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Energy Dispersive X-ray Fluorescence Spectrometer EDX-7200

Application News

Analysis of Recycled Materials by EDXRF

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User Benefits

- Analysis of various forms of materials, including solids, powders, and liquids, is possible.
- Various types of analyses, from screening analysis for toxic elements to qualitative and quantitative analysis for quality control, can be conducted corresponding to the purpose.
- Simple analysis is possible without chemical sample preparation.

■ Introduction

Use of recycled material is increasing in response to heightened environmental awareness. In the automotive industry, bumpers are produced by reusing recovered plastics, and in the electrical and electronics industry, smartphones are manufactured using recycled aluminum.

Against the backdrop of increasing use of recycled materials, each country has established a legal and regulatory system. For example, in 2021, the German government amended the German Packaging Law (VerpackG), and required that the percentage of recycled plastics in disposable PET bottles be at least 25 % from January 1, 2025.

On the other hand, quality control is critical when manufacturing recycled products because raw materials with various unknown origins are used. Since there is also a risk of contamination with toxic elements such as lead and mercury, it is important to confirm that products conform to RoHS/ELV and other regulations.

This article introduces examples of screening analysis for toxic elements and elemental analysis by qualitative and quantitative analysis for various recycled materials, including plastics, rubbers and glasses.

■ Recycled Resin (Toxic Element Analysis)

A screening analysis for a total of 9 elements, including the 5 RoHS elements and Cl, Sb P, and Sn, was conducted for 100 % recycled polyethylene (PE). Fig. 1 shows an image of the sample, and Table 1 shows the results.

Fig. 1 100 % Recycled PE

Table 1 Results of Screening Analysis of 100 % Recycled PE

[ppm]

	Cd	Pb	Cr	Hg	Br	Cl	Sb	P	Sn
Quantitative value	ND*	55.7	3.5	ND*	20.2	152.1	15.0	23.6	5.1
Threshold value** judgment	OK	OK	OK	OK	OK	OK	OK	OK	OK

*ND: Below the lower limit of detection.

**Threshold values assume Cd: 70-130 ppm, elements other than Cd: 700-1300 ppm.

■ Recycled Rubber (Composition Analysis)

In addition to toxic elements, quality control of additives and inclusions in products is also important. The content of each element can be controlled by qualitative and quantitative analysis. Fig. 2 shows an image of the sample, and Table 2 shows the qualitative and quantitative analysis results.

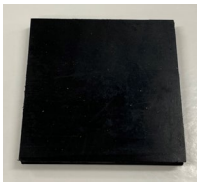

Fig. 2 Recycled Rubber

Table 2 Results of Qualitative and Quantitative Analysis of Recycled Rubber

[%]

Ca	S	Zn	Si	Mg	Fe	Al	Cl	
9.96	2.21	1.20	0.62	0.14	0.11	0.090	0.055	
K	**Ba**	**Cu**	**Br**	**Sr**	**Pb**	**Ti**	**Zr**	**SBR rubber* ($C_{12}H_{14}$)**
0.051	0.027	0.019	0.009	0.008	0.006	0.002	0.001	85.5

* Quantitative calculation assuming the balance is $C_{12}H_{14}$.

■ RPF (Cl Analysis)

Since RPF (Refuse Paper and Plastics Fuel; solid fuel made from used paper and waste plastics) is a low cost, high calorie fuel, it is used as an alternative to fossil fuels. However, control of the Cl concentration is important, as boilers will corrode if low melting point chlorides form during combustion. Table 3 shows the quality and classification (JIS Z 7311) of RPF according to the total chlorine (Cl) content.

A chemical analysis method (JIS Z 7302-6) is also specified as an analysis method for Cl, but because the analysis operation is complicated and time is required before the measurement results are available, EDX is used as the screening analysis for voluntary control purposes. Due to the nature of RPF, the Cl concentration differs depending on the location. Therefore, it is important to measure Cl at multiple locations. Fig. 3 shows the results of quantitative determination of Cl in the sample shown in the figure using a screening analysis kit.

Table 3 Quality and Classification of RPF (Excerpt from JIS Z 7311)

Quality	RPF-coke	RPF		
Class		**A**	**B**	**C**
Total Cl	≤0.6 %	≤0.3 %	>0.3 % to ≤0.6 %	>0.6 % to ≤2 %

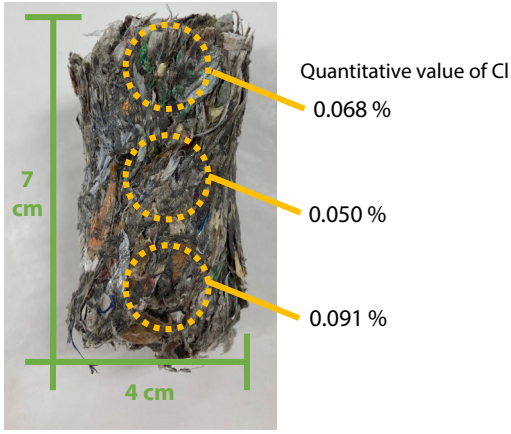

Fig. 3 Results of Cl Quantitative Analysis of RPF

■ Glass (Composition/Toxic Element Analysis)

Waste glass is frequently sorted by color, but because the composition differs in many cases, even in glass of the same color, visual sorting is sometimes difficult. Fig. 5 shows the results of a measurement of colorless transparent glass (Fig. 4: Soda-lime glass/lead glass).

If lead (Pb) is melted together with general soda-lime glass, inclusions may form during recycling due to the difference in the melting points, and this may cause damage of the melting furnace and other problems. However, these risks can be avoided by EDX analysis, enables easy sorting of the materials.

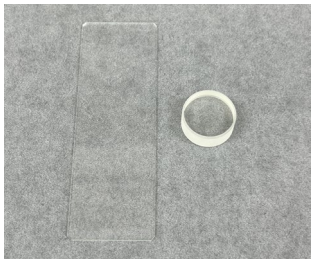

Fig. 4 Colorless Transparent Glass
(Left: Soda-Lime Glass, Right: Lead Glass)

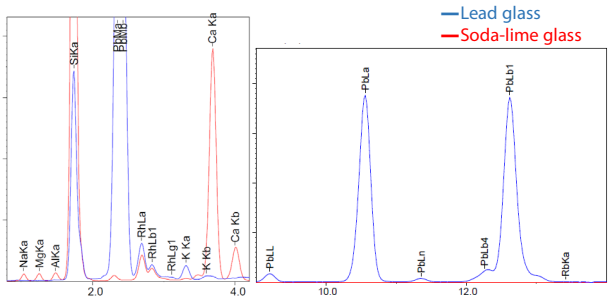

Fig. 5 Overlay of Profiles of Soda-Lime Glass and Lead Glass

Analysis of toxic elements in recycled glass is also important. A screening analysis of the 5 RoHS elements was carried out for toys made from recycled glass (Fig. 6). Table 4 shows the results.

Fig. 6 Toys Made from Recycled Glass

Table 4 Results of Screening Analysis of Toys Made from Recycled Glass

[ppm]

	Cd	Pb	Cr	Hg	Br
Quantitative value	ND*	64.2	11.2	ND*	ND*
Threshold value** judgment	OK	OK	OK	OK	OK

**ND: Below the lower limit of detection.
**Threshold values assume Cd: 70-130 ppm and elements other than Cd: 700-1300 ppm.

■ New Recycled Material (Composition Analysis)

From the viewpoint of the SDGs, reuse of materials that had been discarded as waste until now has progressed in recent years. Examples include packaging paper made from limestone or eggshells and biomaterials using grains or waste wood. Toxic element analysis and composition analysis of these new recycled materials are also important. Table 5 shows the results of a qualitative and quantitative analysis of recycled packaging paper (Fig. 7) made from eggshells.

Fig. 7 Recycled Packaging Paper Made from Eggshells

Table 5 Results of Qualitative and Quantitative Analysis of Recycled Packaging Paper Made from Eggshells

[%]

$CaCO_3$*	Si	Mg	S	Zn	
40.8	0.15	0.10	0.072	0.062	
K	**Al**	**Sr**	**Fe**	**Cu**	**Polyethylene** (**(CH_2)**)
0.017	0.012	0.007	0.005	0.002	58.8

*Ca was determined as calcium carbonate.
**Quantitative calculation assuming the balance is CH_2.

■ Conclusion

EDX supports analysis in various applications, from screening analysis for toxic elements to quality control. Since simple analysis is possible without complex sample preparation, EDX is useful in analyses of recycled materials, which are expected to increase in the future.

■ Analysis Conditions (1): RoHS Screening Analysis

Table 6 Analysis Conditions (RoHS Screening Analysis)

Elements	: $_{48}Cd$, $_{82}Pb$, $_{24}Cr$, $_{80}Hg$, $_{35}Br$, $_{17}Cl$, $_{51}Sb$, $_{15}P$, $_{50}Sn$
Analysis group	: Screening
Detector	: SDD
X-ray tube	: Rh target
Tube voltage	: 10, 15, 30, 50 [kV]
Tube current	: Auto [μA]
Collimator	: 10 [mmφ]
Primary filters	: None, #1, #2, #3, #4
Atmosphere	: Air
Integration time	: 100 [s] × 5 Ch
Dead time	: Max. 30 [%]
Automatic time-reduction function	: OFF

■ Analysis Conditions (2): Qualitative and Quantitative Analysis

Table 7 Analysis Conditions (Qualitative and Quantitative Analysis)

Elements	: $_{11}Na$ – $_{92}U$
Analysis group	: Qualitative and quantitative
Detector	: SDD
X-ray tube	: Rh target
Tube voltage	: 15 [kV] (Na-Sc), (S-Ca), 50 [kV] (Ti-U)
Tube current	: Auto [μA]
Collimator	: 10 [mmφ]
Primary filters	: None (Na-Sc), (Ti-U), #2 (S-Ca)
Atmosphere	: Vacuum
Integration time	: 60 [s] × 3 Ch
Dead time	: Max. 30 [%]

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