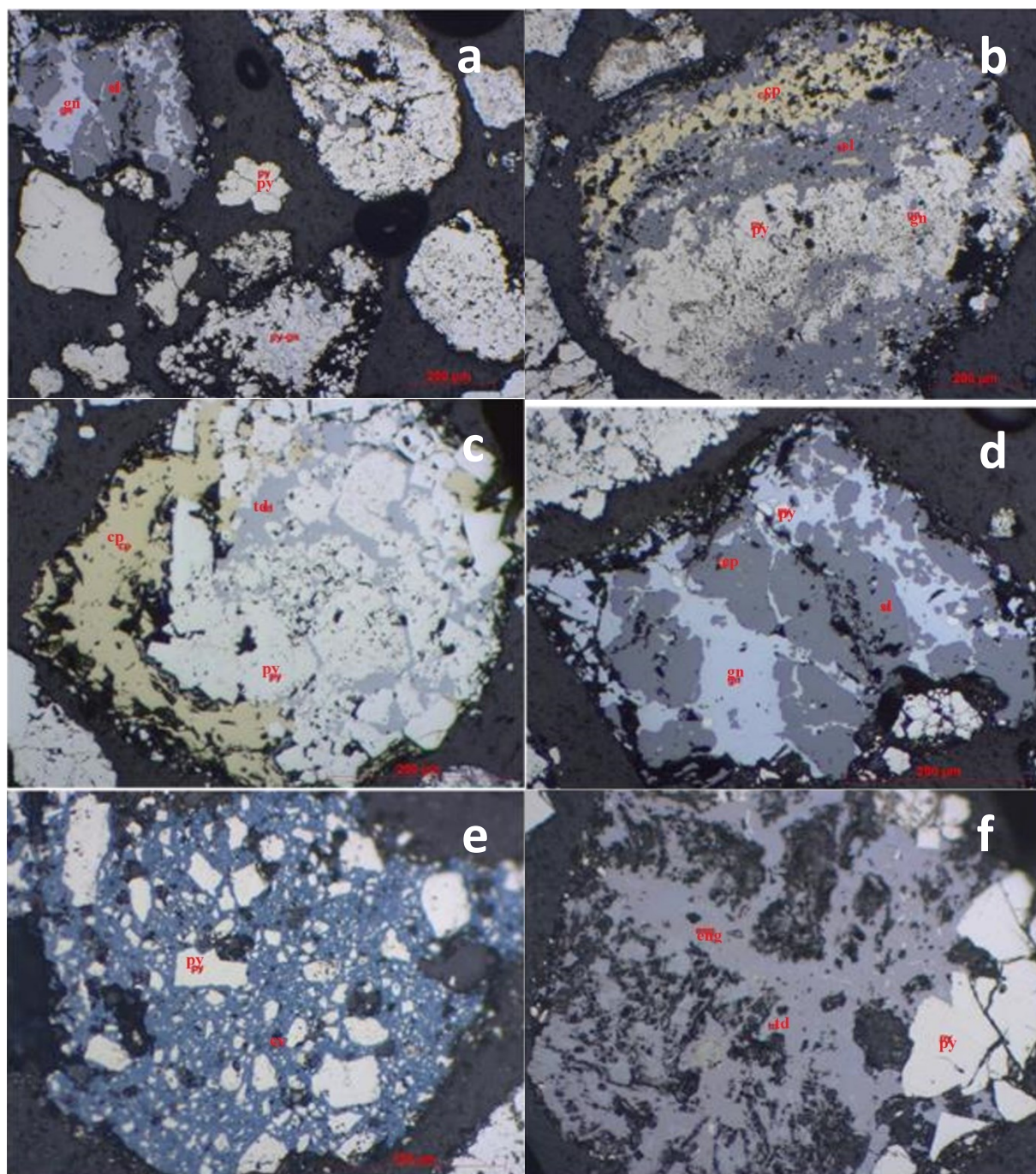


Fig. 2. Images of metallographic microscope of CLC ore: *a* – big particle size of pyrite (py) with subhedral-anhedral and primary-secondary sulphides; *b* – primary sulphide with pyrite (py), sphalerite (sl) and chalcopyrite (cp) in a minor presence of galena (gn); *c* – polymetallic particle with pyrite (py), tetrahedrite (td) and chalcopyrite (cp); *d* – polymineralline fragment of primary mineralization composed of galena (gn) and sphalerite (sl), with chalcopyrite relics/residues; *e* – detail of polymineralline fragment composed of pyrite (py) and covellite (cv); *f* – detail of fragment composed of tetrahedrite (td) that has been massively replaced by enargite (eng).

Summarizing, the CLC ore was composed mainly of pyrite and primary sulphides. Sulphides were associated with each other in polymineral grains and then with pyrite. The particle size was lower than 2mm. There were apparent signs of corrosion observed within samples.

2.2 KGHM ORE

KGHM ore was collected from "Lubin-Małomice" deposit, which is located in the north-west part of the Lower Silesian Voivodeship, Poland. It is a stratoidal type deposit with copper-bearing materials present in sandstone of rotliegend and thill part of Zechstein. It is a seam (layered) deposit, locally discontinuous.

KGHM "Lubin-Małomice" ore consists of sulfide minerals with the following coexisting metals: silver, lead, zinc, cobalt, molybdenum, nickel and vanadium. These are present as their own minerals or isomorphic additives to copper-bearing minerals. In case of lead its content within carbonate-shale ore can be higher than for copper. The average concentration of lead is 0.12%, it is located mainly in galena as well as in copper-containing minerals. An increased amount of zinc is rarely observed and actually ranges from 0.00 to 0.05 %. Zinc is mostly present as sphalerite. Cobalt is represented by cobaltite and smaltite. These minerals are present in form of grains, which can be found in bornite and chalcocite or separately in shales. Molybdenum is located as isomorphic additive in pyrite, chalcopyrite and bornite, sometimes can be found in its own minerals. Vanadium can be found in shales and carbonates that are not making their own minerals and its main concentrations are combined with organic matter.

The elemental composition of KGHM low grade ore was presented in a **table 3**.

Table 3. ELEMENTAL COMPOSITION OF KGHM LOW GRADE ORE, [%]

Sample	Cu	Pb	Zn	As	Ni	V	Bi	Cd	Hg	Mo	Re	Ag	Sb	Co
KGHM	1.19	0.16	<0.05	0.11	0.004	0.0056	0.0008	0.0026	0.0002	0.011	0.00015	0.0057	0.0046	0.011

The density of the material was separately determined for three fractions i.e. carbonate ore, shale ore and sandstone ore, resulting in 2.6 t/m³, 2.5 t/m³ and 2.3 t/m³, respectively. It should be emphasized that the sandstone ore is dominating within the investigated area with a content ca. 58%, while for carbonate ore and shale ore this is about 27% and 15.2%, respectively.

Qualitative and quantitative evaluation of minerals identified within the ore were presented in **table 4** and **5**.

Table 4. MINERALOGY OF KGHM LOW GRADE ORE

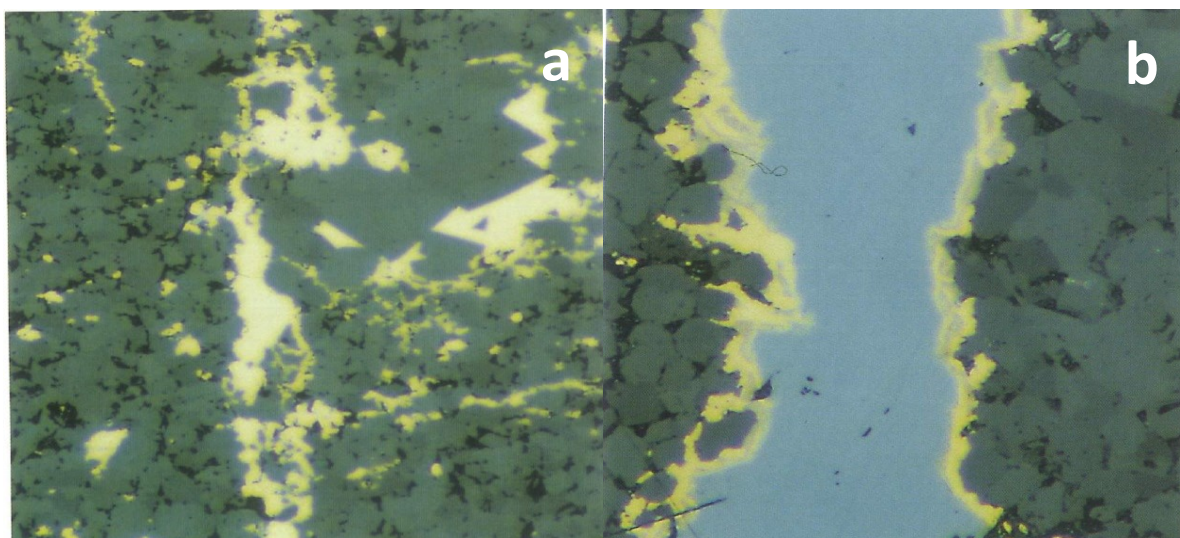
	MINERAL										
Sample	Pyrite	Quartz	Sphalerite	Chalcocite	Chalcopyrite	Galena	Bornite	Enargite	Dolomite	Calcite	Shale
KGHM	FeS ₂	SiO ₂	ZnS	Cu ₂ S	CuFeS ₂	PbS	Cu ₅ Fe ₂ S ₄	Cu ₃ AsS ₄	CaMg (CO ₃) ₂	CaCO ₃	KAlSiCaMgO

The full mineralogical analysis performed by KGHM is composed of 60 minerals. Because of table transparency, only the 11 most common were presented. The presence of tetrahedrite, barite, sanidine, andalusite and rutile was found in much lower amount.

Table 5. MINERALOGY OF KGHM LOW GRADE ORE, [%]

	MINERAL										
Sample	Pyrite	Quartz	Sphalerite	Chalcocite	Chalcopyrite	Galena	Bornite	Enargite	Dolomite	Calcite	Shale
KGHM	0.46	34.21	0.27	0.26	0.85	0.73	0.61	0.08	36.25	7.00	15.26

Microscopic images of the investigated ore were presented in **figure 3**.



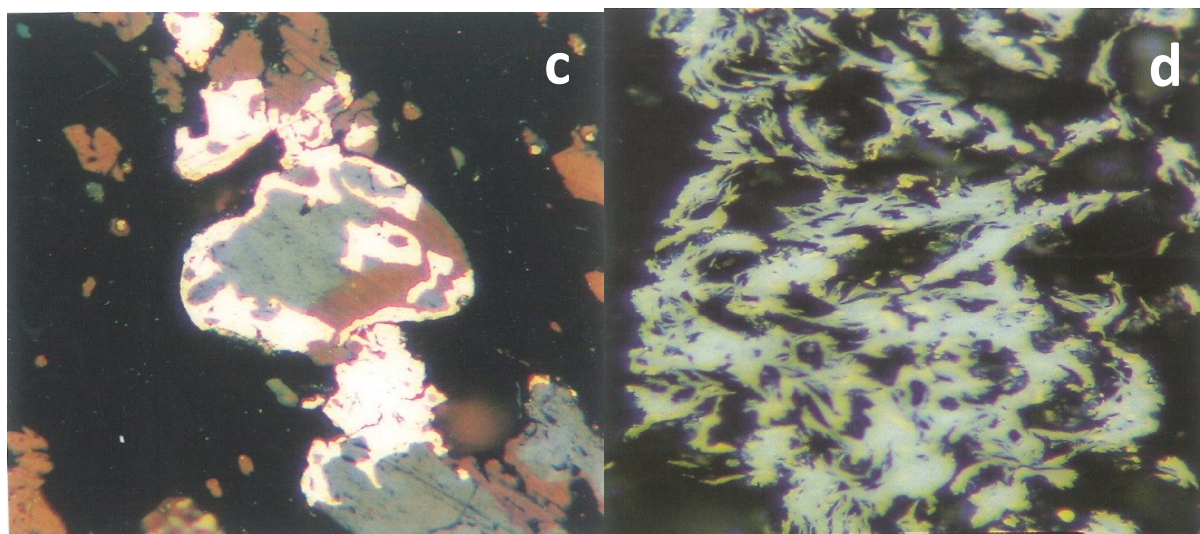


Fig. 3. Images of metallographic microscope of KGHM low grade ore, in reflected light: *a* – chalcopryite (yellow) in a dolomite; *b* – combination of sphalerite (bluish) with chalcopryite (yellow) and bornite (grey); *c* – native silver (white) with stromeyerite (grey), jalpaite (pink-olive) and bornite (dark lilac); *d* – agglomeration of hypidomorphic castaingite (creamy) within epigenetic veins.

2.3 SOMINCOR ORE

Elemental composition of Somincor ROM was presented in **table 6**.

Table 6. ELEMENTAL COMPOSITION OF SOMINCOR POLYMETALLIC ORE, [%]

Method	Cu	Fe	Pb	Zn	As	Bi	Cd	Hg	Mo	Ni	Cr	V	Ag	Sb	Co
Somincor	0.64	-	1.517	6.59	0.347	0.005	0.016	-	0.002	0.002	0.002	0.005	0.006	0.052	0.021

The mineralogy of the sample is very common from Iberian Pyritic Belt:

Pyrite Dominant mineral. Abundant anhedral, subhedral and angular grains. Liberated and locked with (primarily) sphalerite, chalcopryite and galena.

Sphalerite Locked with (primarily) pyrite, to a lesser extent gangue and rarely galena and chalcopryite. Not seen as fully liberated grains, more as pseudomorphic intergrowths and infill within pyrite grain fractures. Subhedral and anhedral grain shapes.

Chalcopryite Locked and liberated grains with (primarily) pyrite, occasionally sphalerite and galena. Anhedral grains shapes with interstitial infilling of pyrite and gangue.

Galena Locked within pyrite, usually in association with sphalerite and to a lesser extent chalcopryite. Not seen as fully liberated grains, more as pseudomorphic intergrowths and infill within pyrite grain fractures

3. CONCENTRATES CHARACTERIZATION

3.1 CLC POLYMETALLIC CONCENTRATE

Material labeled as Bulk PMS4 was a CLC polymetallic concentrate that was analyzed within this task (CLC). Material was produced in a batch mode from a CLC ore. The elemental analysis was done for this sample according to the previously suggested ME-ICPORE methodology (CLC). Data were presented in **table 7**. The results were confirmed by IMNR studies, but with some differences due to IMNR sample correspond to obtained pilot plant bulk concentrate sample.

Table 7. ELEMENTAL COMPOSITION OF CLC POLYMETALLIC CONCENTRATE, [%]

Sample	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Cd	Hg	Mo	S	Ag	Sb	Co
CLC	1.8	37.9	0.2	3.6	5.8	0.1	0.036	0.025	0.004	101*	0.002	47	85*	<0.01	0.055

* – values in ppm

Density of CLC polymetallic concentrate evaluated by IMNR using pycnometric analysis was 4.6295 t/m³.

Qualitative analysis was performed using XRD method with XRD pattern presented in **figure 4**, while list of detected minerals with their structures was presented in **table 8** – data were listed in two rows for analysis obtained from CLC (lab sample) and IMNR (pilot plant sample) .

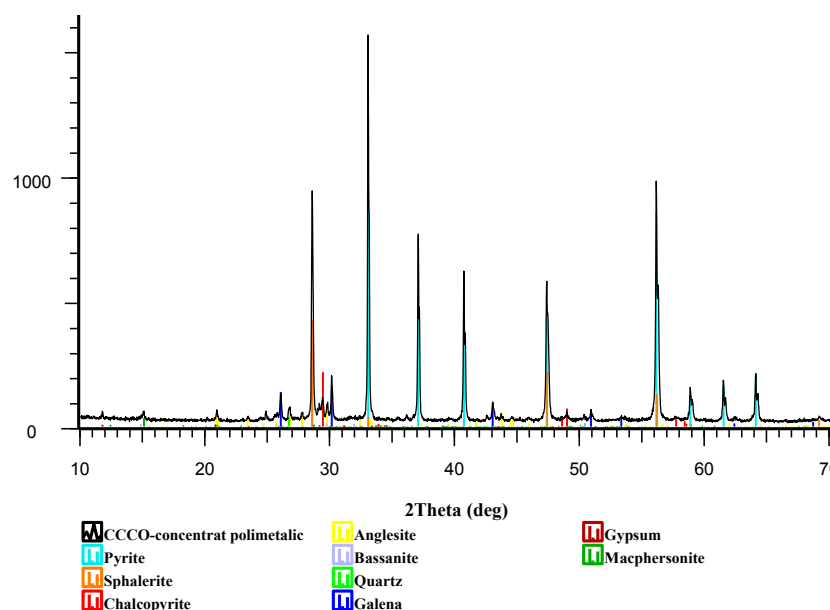


Fig. 4. XRD pattern of CLC polymetallic concentrate (IMNR).

Table 8. XRD RESULTS OF CLC POLYMETALLIC CONCENTRATE

	MINERAL								
Sample	Pyrite	Quartz	Sphalerite	Gypsum	Chalcopyrite	Galena	Anglesite	Bassanite	Macphersonite
CLC (CLC)	FeS ₂	SiO ₂	ZnS	Ca(SO ₄)(H ₂ O) ₂	CuFeS ₂	PbS	PbSO ₄	MoS ₂	Pb ₄ (SO ₄)(CO ₃) ₂ (OH) ₂
Sample	Pyrite	Bornite	Sphalerite	Chalcocite	Chalcopyrite	Galena	Covellite	Enargite	Tetrahedrite
CLC (IMNR)	FeS ₂	Cu ₅ FeS ₄	ZnS	Cu ₂ S	CuFeS ₂	PbS	CuS	Cu ₃ AsS ₄	(Cu,Fe) ₁₂ Sb ₄ S ₁₃

Quantitative evaluation of identified minerals, obtained by CLC and IMNR – by means of RIR (Reference Intensity Ratio) method, was presented in **table 9**.

Table 9. MINERALOGY OF CLC POLYMETALLIC CONCENTRATE, [%]

	MINERAL								
Sample	Pyrite	Quartz	Sphalerite	Gypsum	Chalcopyrite	Galena	Anglesite	Bassanite	Macphersonite
CLC (CLC)	78	2	8	1	5	2	2	2	<1
Sample	Pyrite	Bornite	Sphalerite	Chalcocite	Chalcopyrite	Galena	Covellite	Enargite	Tetrahedrite
CLC (IMNR)	82.59	0	6.46	0.54	6.28	1.08	0.72	1.08	1.26

Microscopic study in reflected light of the analysed sample highlighted the following minerals (listed in order of frequency): pyrite – FeS₂; sphalerite – ZnS; galena – PbS; chalcopyrite – CuFeS₂ and covellite CuS.

Sample examined in reflected light was mainly composed of pyrite as grains with dimensions from less than 1 micrometer up to hundreds of micrometers. Pyrite occurred mostly as individual crystals, but also was associated with other minerals present in the sample. Sphalerite and galena were present as associated, while chalcopyrite was observed as independent crystals or associated with the sphalerite and pyrite.

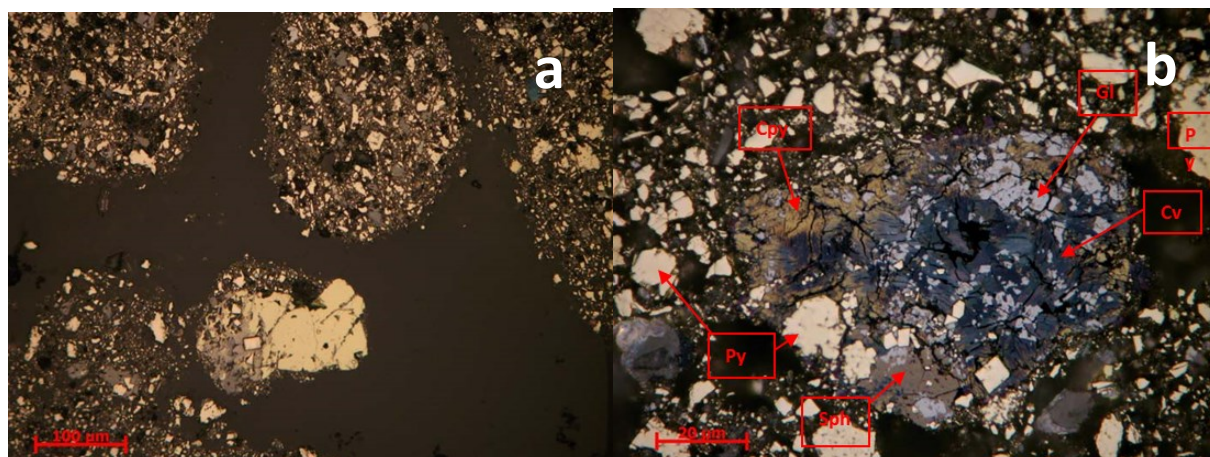
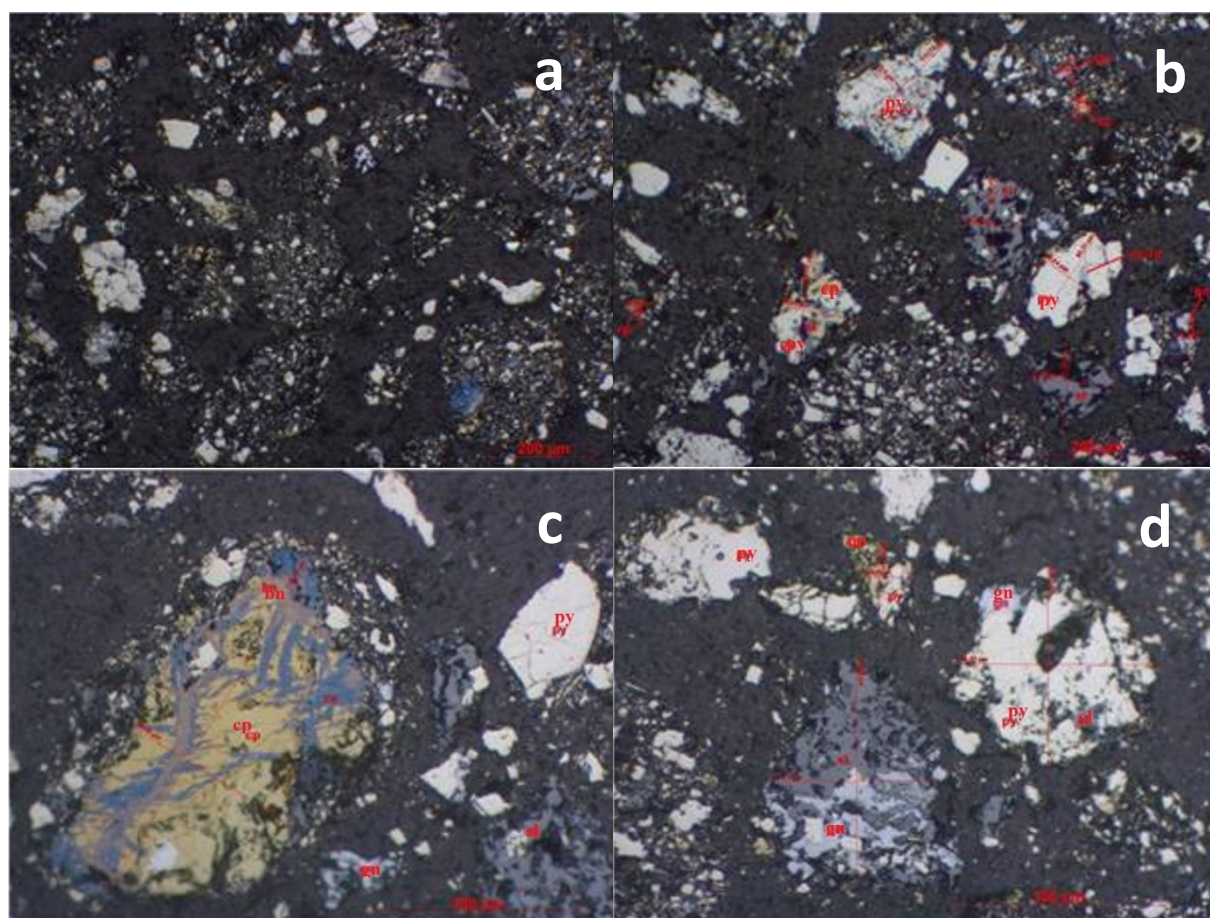


Fig. 5. Images of metallographic microscope of CLC polymetallic concentrate, in reflected light: a – with pyrite (py), sphalerite (sph), chalcopyrite (cpy) and covellite (cv) and in transmitted light: b – with pyrite (py), sphalerite (sph), galena (gl), chalcopyrite (cpy) and covellite (cv).

Comparatively, images obtained from metallographic microscope by CLC were presented in **figure 6**.



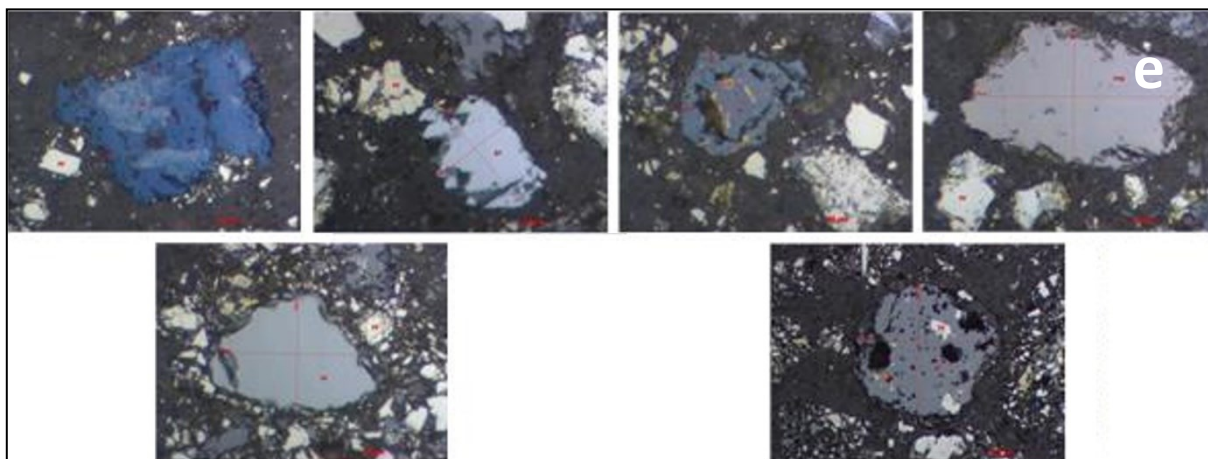


Fig. 6. Images of metallographic microscope of CLC bulk concentrate PMS4: *a* – general appearance of the sample consisting mainly of pyrite (py) euhedral-anhedral and both primary and secondary sulphides; *b* – various mono- and polymineralic grains located within specimen in the following combinations: pyrite (py)–chalcopyrite (cp) – sphalerite (sl), pyrite (py)–tetrahedrite (td)–chalcopyrite(cp), pyrite (py)–covellite (cv), galena (gn), chalcopyrite (cp), sphalerite (sl); *c* – mixed fragments composed of chalcopyrite (cp) partially replaced by bornite (bn) and covellite (cv); *d* – polymineralic fragments in form of: pyrite (py)–chalcopyrite (cp) and pyrite (py)–galena (gn) – sphalerite (sl); *e* – monomineral fragments of covellite (cv), galena (gn), tetrahedrite (td), chalcocite (cc), enargite (eng) and sphalerite (sl).

Results of grain size analysis performed by IMNR were presented in **table 10** and in **figure 7**.

Table 10. GRAIN SIZE DISTRIBUTION OF CLC POLYMETALLIC CONCENTRATE			
Size class, mm	m, %	Undersize, %	Oversize, %
$d > 0.150$	1.80	100.00	1.80
$0.100 < d < 0.150$	1.43	98.20	3.23
$0.090 < d < 0.100$	0.80	96.77	4.02
$0.071 < d < 0.090$	1.80	95.98	5.82
$0.063 < d < 0.071$	0.78	94.18	6.60
$0.040 < d < 0.063$	7.63	93.40	14.23
$0.032 < d < 0.040$	6.54	85.77	20.77
$0.025 < d < 0.032$	14.05	79,23	34.82
$d < 0.025$	65.18	65.18	100.00

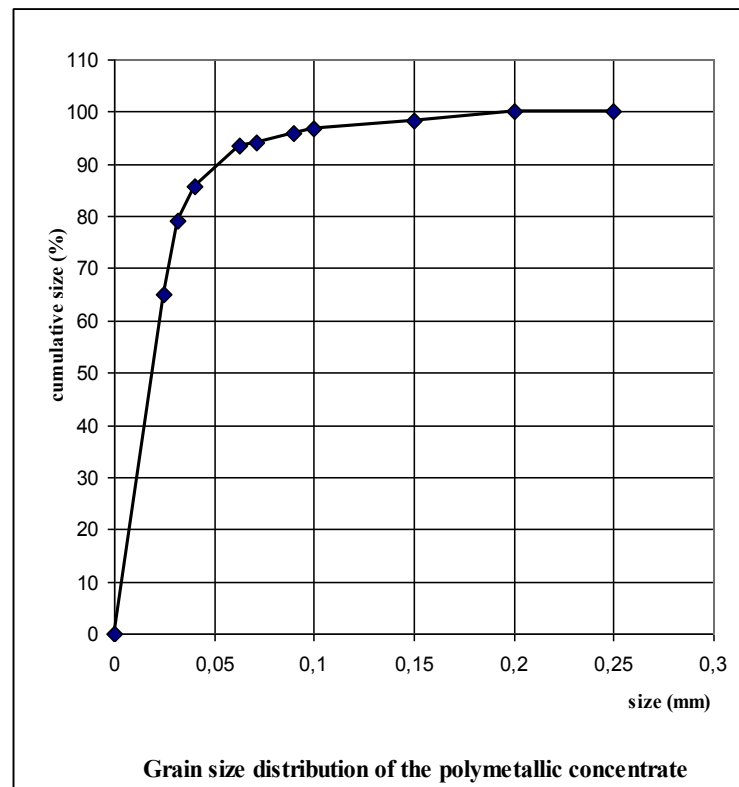


Fig. 7. Particle size distribution of CLC polymetallic concentrate.

3.2 BOR POLYMETALLIC MATERIAL

Polymetallic ore supplied by BOR INST was collected from the open pit of Bobija mines – West Serbia. The entire sample was ground to the size class of – 12.5 mm, homogenized by the cone-ring method and ball-milled to the size of 3.35 mm. As prepared material was used in further chemical and physical analysis.



Fig. 8. The overview of Bobija mine and sampling method that was used.

Table 11. ELEMENTAL COMPOSITION OF BOR POLYMETALLIC MATERIAL, [%]

Sample	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Cd	Hg	Mo	S	Ag	Sb	Co
BOR material	1.77	23.8	0.08	4.62	4.92	0.309	0.037	0.006	0.01	587*	0.007	31.9	120*	0.169	0.001

Results of elemental composition analyzed using unified ME-ICPORE methodology (CLC) were consistent with BOR INST analysis performed using combination of ICP-AES, FAAS (Flame Atomic Absorption Spectroscopy), XRF (X-ray Fluorescence), gravimetry, fire assay and carbon and sulfur analysis as well as with IMNR results obtained for this material. Analysis performed by BOR INST also allowed to estimate ppm of Mo, Ni, Se, Sn and Ti.

Additionally, starting material was drilled for mechanical investigation. This part was performed in a Laboratory for Mechanical Rocks at the Mining and Metallurgy Institute Bor (**figure 9**). These experiments were performed according to the national standards, given in brackets:

- determination of the bulk density with pores and cavities (SRPS B.B7.113)
- determination of the uniaxial compressive strength (SRPS B.B7.126)
- determination of the moisture content (SRPS B.B7.111)
- determination of the bulk density without pores and cavities (SRPS B.B7.112)
- determination of the tensile strength by indirect method (SRPS B.B7.127)

Results were presented in **table 12**.

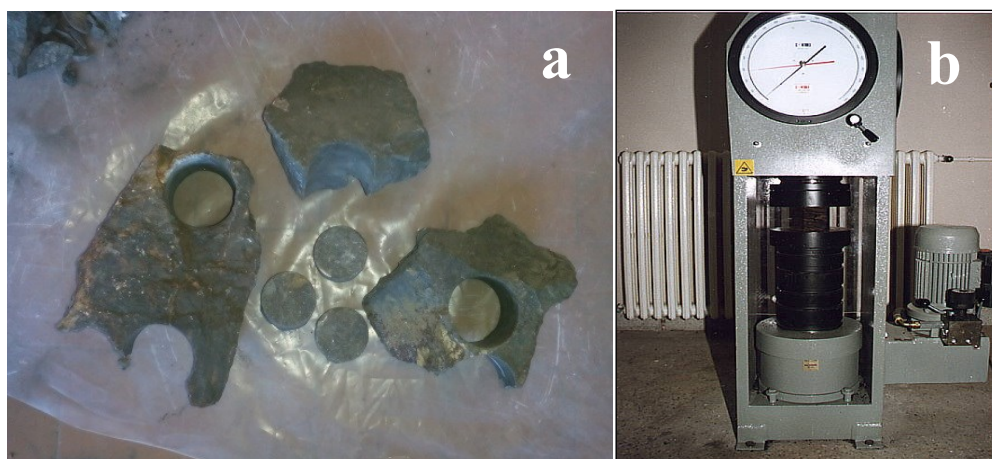


Fig. 9. Mechanical tests of the polymetallic ore from BOR: *a* – drilling sample; *b* –press analysis.

Table 12. MECHANICAL CHARACTERISTIC OF BOR POLYMETALLIC MATERIAL					
Sample	Moisture w [%]	Density γ_z [g/cm ³]	Specific mass γ_s [g/cm ³]	Compressive strength σ_p [MPa]	Tensile strength σ_z [MPa]
1	0.55	4.670	4.778	68.37	10.80
2	0.51	4.761	4.792	89.24	6.00
3	0.53	4.752	4.770	60.65	7.45
Mean value	0.53	4.728	4.780	72.75	8.08

Density presented by BOR INST was in accordance with IMNR results that obtained 4.547 t/m³ using pycnometer method. On the contrary, the bulk density of ore of size -12.7mm was calculated to 2.531 t/m³ while for class 3.35 mm it was 2.619 t/m³.

The qualitative analysis performed by BOR INST for this material was done using X-ray powder diffraction. The XRD pattern was presented in **figure 10** and analysis results in **table 13**.

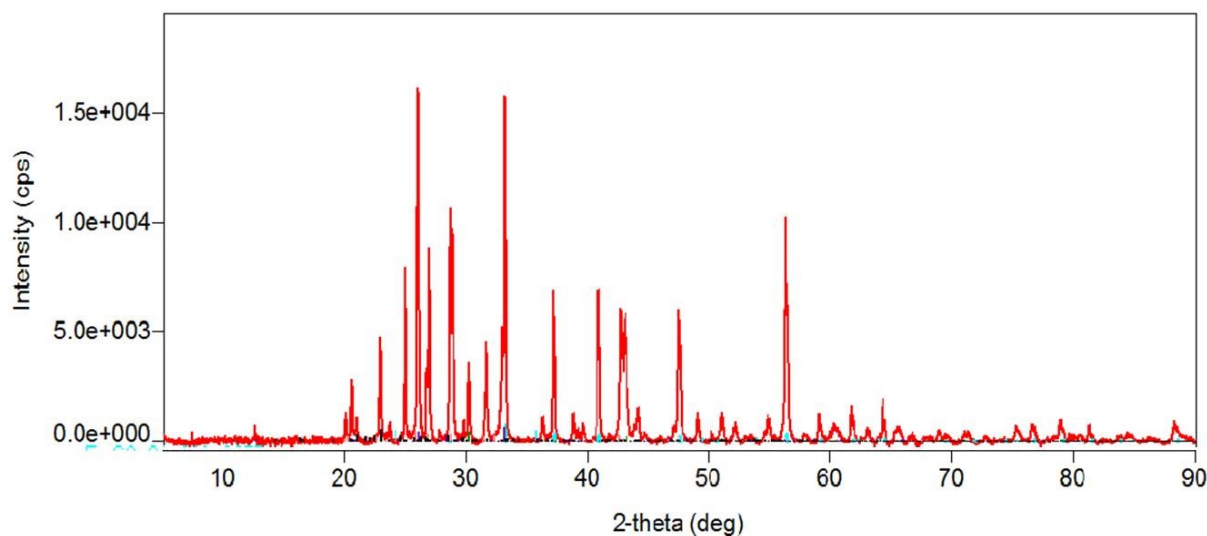


Fig. 10. XRD pattern of BOR polymetallic material.

Table 13. XRD RESULTS OF BOR POLYMETALLIC MATERIAL									
	MINERAL								
Sample	Pyrite	Sphalerite	Galena, syn	alpha-SiO ₂	Zeolite A, (K, Zn)	Hematite	Barite	Trechmannite	beta-S
BOR	FeS ₂	ZnS	PbS	SiO ₂	Zn ₅ K ₂ Al ₁₂	Fe ₂ O ₃	BaSO ₄	AgAsS ₂	S

With respect to these data, the XRD analysis performed at IMNR detected also cubanite, graphite, ramsbeckite and molybdenite.

The qualitative mineralogical analysis was performed using polarizing microscope for reflected and transmitted light brand "JENAPOL-U", company Carl Zeiss-Jena. The material was composed of pyrite, sphalerite, galena, tetrahedrite, chalcopryrite, covellite, arsenopyrite and gangue minerals, which were *quartz, silicates* and *secondary minerals of Pb and Cu (anglesite, malachite, etc.)*. The content of barite was separated and obtained on the basis of chemical analysis. The quantitative mineralogical analysis allowed to evaluate the content of each mineralogical phase in the ore (**table 14**). The surfaces of tested aggregates were determined using the software package OZARIA v2.5 and system for photomicrography. The RIR (Reference Intensity Ratio) method for minerals analysis, which was performed by the IMNR, showed also presence of cubanite, graphite, ramsbeckite and molybdenite in amount of 3.9 %, 2.4 %, 1.7 % and 0.3 %, respectively.

Table 14. MINERALOGY OF BOR POLYMETALLIC MATERIAL, [%]

	MINERAL								
Sample	Pyrite	Sphalerite	Galena	Chalcopryrite	Covellite	Arsenopyrite	Barite	Tetrahedrite	Waste
BOR	45.15	7.71	7.57	0.04	0.04	<0.01	25.26	3.11	11.13

Based on the mineral microscopic analysis it was showed that polymetallic Bobija deposit was comprised of the following minerals: sulphides (pyrite, sphalerite, wurtzite, galena, arsenopyrite, marcasite, pyrrhotite, chalcopryrite, covellite, chalcocite), sulphosalts (tetrahedrite, tennantite), metals (native silver), non-metallic minerals (barite) and gangue minerals (quartz, chalcedony, carbonates).

Material examined in reflected light showed prevalence of pyrite in form of crystalline aggregates with a size between 1 and hundreds of micrometers. Other minerals usually occurred inside the crystalline aggregates of pyrite, localized within cracks. Sphalerite and galena occurred as independent grains or intercrystalline grains inside of pyrite grains forming domains with dimensions between microns and tens of microns. Covellite appeared as micron size granules, most often associated with sphalerite inside the pyrite granules or independently. Chalcocite and chalcopryrite occurred most often as crystals with sizes below one micrometer up to micrometers, disseminated within crystalline aggregates of pyrite. Micronic crystals of tennantite-tetrahedrite were sometimes observed with chalcocite. Transmitted light of sample immersed in nitrobenzene revealed the abundant presence of barite as crystals with sizes between microns to tens microns.

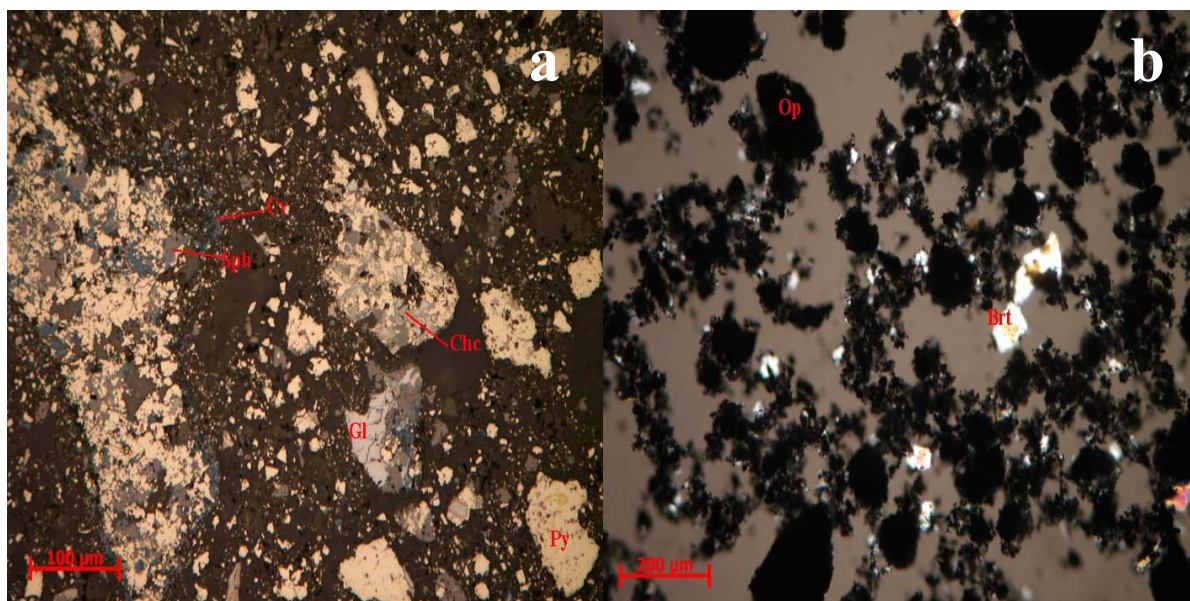


Fig. 11. Images of metallographic microscope of BOR polymetallic ore, in reflected light: *a* – with pyrite (py), sphalerite (sl), covellite (cv), galena (gn), and in transmitted light: *b* – with barite (brt) and opaque minerals (op).

Grain size distribution was analyzed using laser particle size analyzer as well as a series of standard sieves (BOR INST). Results of sieving was presented in **table 15** and laser analysis schematically in **figure 12**. Results obtained using both techniques were the same. The average particle diameter obtained by IMNR was as high as 41.82 microns.

Table 15. GRAIN SIZE DISTRIBUTION OF BOR POLYMETALLIC MATERIAL			
Size class, mm	m, %	Undersize, %	Oversize, %
-0.500+0.425	1.20	100.00	1.20
-0.425+.0300	1.80	98.80	3.00
-0.300+0.212	2.20	97.00	5.20
-0.212+0.150	5.40	94.80	10.60
-0.150+0.106	8.40	89.40	19.00
-0.106+0.075	10.30	81.00	29.30
-0.075+0.053	5.30	70.70	34.60
-0.053+0.038	6.70	65.40	41.30
-0.038	58.70	58.70	100.00

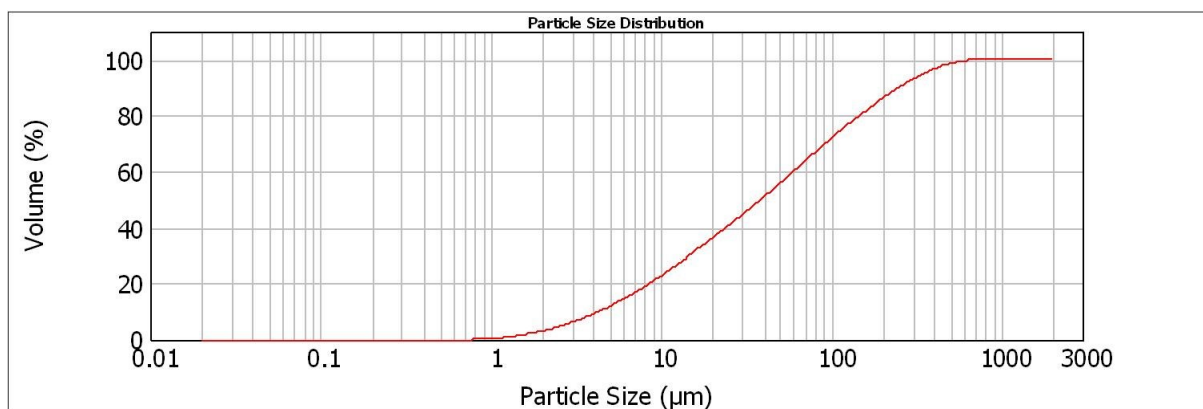


Fig. 12. Particle size distribution obtained using laser particle size analyzer.

3.3 KGHM POLYMETALLIC CONCENTRATE

Elemental composition of KGHM material was determined using previously described ME-ICPORE methodology (CLC), additionally similar results were obtained by IMNR. Data were presented in **table 16**.

Table 16. ELEMENTAL COMPOSITION OF KGHM LOW GRADE CONCENTRATE, [%]

Sample	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Cd	Hg	Si	S	Ag	Sb	Co
KGHM	12,9	8.24	4	4.67	1.12	0.53	0.11	<0.005	0.006	<8*	6,5	13	642*	0.012	0.142

* value in ppm

Density of the material was established by pycnometer method to level 2.8138 t/m^3 (IMNR).

Qualitative analysis of minerals using XRD method and semiquantitative using RIR method, performed at IMNR, were presented in **figure 13** and **table 17** and **18**.

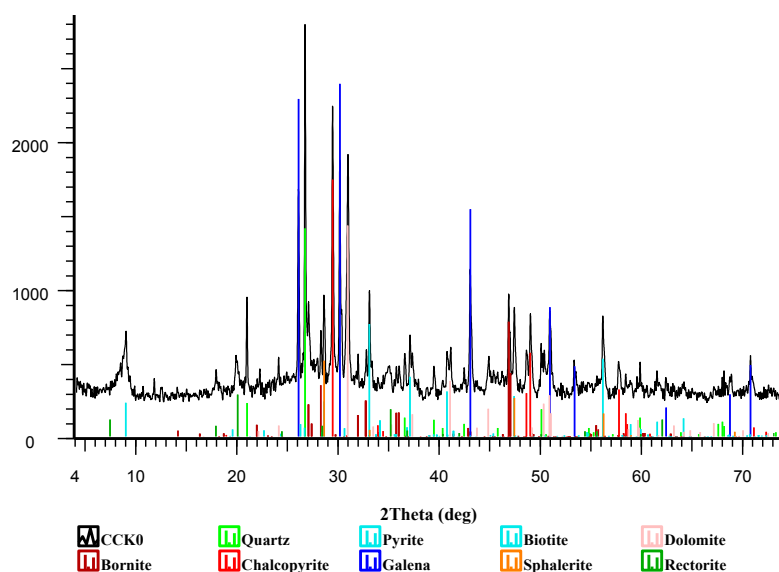


Fig. 13. XRD pattern for KGHM low grade concentrate.

Table 17. XRD RESULTS OF KGHM LOW GRADE CONCENTRATE									
	MINERAL								
Sample	Pyrite	Galena	Sphalerite	Chalcopyrite	Bornite	Dolomite	Rectorite	Biotite	Quartz
KGHM	FeS ₂	PbS	ZnS	CuFeS ₂	Cu ₅ FeS ₄	CaMg(CO ₃) ₂	K _{1.2} Al ₄ Si ₈ O ₂₀ (OH) ₄ ·4H ₂ O	KFeMg ₂ (AlSi ₃ O ₁₀)(OH) ₂	SiO ₂

The semiquantitative analysis obtained using RIR method resulted in content evaluation for identified minerals (**table 18**) – IMNR.

Table 18. MINERALOGY OF KGHM LOW GRADE CONCENTRATE, [%]									
	MINERAL								
Sample	Pyrite	Galena	Sphalerite	Chalcopyrite	Bornite	Dolomite	Rectorite	Biotite	Quartz
KGHM	10	5	2	7	19	19	9	5	14

Microscopic study in reflected and transmitted light highlighted the following minerals (listed in order of frequency): chalcopyrite – CuFeS₂; carbonaceous matter – C; quartz – SiO₂; bornite – Cu₅FeS₄; rhombic chalcocite - Cu₂S; pyrite – FeS₂; carbonate (Ca,Mg)CO₃; sphalerite – ZnS; cubic digenite - Cu₂S; galenite– PbS and covellite.

Material examined in reflected light was mainly composed of chalcopyrite in form of granules with dimensions from less than 1 micrometer up to hundreds of micrometers. Chalcopyrite was associated with pyrite, bornite and digenite. Apart from its associations with chalcopyrite, bornite appeared independently or was associated with digenite. Chalcocite appeared as independent granules or was associated with digenite and bornite. Apart from

the above combinations, digenite occurred rarely in the form of independent granules. Sphalerite and galena were present as independent or associated granules. Sometimes, in the structure of sphalerite chalcopyrite appeared. Covellite, as separated or associated with bornite, was rarely observed. Carbonaceous matter occurred as aggregates, layered, with dimensions of the order of hundreds of microns. These aggregates included micronized sulphur granules. The microscopic study carried out in transmitted light on a sample immersed in nitrobenzene showed the presence of some quartz and carbonate granules, of tens of microns, most often associated with opaque minerals (sulphides).

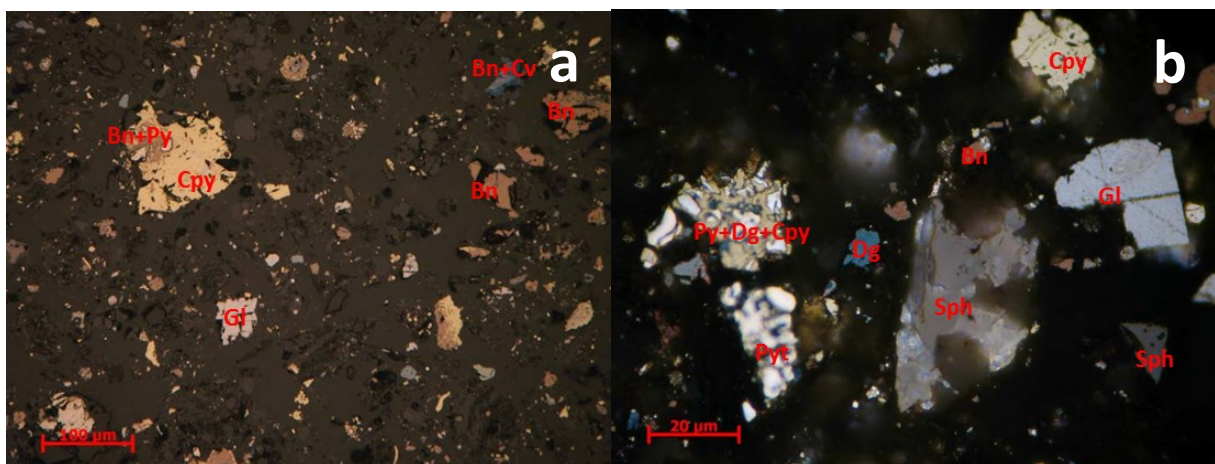
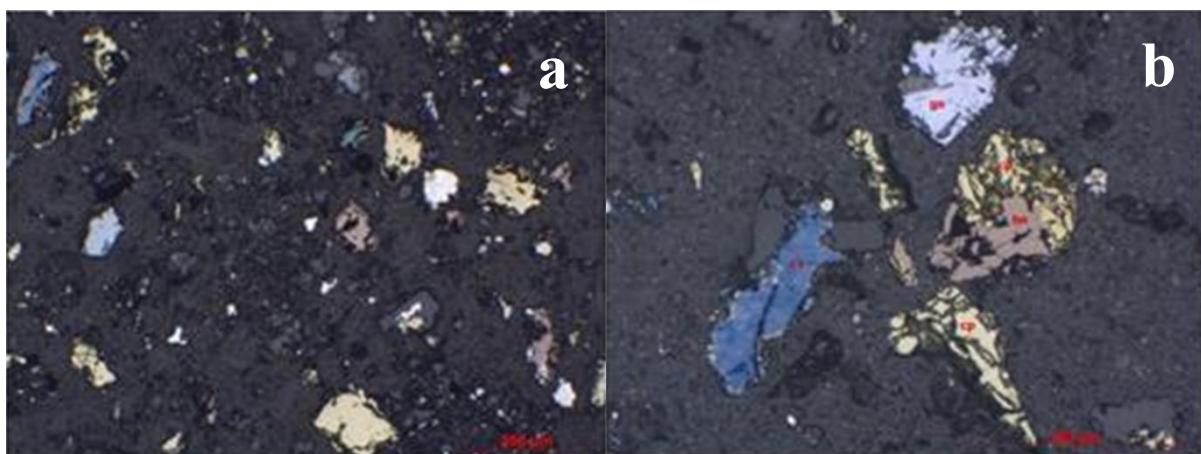


Fig. 14. Images of metallographic microscope of KGHM low grade concentrate, in reflected light: a – with pyrite(py), bornite (bn), covellite (cv), chalcopyrite (cpy) and galena (gl), and in transmitted light: b – with chalcopyrite (cpy), bornite (bn), pyrite (py), digenite (dg), sphalerite (sph) and galena (gl).

Additionally, microscopic images obtained by CLC were comparatively presented in **figure 15**.



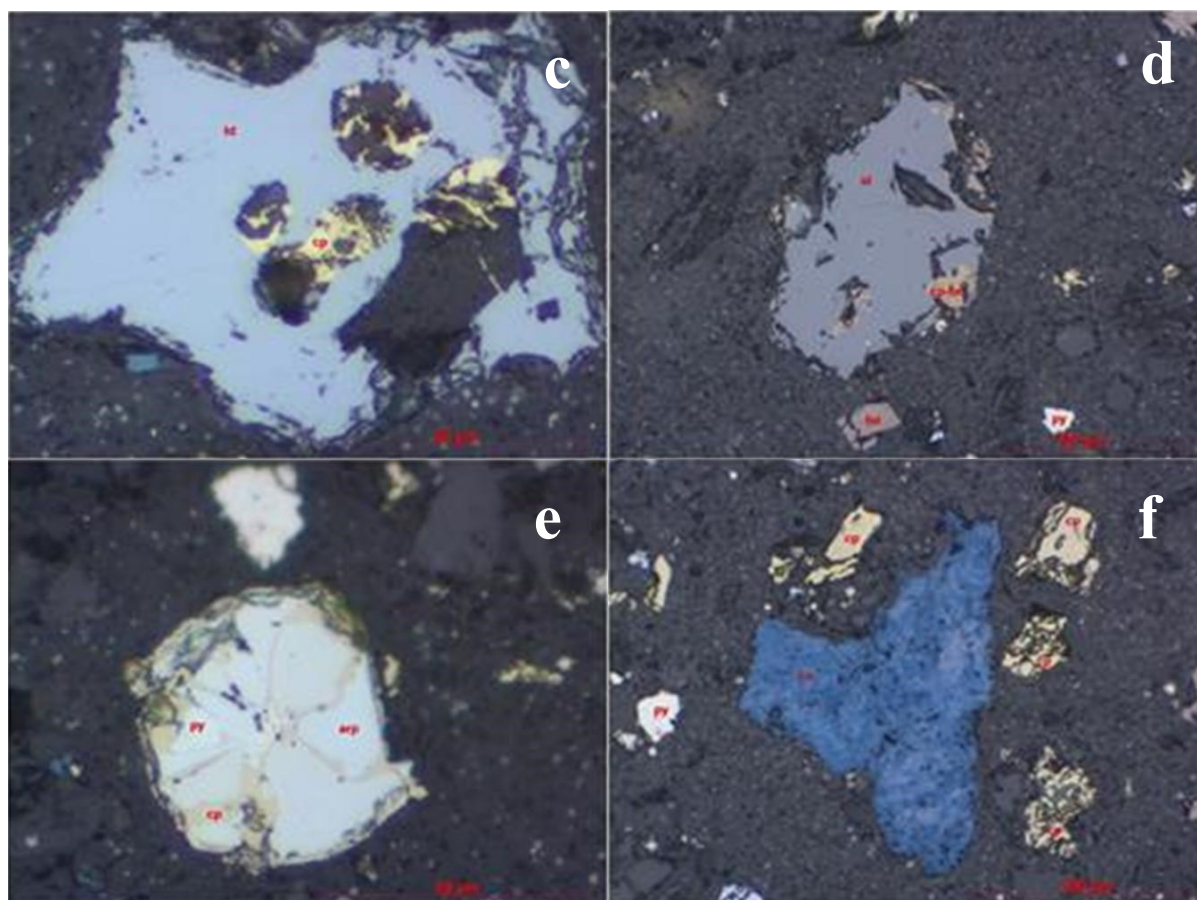


Fig. 15. Images of metallographic microscope of KGHM low grade concentrate: *a* – heterogenous grain size with low pyrite (py) content, primary and secondary copper sulphides present; *b* – free species of galena (gn), chalcopyrite (cp), covellite (cv) and associated species chalcopyrite (cp)-bornite (bn); *c* – primary mineralization composed of tetrahedrite (td) and chalcopyrite (cp); *d* – primary mineralization composed of sphalerite (sl) and chalcopyrite (cp) with bornite inclusions (bn); *e* – primary mineralization composed of pyrite (py), chalcopyrite (cp) and arsenopyrite (arp); *f* – Cu particles: covellite (cv) and chalcopyrite (cp).

Results of particle size distribution determination obtained for KGHM concentrate at IMNR were presented in **table 19** and laser analysis plot in **figure 16**.

Table 19. GRAIN SIZE DISTRIBUTION OF KGHM LOW GRADE CONCENTRATE			
Size class, mm	m, %	Undersize, %	Oversize, %
$d > 0.150$	0.99	100.00	0.99
$0.100 < d < 0.150$	2.98	99.01	3.96
$0.090 < d < 0.100$	6.08	96.04	10.04
$0.071 < d < 0.090$	10.00	89.96	20.04
$0.063 < d < 0.071$	4.21	79.96	24.25
$0.040 < d < 0.063$	20.15	75.75	44.40
$0.032 < d < 0.040$	16.18	55.60	60.58
$0.025 < d < 0.032$	19.37	39.42	79.95
$d < 0.025$	20.05	20.05	100.00

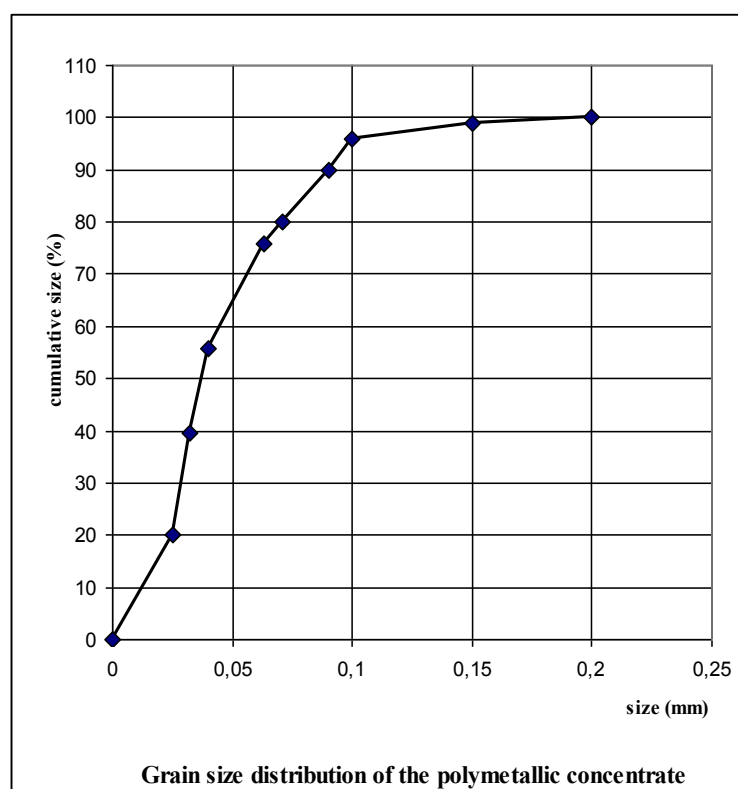


Fig. 16. Particle size distribution of KGHM low grade concentrate

3.4 SOMINCOR POLYMETALLIC CONCENTRATE

Elemental composition of Somincor material was determined using previously described ME-ICPORE methodology. Results, both obtained by CLC and IMNR were very similar, data were presented in **table 20**.

Table 20. ELEMENTAL COMPOSITION OF SOMINCOR POLYMETALLIC CONCENTRATE, [%]

Sample	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Cd	Hg	Si	S	Ag	Sb	Co
SOMINCOR concentrate	5.20	29.9	0.025	14.8	9.10	0.28	0.011	0.015	0.022	0.0081	0.5	40.4	0.0267	0.14	0.011

Density of this material was estimated at 4.7028 t/m³ (IMNR).

Qualitative analysis was done by X-ray powder diffraction method (IMNR). The obtained XRD pattern was presented in **figure 17**, while qualitative evaluation of possible structures in **table 21**.

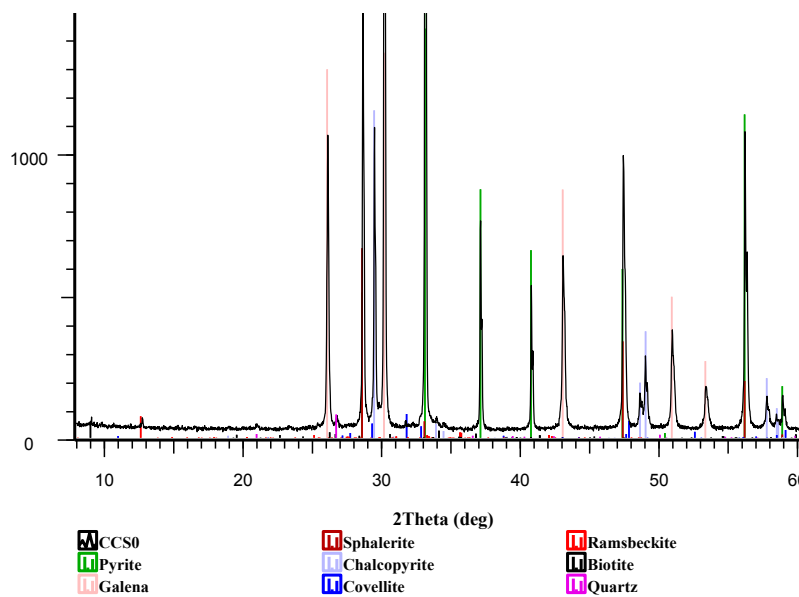


Fig. 17. XRD pattern of SOMINCOR polymetallic concentrate.

Table 21. XRD RESULTS OF SOMINCOR POLYMETALLIC CONCENTRATE

	MINERAL							
Sample	Pyrite	Galena	Sphalerite	Chalcopyrite	Covellite	Ramsbeckite	Biotite	Quartz
SOMINCOR	FeS ₂	PbS	ZnS	CuFeS ₂	CuS	Cu ₁₅ (OH) ₂₂ (SO ₄) ₄ (H ₂ O) ₆	KFeMg ₂ (AlSi ₃ O ₁₀)(O H) ₂	SiO ₂

The semiquantitative analysis obtained using RIR method resulted in content evaluation for identified minerals (table 22) – IMNR.

Table 22. MINERALOGY OF SOMINCOR POLYMETALLIC CONCENTRATE, [%]								
	MINERAL							
Sample	Pyrite	Galena	Sphalerite	Chalcopyrite	Covellite	Ramsbeckite	Biotite	Quartz
SOMINCOR	54	15.4	13.4	13	1.2	1.1	1.0	0.9

Microscopic study (IMNR and CLC) in reflected and transmitted light highlighted the following minerals (in order of frequency): pyrite – FeS_2 ; chalcopyrite – CuFeS_2 ; galena – PbS ; sphalerite – ZnS ; cubanite– CuFe_2S_3 and undetermined transparent minerals (possibly quartz). Sample examined in reflected light was mainly composed of pyrite as grains with dimensions from less than 1 micrometer up to hundreds of micrometers. Pyrite grains similarly to other minerals, were of micrometer dimension – less than micrometer. The other minerals occurred as independent or associated with pyrite. Pyrite grains with low anisotropy were highlighted, characteristic for arseno-pyrite. The study carried out in transmitted light for material immersed in nitrobenzene showed the presence of transparent grains, micrometric – undermicrometric, microscopically indeterminable. Microscopic images were presented in **figure 18**.

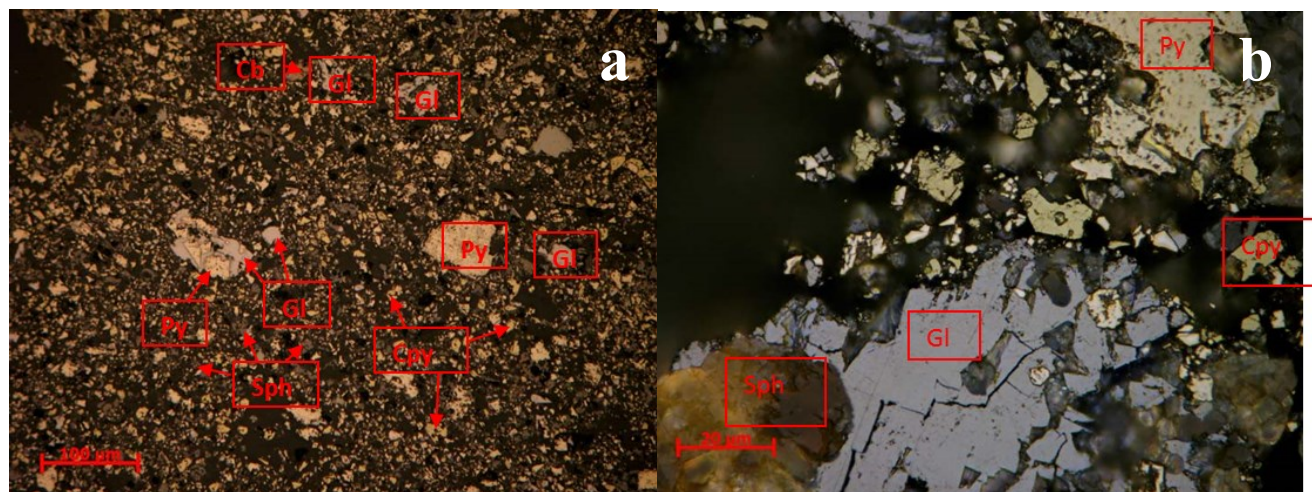


Fig. 18. Images of metallographic microscope of SOMINCOR polymetallic concentrate, in reflected light: *a* – with pyrite (py), sphalerite (sph), galena (gl), chalcopyrite (cpy) and cubanite (cb), and in transmitted light: *b* – with pyrite (py), chalcopyrite (cpy), galena (gl) and sphalerite (sph) with yellow-brown internal reflections.

Mineralogical analysis was performed using metallographic microscope. Images obtained from the microscope were presented in **figure 19**.

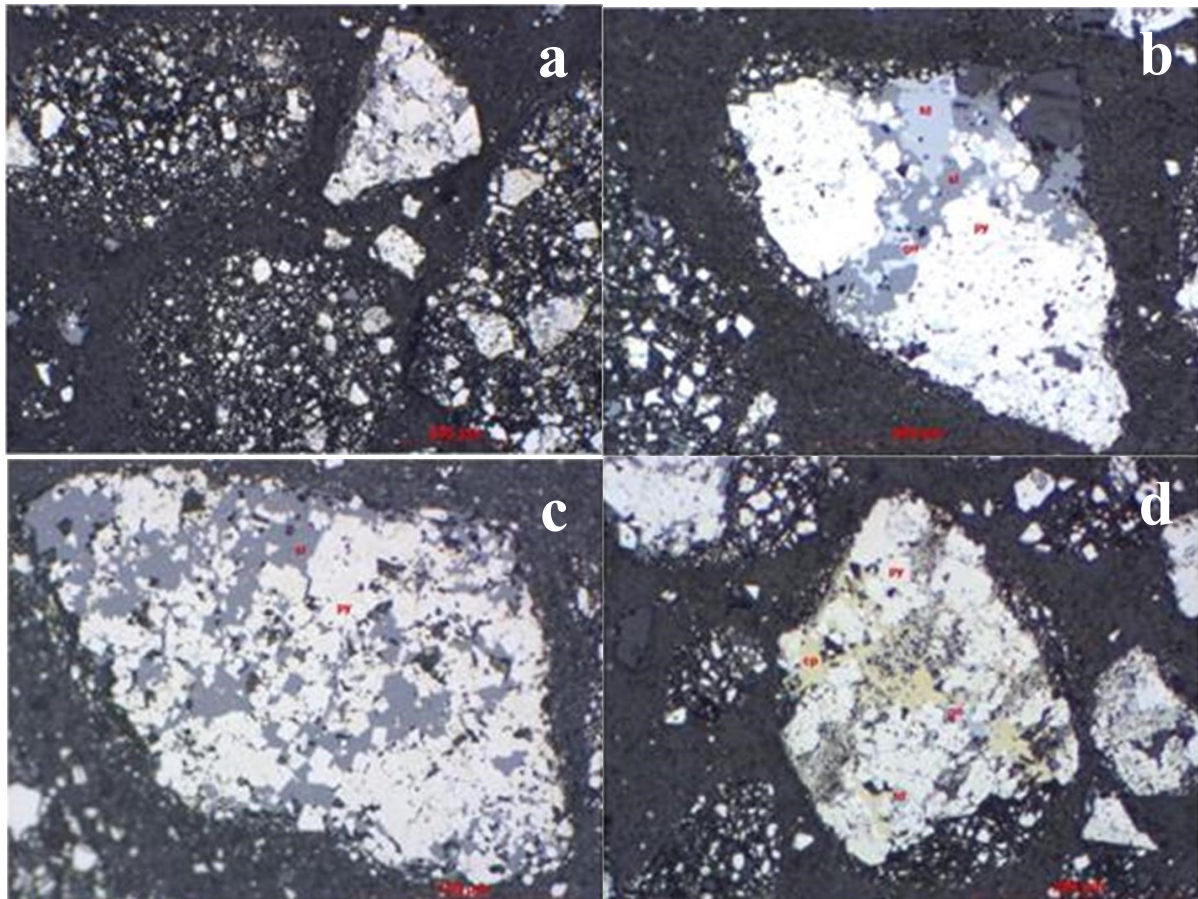


Fig. 19. Images of metallographic microscope of Somincor sample: *a* – heterogenous grain size, pyrite (py) as a main species, high level of primary sulphides associated to pyrite (py); *b* – pyrite (py) detail with primary sulphides, sphalerite (sl), galena (gn) and tetrahedrite (td); *c* – pyrite (py) particle with sphalerite (sl); *d* – primary sulphide particle containing pyrite (py), galena (gn) and chalcopyrite (cp).

Results of grain size analysis performed by IMNR were presented in **table 23** and in **figure 20**.

Table 23. GRAIN SIZE DISTRIBUTION OF SOMINCOR POLYMETALLIC CONCENTRATE			
Size class, mm	m, %	Undersize, %	Oversize, %
d > 0.150	0.57	100.00	0.57
0.100 < d < 0.150	0.57	99.43	1.14
0.090 < d < 0.100	0.37	98.86	1.52
0.071 < d < 0.090	0.62	98.48	2.13
0.063 < d < 0.071	0.60	97.87	2.73
0.040 < d < 0.063	4.76	97.27	7,50
0.032 < d < 0.040	4.46	92.50	11.95
0.025 < d < 0.032	23.36	88,05	35.31
d < 0.025	64.69	64.69	100.00

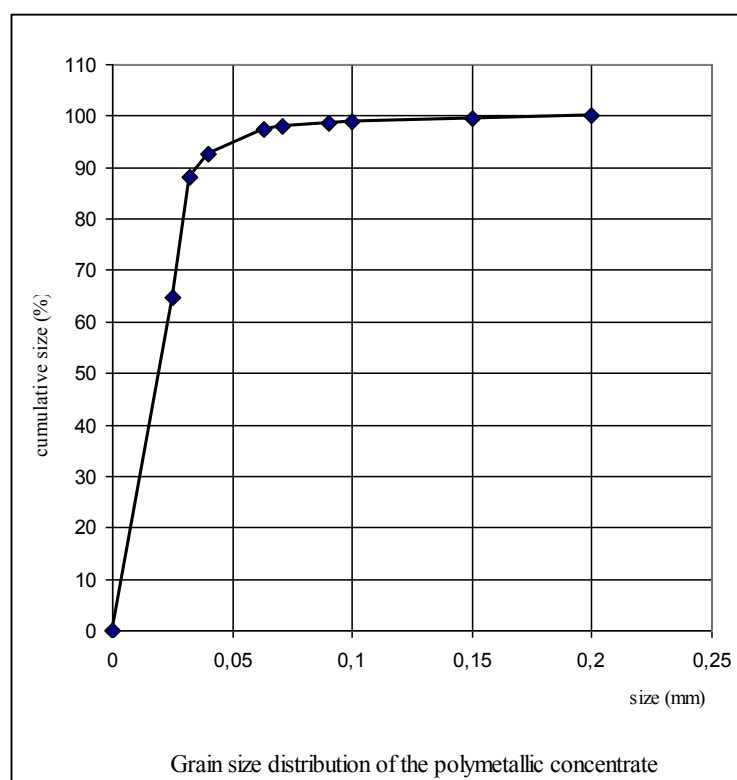


Fig. 20. Particle size distribution of SOMINCOR polymetallic concentrate.

3.5 BOR PYRITE CONCENTRATE

The BOR pyrite concentrate was analyzed according to the similar methodology. At first elemental composition was determined using combination of ICP-AES, FAAS, XRF, gravimetry, fire assay and carbon and sulfur analysis. Results obtained by BOR INST and IMNR were similar and were presented in **table 24**.

Table 24. ELEMENTAL COMPOSITION OF BOR PYRITE CONCENTRATE, [%]												
SAMPLE	Cu	Fe	CaO	Pb	Zn	As	Hg	S	Si	Ag	Al ₂ O ₃	C
BOR pyrite	1.77	39.29	0.83	0.014	0.009	0.011	0.0001	44.04	4.11	0.0008	1.75	0.7
SAMPLE	MgO	Se	SiO ₂	Ti								
BOR pyrite	0.07	0.009	7.96	0.027								

Additionally, Ba, Cd, Ga, La, Te and Zr were found at levels lower than 0.001 % , while Cr, Ge and V at levels lower than 0.005 %.

BOR pyrite concentrate density evaluated by IMNR using pycnometric analysis was 4.3225 t/m³.

Qualitative analysis performed at BOR INST using XRD method (**figure 21**) revealed 4 structures i.e. pyrite (FeS₂), quartz (SiO₂), calcite (Ca(CO)₃) and potassium in form of Cu₈K₃S₆.

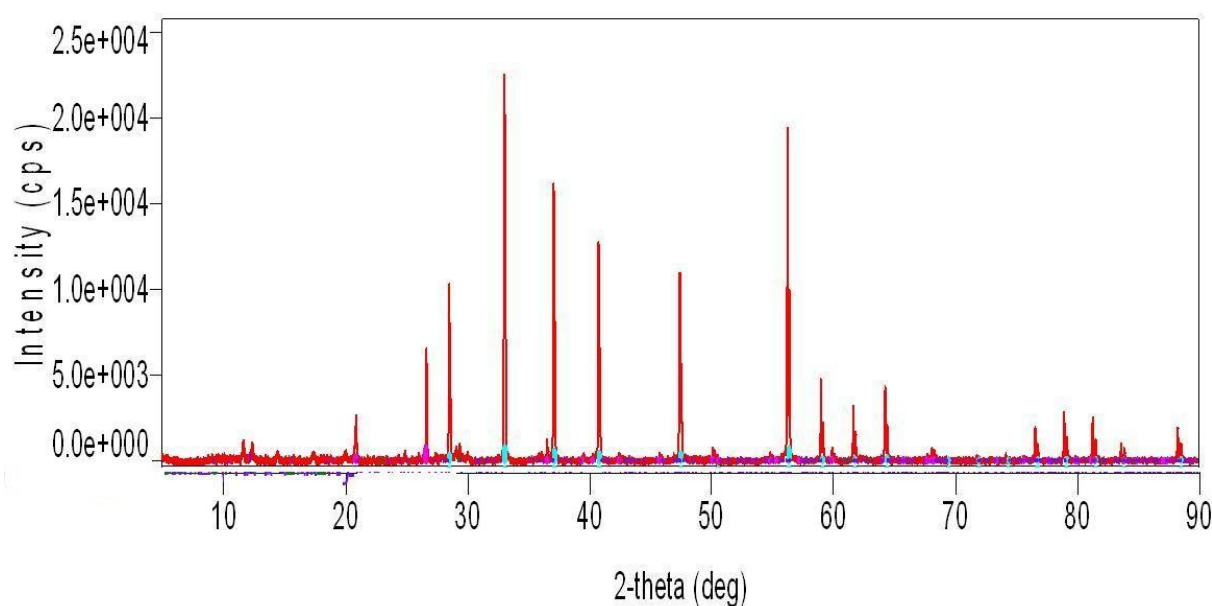


Fig. 21. XRD pattern of BOR pyrite concentrate (BOR INST).

The same type of analysis prepared by IMNR demonstrated 14 different minerals, and 8 most common were presented in **table 25**.

Table 25. XRD RESULTS OF BOR PYRITE CONCENTRATE

	MINERAL							
Sample	Pyrite	Quartz	Sphalerite	Gypsum	Chalcoppyrite	Rutile	Covellite	Molybdenite
BOR pyrite	FeS ₂	SiO ₂	ZnS	Ca(SO ₄)(H ₂ O) ₂	CuFeS ₂	TiO ₂	CuS	MoS ₂

The other possible structures include: hydronium jarosite – (K,H₃O)Fe₃(SO₄)₂(OH)₆, wroewolfeite – Cu₄(OH)₆(SO₄)(H₂O)₂, alunite – KAl₃(SO₄)₂(OH)₆, pyrophyllite – (Al₄Si₈O₂₀(OH)₄)_{.3333}, diaspore – AlOOH and biotite – KFeMg₂(AlSi₃O₁₀)(OH)₂.

Quantitative analysis of pyrite concentrate performed by IMNR was presented in **table 26**. Content of sulfide mass in the whole sample was as high as 84.4%.

Table 26. MINERALOGY OF BOR PYRITE CONCENTRATE, [%]

	MINERAL								
Sample	Pyrite	Quartz	Wroewolfeite	Hydronium jarosite	Sphalerite	Gypsum	Chalcoppyrite	Covellite	Alunite
BOR pyrite	79.6	10.4	2.3	1.6	0.01	1.3	0.8	0.6	1.3

Microscopic study (IMNR) in reflected light and in transmitted light of the analysed sample highlighted the following minerals (in order of frequency): pyrite – FeS₂; sphalerite – ZnS; covellite – CuS; chalcoppyrite – CuFeS₂; bornite – Cu₅FeS₄; magnetite – Fe₃O₄; carbonaceous material (coke, charcoal) and quartz – SiO₂.

Sample examined in reflected light was mainly composed of pyrite in a form of grains with size between 1 micrometer to hundreds of micrometers. Other minerals were usually associated with pyrite. Sphalerite appeared as independent grains or was associated with pyrite and covellite. Covellite appeared as having dimensions from 1 micron up to hundreds of microns, most often associated with pyrite, or in the form of globular particles with dendritic structure. Covellite was present on the cracks into bornite granules or as bornite exclusions. Chalcoppyrite was observed as grains with dimensions of up to hundreds of microns, independent or associated with pyrite. Bornite occurred as crystals with under-micron dimension, with dendritic structure embedded into globular covellite. In addition, bornite was found as granules with dimension up to hundreds of microns, independent or associated with covellite. Magnetite was accidentally found in globular glass particles, perhaps fayalitic glass or as microcrystals in bornite. Carbonaceous matter was accidentally detected as charcoal and coke. The study carried out in transmitted light on a sample immersed in nitrobenzene showed the abundant presence of quartz as crystals with sizes from microns up to hundreds of microns.

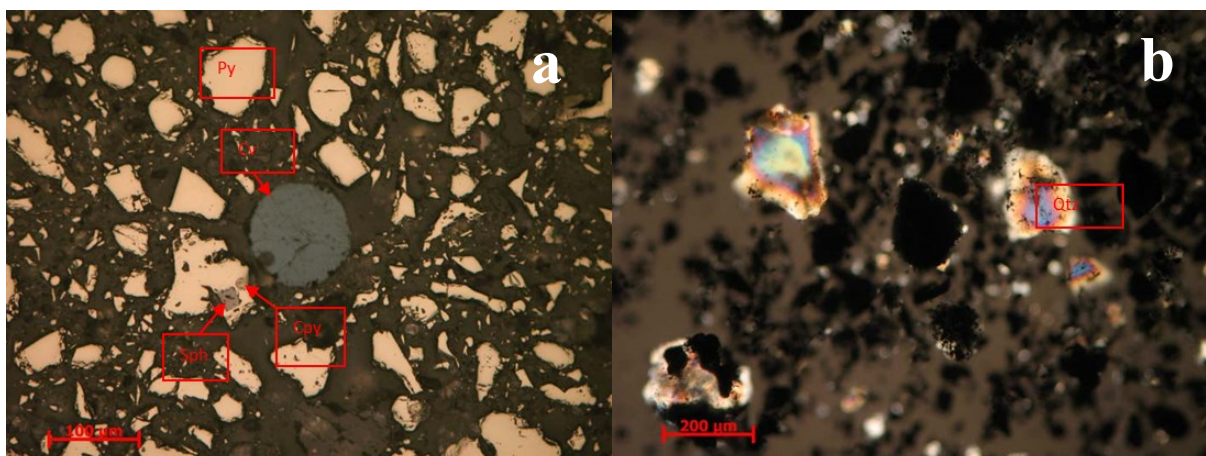


Fig. 22. Images of metallographic microscope of BOR pyrite concentrate, in reflected light: *a* – with pyrite (py), sphalerite (sph), covellite (cv), chalcopyrite (cpy), and in transmitted light: *b* – with quartz (qtz).

Particle size distribution was determined using laser particle size analyzer as well as a series of standard sieves. Results of sieving was presented in **table 27** and laser analysis in **figure 23**.

Table 27. GRAIN SIZE DISTRIBUTION OF BOR PIRYTE CONCENTRATE			
Size class, mm	m, %	Undersize, %	Oversize, %
-0.300+0.212	5.00	100.00	5.00
-0.212+0.150	2.50	95.00	7.50
-0.150+0.106	6.00	92.50	13.50
-0.106+0.075	16.00	86.50	29.50
-0.075+0.053	10.50	70.50	40.00
-0.053+0.038	11.50	60.00	51.50
-0.038	48.50	48.50	100.00

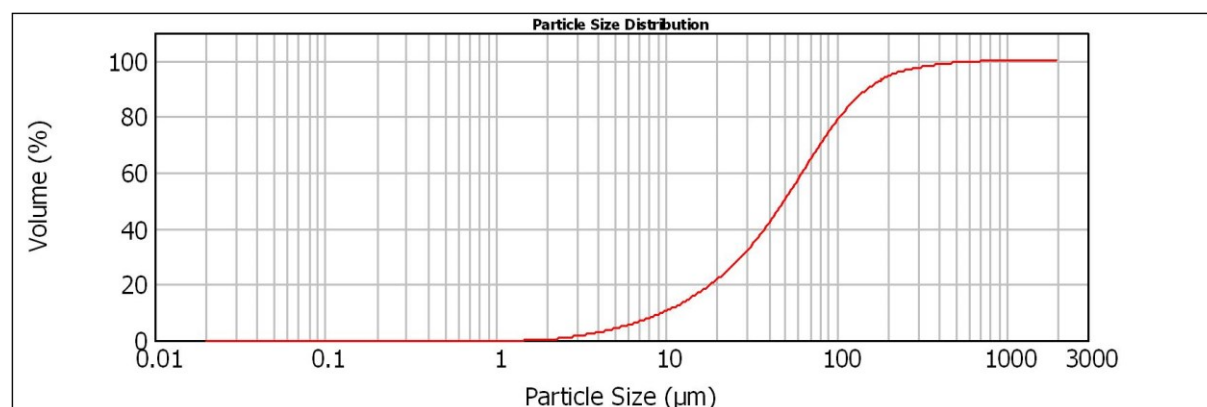


Fig. 23. Grain size distribution obtained using laser particle analyzer.

These data were in accordance with results presented by IMNR.

3.6 CLC PYRITE TAILINGS

Residual pyrite was characterized by IMNR. The elemental composition was obtained according to the methods presented above.

Table 28. ELEMENTAL COMPOSITION OF CLC PYRITE CONCENTRATE, [%]

Sample	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Cd	Hg	Si	S	Ag	Sb	Co
CLC pyrite	0.75	38.20	0.70	1.7	1.4	0.36	0.021	0.012	0.012	-	2.09	51.58	-	0.048	-

Density of this material was estimated at 4.6353 t/m³ (IMNR).

Qualitative analysis was performed using XRD method with a pattern presented in **figure 24**, while list of detected minerals with their structures was presented in **table 29**.

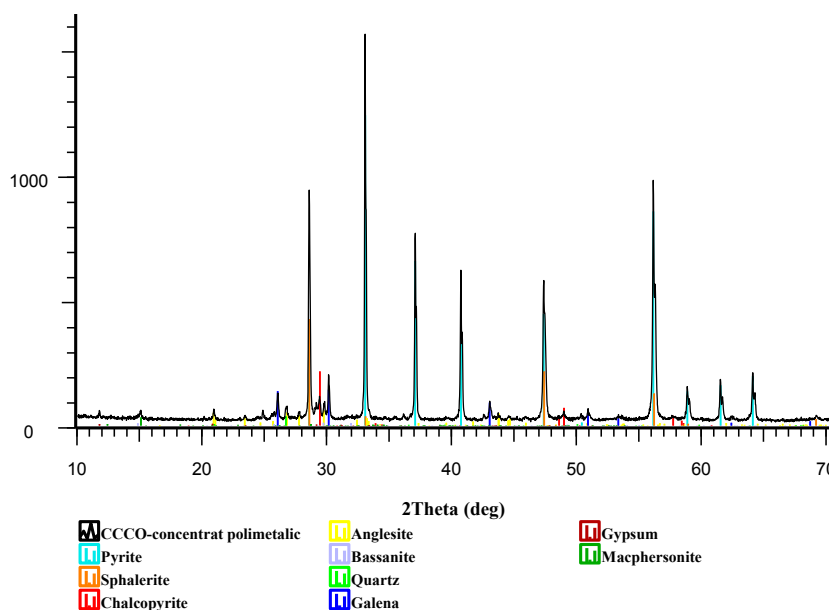


Fig. 24. XRD pattern of CLC pyrite concentrate.

Table 29. XRD RESULTS OF CLC PYRITE CONCENTRATE

	MINERAL								
Sample	Pyrite	Quartz	Sphalerite	Gypsum	Chalcopyrite	Galena	Anglesite	Bassanite	Macphersonite
CLC pyrite	FeS ₂	SiO ₂	ZnS	Ca(SO ₄)·(H ₂ O) ₂	CuFeS ₂	PbS	PbSO ₄	MoS ₂	Pb ₄ (SO ₄)(CO ₃) ₂ (OH) ₂

Quantitative evaluation of identified minerals, obtained by IMNR – using RIR (Reference Intensity Ratio) method, was presented in **table 30**.

Table 30. MINERALOGY OF CLC PYRITE CONCENTRATE, [%]									
	MINERAL								
Sample	Pyrite	Quartz	Sphalerite	Gypsum	Chalcopyrite	Galena	Anglesite	Bassanite	Macphersonite
CLC pyrite	78	2	8	1	5	2	2	2	<1

Microscopic study in reflected light highlighted the following minerals (in order of frequency): pyrite – FeS_2 ; sphalerite – ZnS ; chalcopyrite – CuFeS_2 ; galena – PbS ; bornite – Cu_5FeS_4 ; chalcocite – Cu_2S and covellite – CuS . Sample examined in reflected light was composed mainly of pyrite as grains with dimensions from less than 1 micrometer up to hundreds of micrometers. Pyrite occurred mostly as individual crystals, and associated with other minerals present in the sample. Sphalerite occurred as associated with pyrite, galena, chalcopyrite. Chalcopyrite occurred individually as well as was associated with pyrite, bornite. Galena occurred individually and associated with pyrite. Bornite was present as individual and associated with chalcocite and pyrite. Covellite was associated with pyrite.

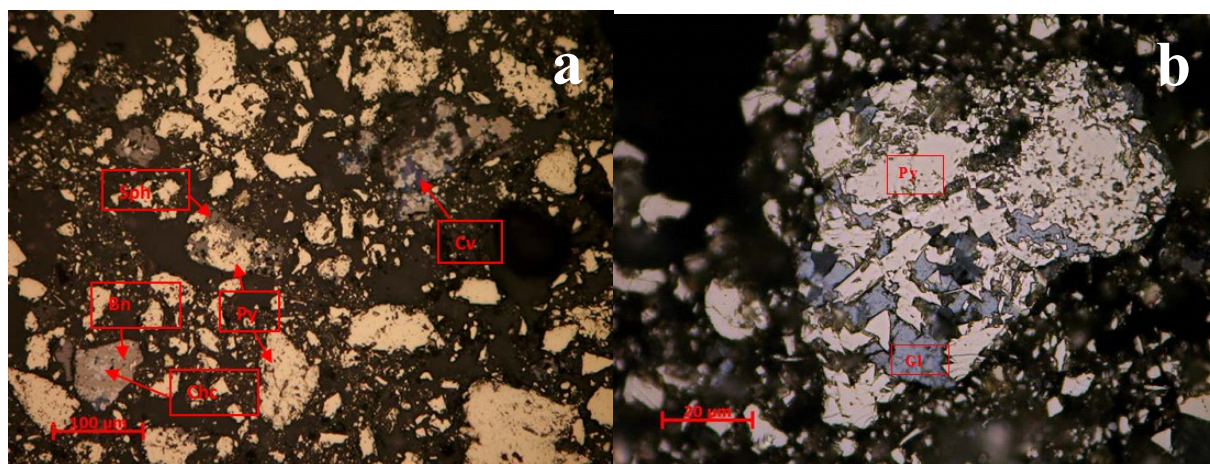


Fig. 25. Images of metallographic microscope of CLC pyrite tailings, in reflected light: *a* – with pyrite (py), sphalerite (sph), chalcocite (chc), bornite (bn) and covellite (cv), and in transmitted light: *b* – with pyrite (py), and galena (gl).

Results of grain size analysis performed by IMNR were presented in **table 31** and in **figure 26**.

Size class, mm	m, %	Undersize, %	Oversize, %
$d > 0.150$	2.32	100.00	2.32
$0.100 < d < 0.150$	4.22	97.68	6.55
$0.090 < d < 0.100$	3.55	93.45	10.09
$0.071 < d < 0.090$	6.12	89.91	16.21
$0.063 < d < 0.071$	3.21	83.79	19.42
$0.040 < d < 0.063$	18.59	80.58	38.01
$0.032 < d < 0.040$	13.67	61.99	51.68
$0.025 < d < 0.032$	10.13	48.32	61.82
$d < 0.025$	38.18	38.18	100.00

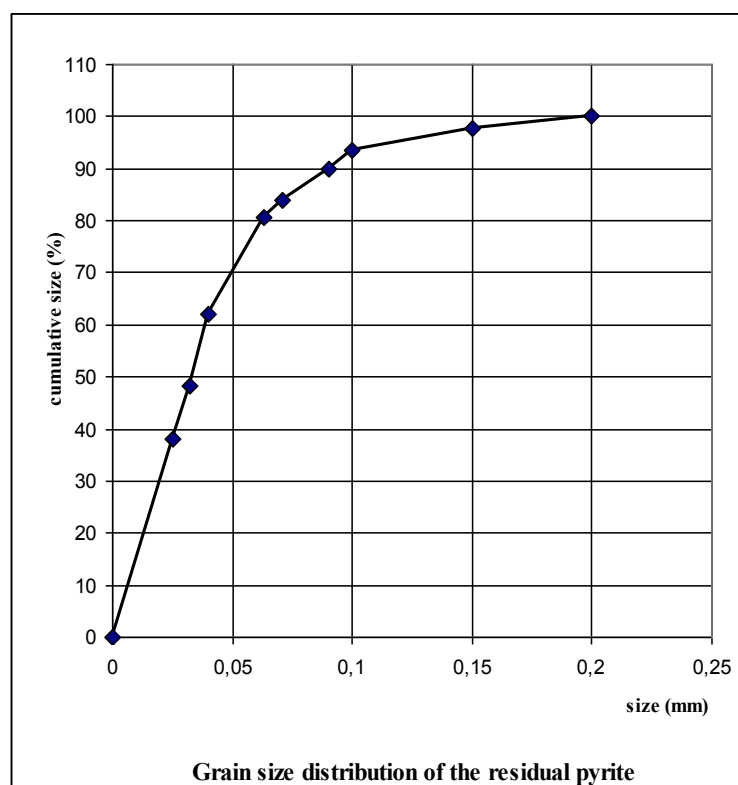


Fig. 26. Particle size distribution of CLC pyrite tailings.

3.7 SOMINCOR PYRITE CONCENTRATE

Material delivered by Somincor was thoroughly analyzed at IMN with respect to elements composition, particle size distribution and qualitatively with respect to its content of minerals.

Table 32. ELEMENTAL COMPOSITION OF SOMINCOR PYRITE TAILINGS, [%]

Sample	Cu	Fe	Ca	Pb	Zn	As	Mn	Bi	Si	S	Ag	Sb	Co
Somincor pyrite	0.42	42.4	–	0.98	2.06	–	–	–	–	46.05	0.0045	0.049	0.012

Qualitative analysis was performed using XRD method, the resulting pattern was presented in **figure 27**.

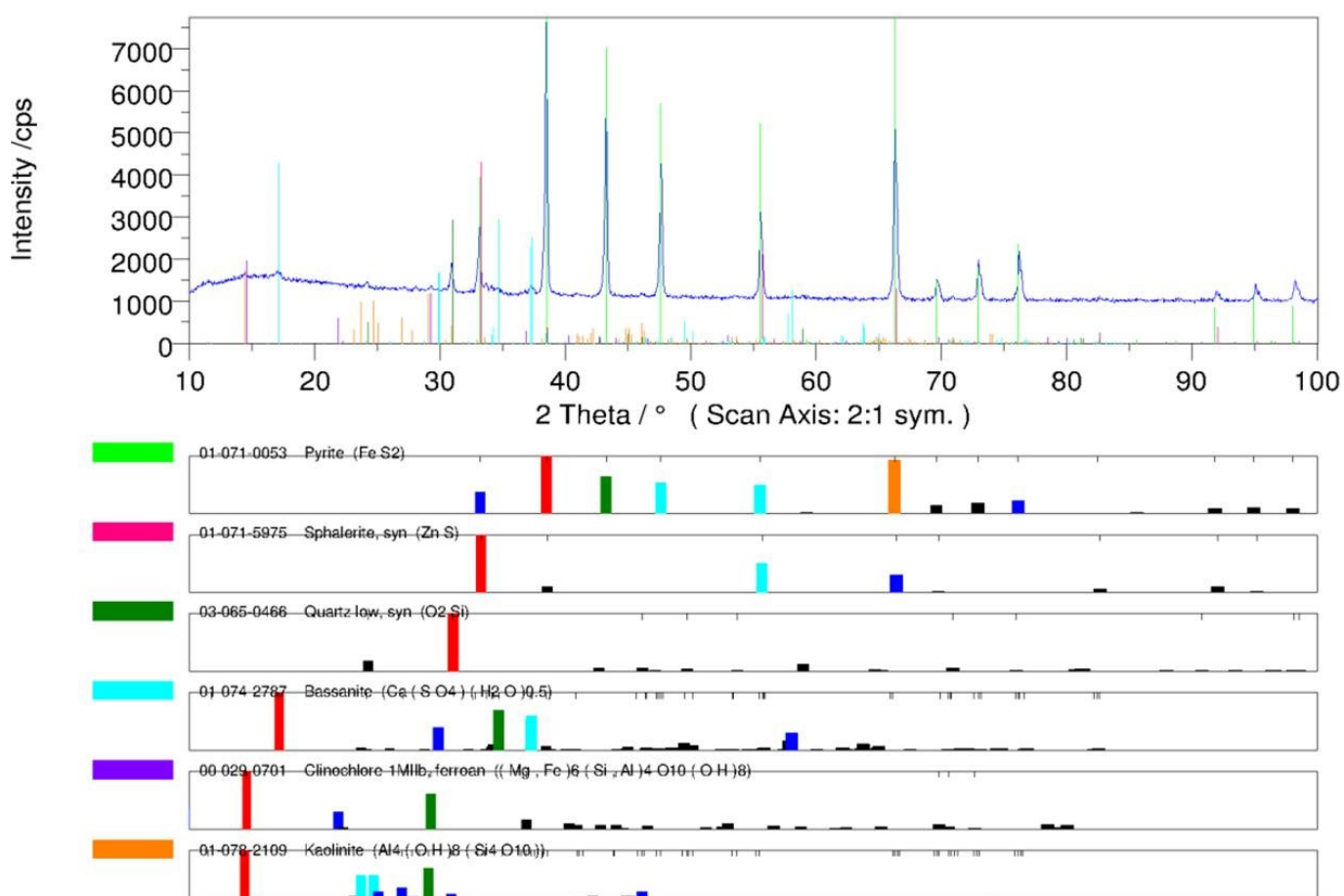


Fig. 27. XRD pattern of Somincor pyrite tailings.

Material contained mostly pyrite – FeS_2 , but also sfalerite – ZnS and quartz – SiO_2 . In very tiny amount basanite $\text{CaSO}_4(\text{H}_2\text{O})_{0.5}$ was present and probably in trace amount aluminosilicate of kaolinite structure $\text{Al}_4(\text{OH})_8\text{Si}_4\text{O}_{10}$ or minerals such as serafinite $(\text{Mg,Fe})_6(\text{Si,Al})_4\text{O}_{10}(\text{OH})_8$. However, diffraction lines intensity from these minerals were too weak to define it perfectly.

Structural analysis using X-ray microanalyzer JXA 8230, JEOL was performed (IMN). Samples were sputtered with thin gold layer to avoid charge agglomeration. This produced a signal in recorded spectra due to M_α Au line. This technique allowed to prepare elements distribution and perform qualitative and quantitative point analysis using EDS mode. Maps of elements were presented in **figure 28**.

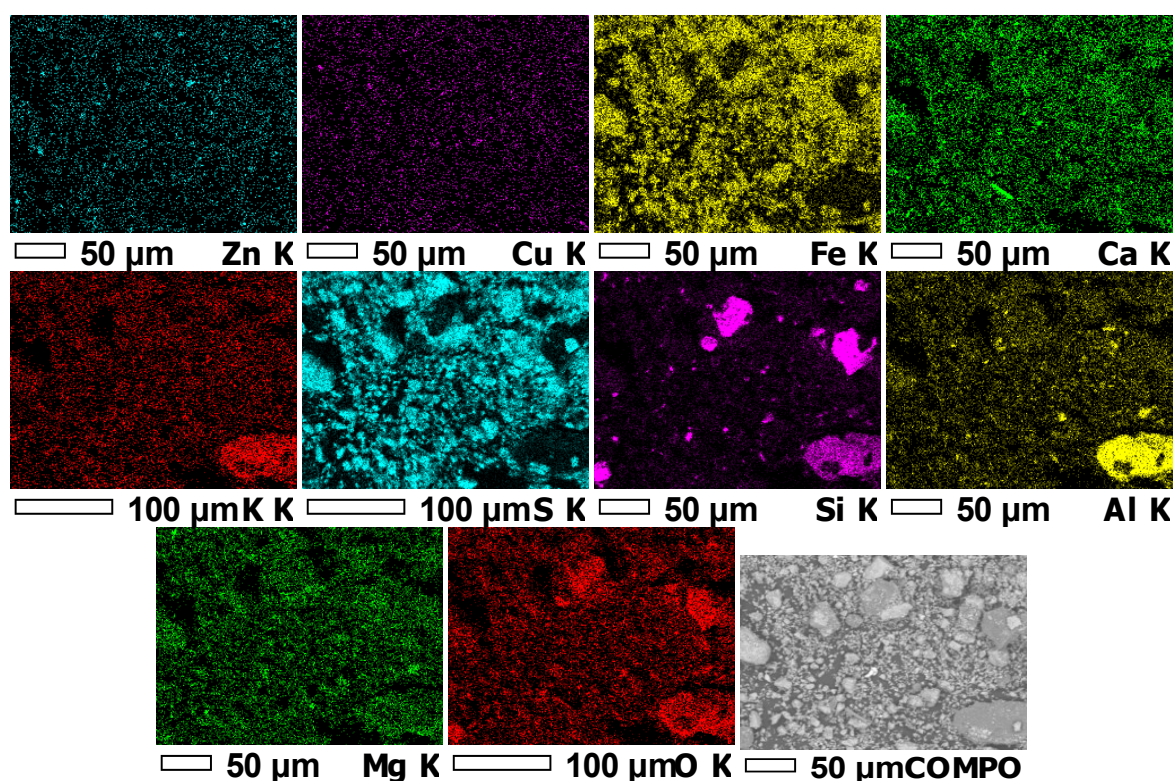
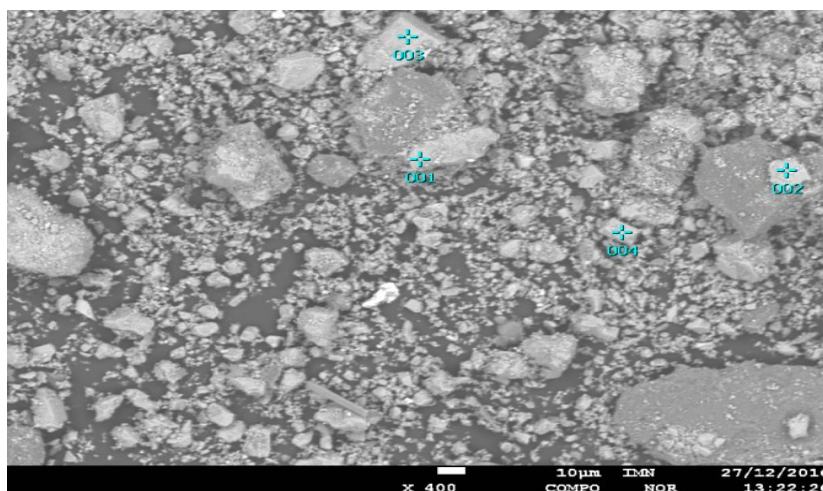
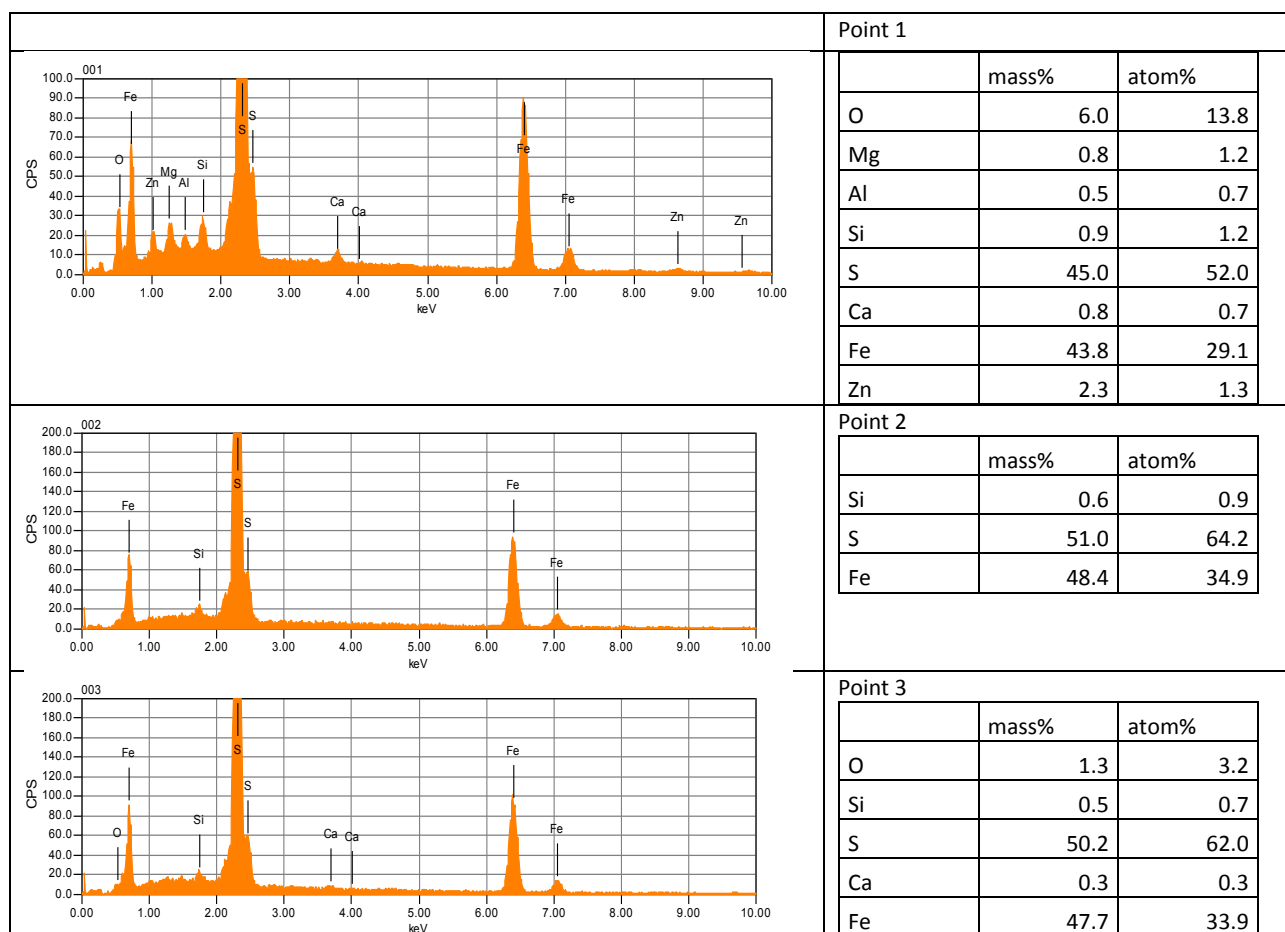


Fig. 28. Elements distribution within selected area using magnification 400x.

Qualitative and quantitative analyses were performed for selected points – presented in **figure 29**.



Spectra for points 1, 2, 3 and 4, where points 1 and 4 were attributed to iron in form of FeS_2 .



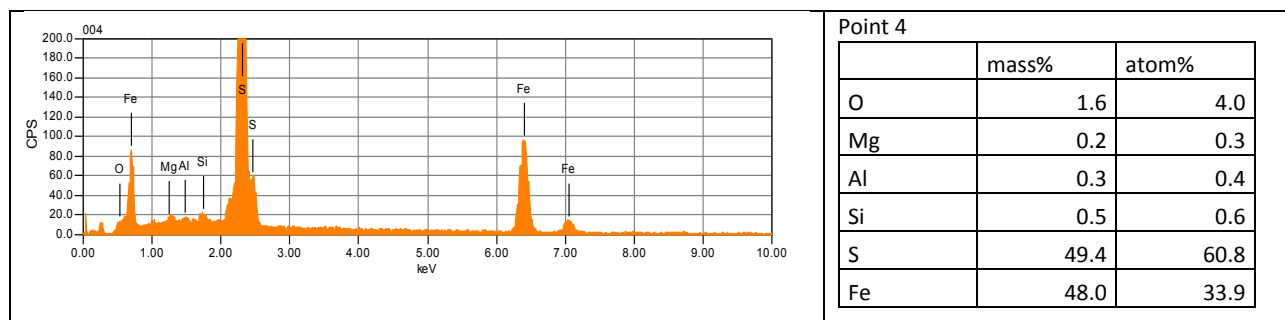
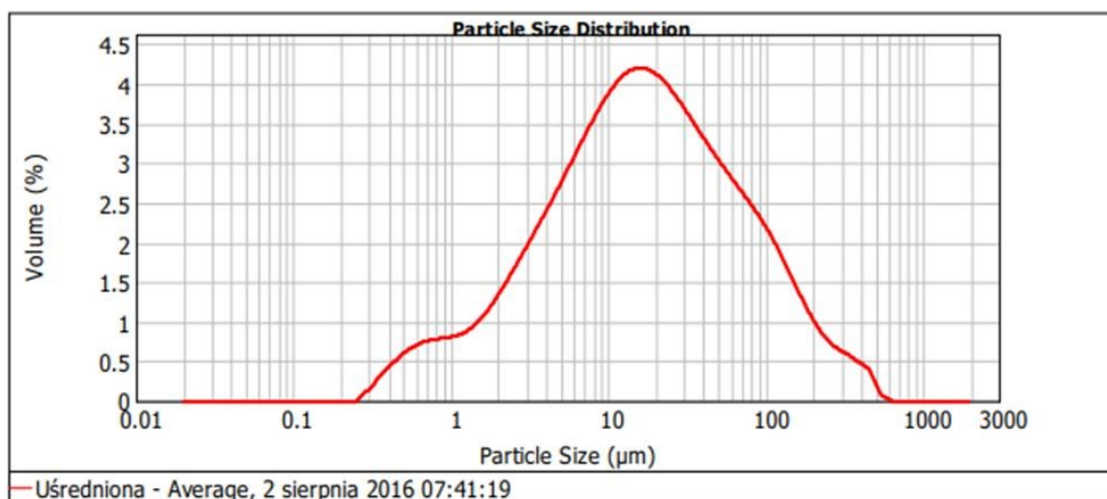


Fig. 29. Quantitative analysis using EDS method for previously selected points.

Additionally particle size analysis, using laser particle analyzer, was performed (IMN).



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	21.54	45.000	9.54	500.000	0.09
5.000	25.97	75.000	4.48	1000.000	0.00
15.000	13.74	100.000	7.29	2000.000	
25.000	8.16	200.000	2.09		
35.000	5.47	300.000	1.62		
45.000		500.000			

Fig. 30. Particle size distribution of Somincor pyrite tailings.

4. SUMMARY

Within the scope of the study materials delivered by CLC, KGHM, BOR and Somincor were characterized. These included polymetallic ores and concentrates and complex or low grade copper ores and concentrates and pyrite concentrates.

The complete elemental analysis was done for every material using the best available methodology (ICP). Additionally, some samples were treated by different techniques like XRF, FAAS, carbon and sulphur analysis and gravimetry to entirely show the composition of the materials.

Real density of materials was determined using pycnometer method. In several cases also bulk density was determined.

XRD method was used for qualitative analysis of all materials. Except XRD patterns the obtained results were collected in tables showing the determined structure of the mineral.

Microscopy analysis with additional equipment setups was used to supplement XRD results i.e. the qualitative results as well as to perform quantitative evaluation of the particular minerals within each sample. This allowed to establish association between minerals within analyzed materials.

Particle size distribution was determined for every material either using laser particle analyzer or by set of sieves, or using both these methods.

Although similar level of copper can be found in CLC ROM (~1%) and KGHM ROM (~1.2%) the amount of other metals is varied. Lead content in CLC material is ca. 15 time higher (2.4% instead of 0.16% for KGHM), zinc content is 3.35% and lower than 0.05% for CLC and KGHM, respectively. Almost 20 times higher antimony content can be observed in CLC material and only 3 times bigger value of cobalt. In both materials the same silver amount was observed i.e. 0.0057%.

All concentrates, i.e. the polymetallic from CLC, Somincor and BOR as well as low grade from KGHM, were thoroughly analyzed to determine their elemental composition. Mostly, 15 elements were analyzed, which was sufficient for further research. In one case analysis covered more than 30 elements to show the precision of assumed methodologies. X-ray powder diffraction with microscopic techniques allowed to evaluate differences between materials with respect to their mineralogy. BOR and CLC have very similar Cu content (~1.8%), tripled value was obtained for Somincor (5.2%), while the highest (13%) for material from KGHM. The highest Fe content was found in CLC (38%), also in Somincor (29%) and BOR concentrate (23%) these value were high. On the contrary KGHM concentrate has about 8% Fe. Zn and Pb content were as follows: 5.8% and 3.6%; 4.6% and 4.6%; 9.1% and 14.8%; 1.1% and 4.6%, respectively for CLC, BOR, Somincor and KGHM. The highest Ag content was found in KGHM material circa 640 ppm and only 85 ppm in the CLC one.

CLC material was mainly composed of pyrite (80%) with some sphalerite and chalcopyrite. BOR consisted of 45% pyrite, 25% barite and also sphalerite, galena and waste (11%). Somincor contained 54% pyrite and almost equal

(~15%) galena, sphalerite and chalcopyrite. Analysis of KGHM material showed 10% pyrite, ~20% bornite, ~20% dolomite, 9% rectorite and 14% quartz.

Additionally, BOR pyrite concentrate was taken into consideration in this report.