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Application News

X-ray Analysis EDX-7000 / XRD-6100

Identification of Copper Concentrates Using EDX and XRD

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User Benefits

- EDX enables non-destructive and rapid qualitative analysis of constituent elements with only simple pretreatment.
- The elemental information from EDX can be utilized for XRD to identify minerals with greater accuracy.
- Since XRD can determine the compound form of an element, the accuracy of qualitative-quantitative analysis of EDX can be improved.

■ Introduction

The copper ore mined from a deposit has a copper content of about 1 %, and is refined to a copper concentrate with a grade of about 20 to 40 % through a process called ore dressing, including crushing and polishing. This copper concentrate is then smelted in a refinery to raise the copper grade to nearly 99 %.

Copper ore contains a variety of minerals other than copper-containing minerals. Although EDX can perform qualitative-quantitative analysis of elements, it cannot determine their compound forms or identify minerals. However, by combining the elemental information from EDX and database search based on X-ray diffraction, it is possible to identify minerals in copper ores and their residues after dressing and obtain useful information for the next process. In addition, the compound form information obtained from X-ray diffraction can be utilized for calculating the qualitative-quantitative results of EDX with greater accuracy.

■ Samples

Fig. 1 shows two samples. Sample ① is copper concentrate powder and sample ② is residue after dressing of copper ore.

Sample ①
(Copper Concentrate Powder)

Sample ②
(Residue After Copper Dressing)

Fig. 1 Samples

■ Sample Preparation

As sample preparation, the powdered samples were dried by heating at 110 ˚C for 2 hours and then ground in a mortar to be uniform.

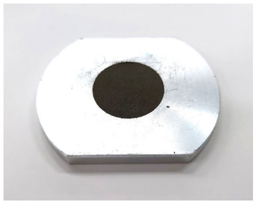
The sample for EDX analysis was placed in a sample container lined with a 5 µm-thick polypropylene film and lightly pressed down for measurement.

The cavity of an aluminum holder for XRD was filled with powder sample and pressed to make the surface flat for measurement.

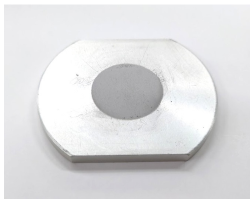
Fig. 2 shows the samples after preparation.

Sample ①: EDX

Sample ②: EDX



Sample ①: XRD



Sample ②: XRD

Fig. 2 Sample Preparation

1. Qualitative Analysis via EDX

■ Elements

$_{11}$Na to $_{92}$U

■ Results of EDX Qualitative Analysis

Qualitative analysis was conducted using EDX-7000.

In order to obtain the elemental information necessary for qualitative analysis with XRD, a quantitative analysis was performed using the fundamental parameter (FP) method, briefly assuming the compound form to be oxides. Table 1 shows the top 10 elements contained in each sample.

Table 1 Top 10 Elements in Content

Sample name	Elements
Sample ①	S, Fe, Zn, Cu, Si, Ca, Al, Mg, Pb, K
Sample ②	Si, Al, S, Fe, Mg, K, Zn, Cu, Ca, Ba

2. Qualitative Analysis via XRD

■ Measurement with XRD

Copper-containing ores are composed of a variety of minerals. To identify such minerals, the use of an X-ray diffractometer (XRD) is effective. In this study, the powders of sample ① and sample ② were measured with the XRD-6100. The automatic 5-sample change stage was mounted to the XRD-6100, and the sample packed in the aluminum holder was loaded. Fig. 3 shows the appearance. Also, Table 4 shows the measurement conditions.

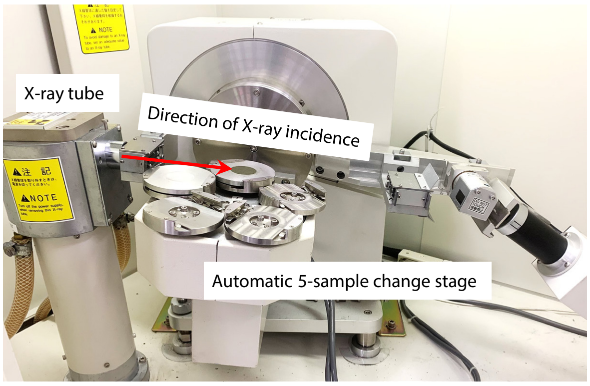


Fig. 3 Attachment and Sample Mounted on XRD-6100

■ X-ray Diffraction Patterns and Qualitative Analysis

Fig. 4 shows the X-ray diffraction patterns of sample ① and sample ②.

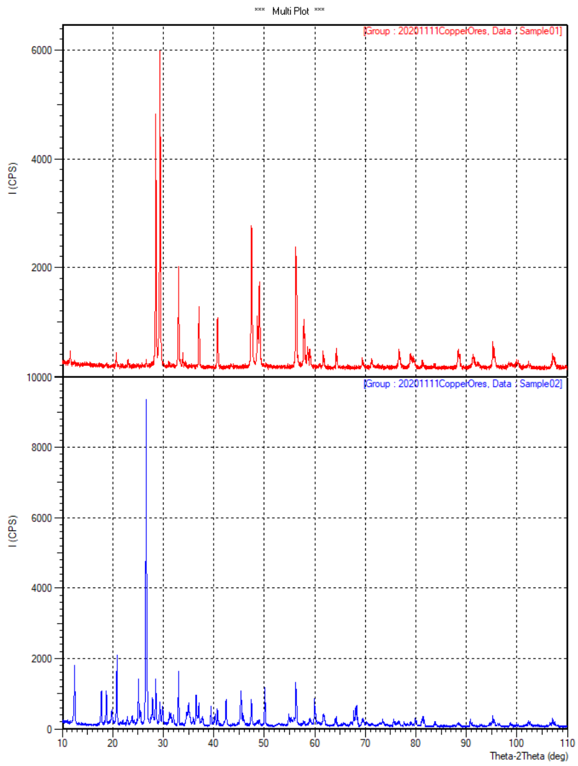


Fig. 4 X-ray Diffraction Patterns of Sample ① (upper) and Sample ② (lower)

In the qualitative analysis with XRD, if a database search is performed without setting the constituent elements, a large number of substances with closely similar crystal systems but composed of different elements may be returned as candidates. It is therefore important to register the appropriate elemental information in advance to obtain the correct search results.

In this study, we used the qualitative analysis results obtained by EDX (Table 1), registered them as effective elements in the qualitative analysis software, and performed a search using the ICDD (International Centre for Diffraction Data) PDF2 database.

At that time, we added H, which cannot be detected by EDX, and O, which is not used in the qualitative analysis, assuming the compound forms to be hydroxides, oxides, etc. Figs. 5 and 6 show the results of the qualitative analysis.

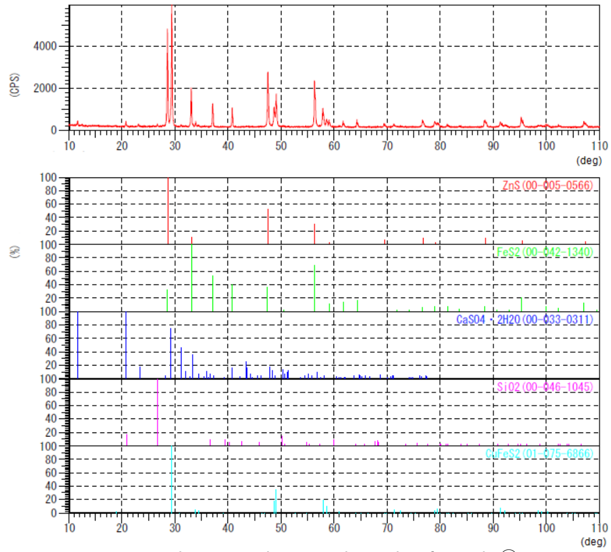


Fig. 5 Qualitative Analysis Search Results of Sample ①

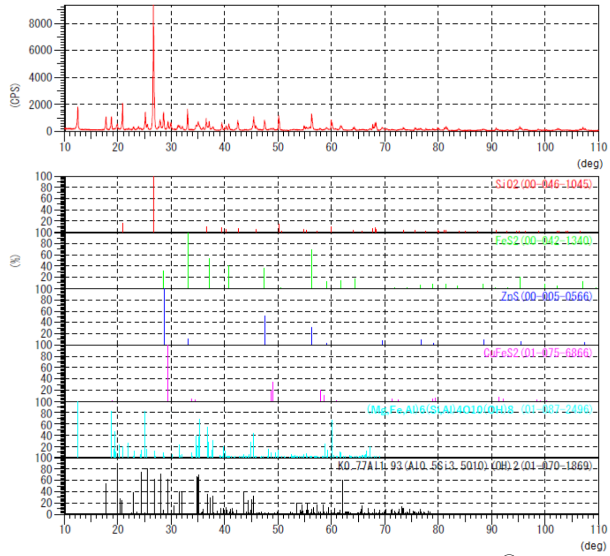


Fig. 6 Qualitative Analysis Search Results of Sample ②

Table 2 shows a list of the minerals contained and their simple quantitative values based on the RIR method. In sample ①, metal sulfides such as sphalerite, pyrite, and chalcopyrite were abundant, with small amounts of gypsum and quartz, a silicate mineral. In contrast, sample ② contained a large amount of silicate minerals such as quartz, clinochlore, and muscovite, as well as sulfides such as pyrite, a small amount of sphalerite, and chalcopyrite.

Table 2 List of Minerals Contained

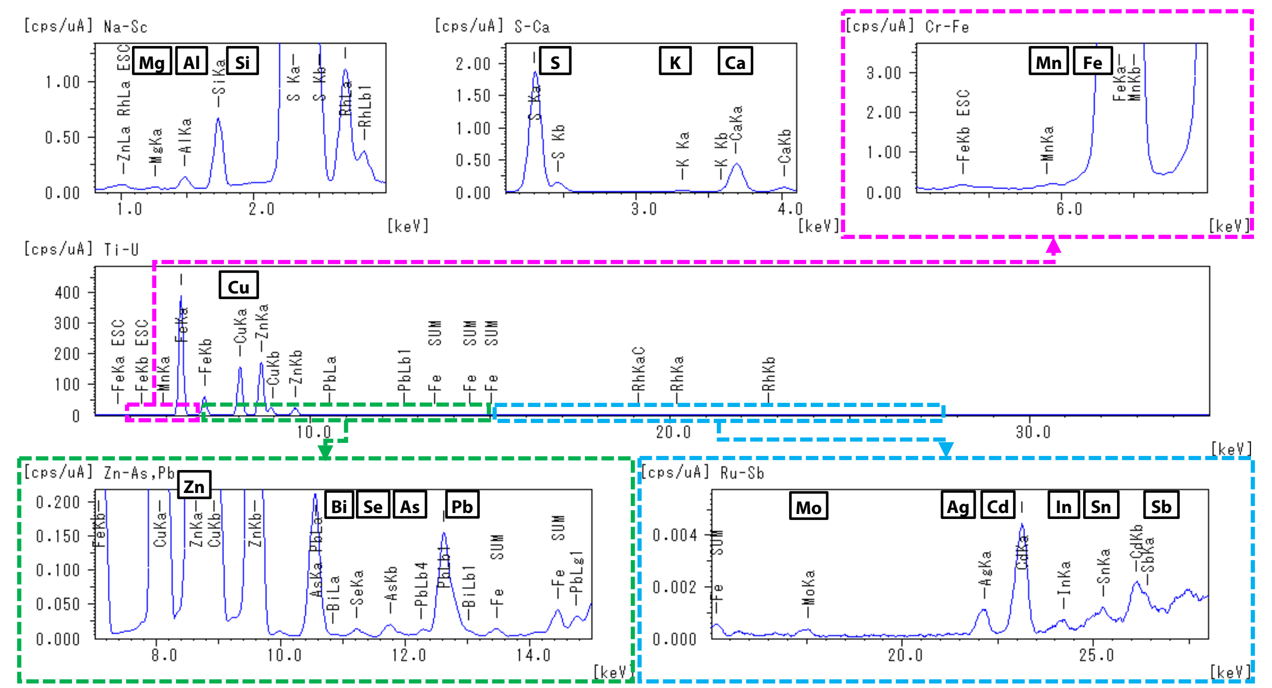
Sample name	Component		Quantitative value (RIR method)
Sample ①	ZnS	Sphalerite	36
	FeS_2	Pyrite	37
	$CaSO_4 \cdot 2H_2O$	Gypsum	3
	SiO_2	Quartz	1
	$CuFeS_2$	Chalcopyrite	23
Sample ②	SiO_2	Quartz	35
	FeS_2	Pyrite	13
	ZnS	Sphalerite	4
	$CuFeS_2$	Chalcopyrite	1
	$(Mg, Fe, Al)_6(Si, Al)_4O_{10}(OH)_8$	Clinochlore	35
	$KAl_2(Si, Al)_4O_{10}(OH)_2$	Muscovite	11

(Unit: wt%)

3. Quantitative Analysis via EDX

Figs. 7 and 8 show the results of qualitative-quantitative analysis with EDX.

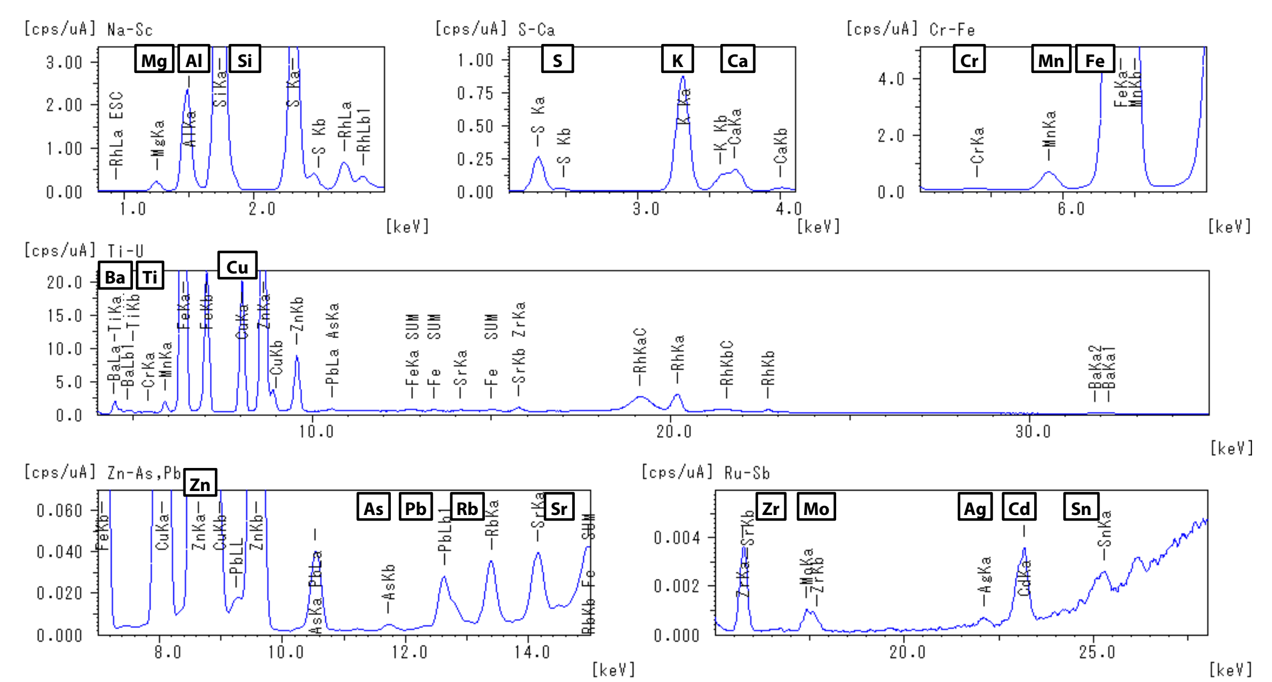
From the XRD analysis results, it came out Mg, Al, Si, and K are largely contained as oxides, Fe, Cu, and Zn as sulfides, and Ca as a sulfate. We therefore performed quantitative analysis based on the EDX measurement results mentioned above, using the FP method, assuming the compound forms of Mg, Al, Si, and K to be oxides.



Component	S	Fe	Zn	Cu	SiO_2	Ca	Al_2O_3	MgO	Pb	K_2O
Quantitative value [wt%]	40.1	26.0	13.0	12.1	3.8	1.6	1.5	1.3	0.24	0.20

Component	As	Cd	Mn	Se	Ag	Sb	Bi	Sn	In	Mo
Quantitative value [wt%]	0.077	0.027	0.024	0.0069	0.0056	0.0046	0.0041	0.0040	0.0025	0.0024

Fig. 7 Results of Qual-Quantitative Analysis of Sample ①



Component	SiO_2	Al_2O_3	S	Fe	MgO	K_2O	Zn	Cu	Ca	Ba	Ti
Quantitative value [wt%]	52.4	21.1	6.3	6.1	6.0	4.7	1.8	0.65	0.42	0.26	0.15

Component	Mn	Pb	Cr	Zr	Cd	As	Rb	Sr	Sn	Mo	Ag
Quantitative value [wt%]	0.085	0.012	0.010	0.0090	0.0052	0.0041	0.0039	0.0035	0.0023	0.0018	0.00065

Fig. 8 Results of Qual-Quantitative Analysis of Sample ②

■ Measurement Conditions

Table 3 EDX Measurement Conditions

Instrument	: EDX-7000
Elements	: Na-U
Analysis group	: Qualitative analysis and quantitative analysis
Detector	: SDD
X-ray tube	: Rh target
Tube voltage	: 50 [kV] (Ti-U) (Cr-Fe) (Zn-As, Pb) (Ru-Sb), 15 [kV] (Na-Sc) (S-Ca)
Tube current	: Auto [μA]
Collimator	: 10 [mmφ]
Primary filter	: Non (Ti-U) (Na-Sc), #2 (S-Ca), #3 (Cr-Fe), #4 (Zn-As, Pb), #1 (Ru-Sb)
Atmosphere	: Vacuum
Integration time	: 60 [s] x 6 Ch
Dead time	: Max. 30 [%]

Table 4 Measurement Conditions for Qualitative Analysis with X-ray Diffractometer

Instrument	: XRD-6100
X-ray tube	: Cu target
Kβ cut	: Counter monochromator
Detector	: Scintillation detector
Attachment	: Automatic 5-sample change stage
Voltage - current	: 40 kV-40 mA
Slit condition	: DS=1˚, SS=1˚, RS=0.3 mm
Measurement mode	: θ-2θ continuous scan
Measurement speed	: 2˚/min
Measurement step	: 0.02˚
Measurement angle range	: θ-2θ=10-110˚
Rotation	: 60 rotations/min

■ Conclusion

The copper ores before and after dressing were analyzed to determine the composition and compound forms with EDX and XRD.

By using the elemental information obtained from the qualitative analysis with EDX, a more accurate XRD database search became possible. Furthermore, more accurate quantitative analysis with EDX also became possible by taking into account the compound form obtained from qualitative analysis with XRD.

From these results, it was found that utilizing the results of both EDX and XRD as preliminary information was effective to analyze copper ore before and after dressing.

<References>
· K. Tomita: Industrial Raw Materials Dressing Handbook, Kyoritsu Shuppan (1966)
· H. Sekiguchi: Some Applications of Automated Fundamental X-ray Diffraction Techniques, Shimadzu Review Vol.52 [2] (1995)

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01-00104-EN First Edition: Jun. 2021