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Application News

Dynamic Particle Image Analysis System
Fourier Transform Infrared Spectrophotometer
Infrared Microscope

Evaluation of Microscopic Foreign Matter in Recycled Plastics Using Dynamic Image Analysis, Infrared Microscopy, and SEM-EDS

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User Benefits

- It is easy to evaluate the size and quantity of microscopic foreign particles, which are key quality criteria of recycled plastics.
- With the microcell method of DIA, microscopic foreign matter can be detected and counted quickly using a small sample quantity.
- Infrared microscope and SEM-EDS systems provide information about the morphology and composition of microscopic foreign matter that is useful for improving recycling processes.

■ Introduction

One of the quality criteria for recycled plastic flakes and pellets is the content of foreign matter. Quantitatively evaluating the size and quantity of foreign particles contained in flakes and pellets is important for examining foreign matter removal processes intended to improve pellet quality and optimize manufacturing processes for products made with recycled materials.

In this article, pellets of virgin and recycled polyethylene terephthalate (hereafter PET) were dissolved in hexafluoro-2-propanol (hereafter HFIP), a solvent for dissolving PET, to prepare suspensions of insoluble microscopic foreign particles. These suspensions were measured with a dynamic particle image analysis (DIA) system (iSpect™ DIA-10) to evaluate the size and number of insoluble microscopic foreign particles (patent pending). In addition, part of the suspensions were diluted tenfold and filtered through a membrane filter to collect the insoluble microscopic foreign particles on the filter. The morphology and composition of the collected microscopic foreign particles were evaluated using a digital microscope, an infrared microscope system (IRTracer™-100 and AIM-9000), and an SEM-EDS system. These evaluations enabled comparison of the sizes, quantities, and types of foreign particles contained in virgin and recycled materials.

Fig. 1 iSpect™ DIA-10 Dynamic Particle Image Analysis System

Fig. 2 Infrared Microscope System
Left: IRTracer™-100; Right: AIM-9000

■ PET Bottle Recycling and Foreign Matter

A typical PET bottle recycling process is shown in Fig. 3. One issue caused by foreign matter contained in pellets during PET bottle manufacturing is bursting during molding. Recycled materials tend to burst more frequently than virgin materials, but because foreign matter is microscopic and scatters, collecting foreign matter after bursting is not possible and identifying the cause is difficult.

Another issue is clogging of filters during melting and extrusion processes. Metal filters are used to remove foreign matter during processing, but filter clogging causes pressure to rise, so they must be replaced periodically. However, because the size and quantity of foreign particles differ depending on the pellets, optimizing the filter type and replacement timing is difficult.

Information on the size and quantity of foreign particles in pellets is expected to be useful for making process improvements in the following ways:

- Improving yield and productivity through optimization of parameters by selecting metal filters matched to pellet quality.
- Improving foreign matter removal steps during pellet manufacturing intended to improve recycled material quality.

■ Analysis Conditions

Preparation of Microscopic Foreign Matter Suspensions

100 mg/mL suspensions were prepared by adding HFIP to PET pellets (virgin and recycled) and left to stand at room temperature. Because PET dissolves in HFIP, microparticles that do not dissolve in HFIP remain in the suspensions. The dissolution behavior of pellets is shown in Fig. 4. Due to the different dissolution rates of virgin and recycled materials, pellets became invisible to the naked eye within 4 days for the virgin material and within 6 hours for the recycled material.

Acquisition of Particle Images and Evaluation of Particle Size and Particle Concentration by Dynamic Image Analysis

The suspensions were measured with a dynamic particle image analysis system (iSpect DIA-10, Shimadzu Corporation) to acquire images of microscopic foreign particles. Particle size and particle concentration were evaluated based on the acquired data. The analysis conditions are indicated in Table 1. As a control, HFIP stored in a glass bottle of the same type used for the suspension preparations was also measured to check for any contributions from glass bottles and solvent.

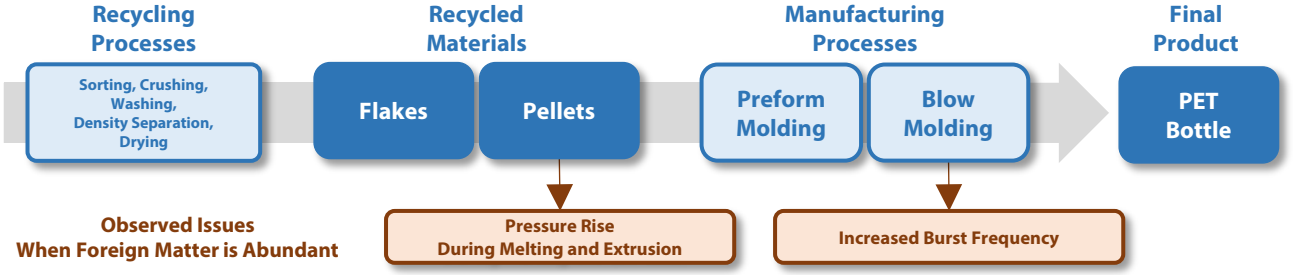


Fig. 3 Recycling Process (PET Bottle Case)

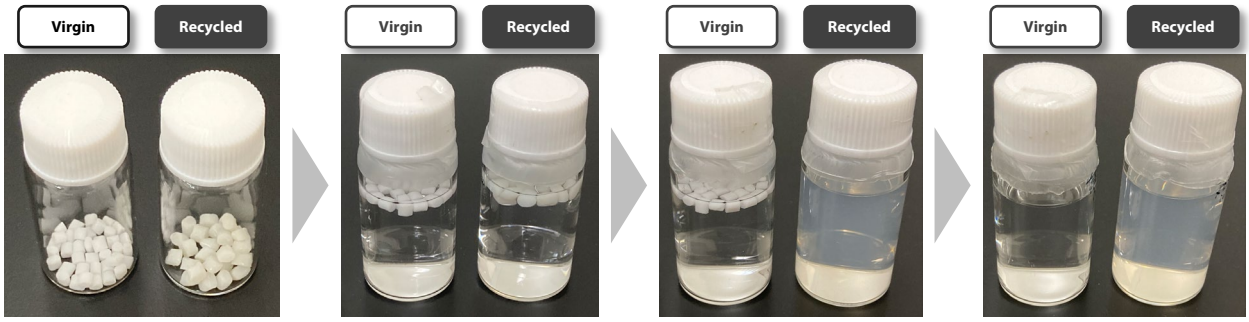


Fig. 4 Dissolution of Pellets in HFIP

Table 1 iSpect DIA-10 Analysis Conditions

Frame Rate:	8 fps
Efficiency:	96.5 %
Pump Volume:	250 µL
Threshold:	216 (90 % of background brightness)
Background Correction:	Enabled

Morphology Observation and Composition Analysis of Microscopic Foreign Matter by Infrared Microscope and SEM-EDS

Due to the high viscosity of the suspensions, 50 µL quantities were diluted tenfold. Insoluble microscopic foreign particles in the tenfold diluted suspensions were collected on a silver membrane filter by suction filtration. Fig. 5 shows the silver membrane filter after collection, observed with a digital microscope. The region within the red dashed circle is where particles were collected. Some of the particles collected on the silver membrane filter were measured by the micro-reflectance method using an infrared microscope system (IRTracer-100 and AIM-9000, Shimadzu Corporation). The measurement conditions are indicated in Table 2. In addition, particles identified as inorganic or metallic based on their images or infrared microscope results were further analyzed using an SEM-EDS system (ProX, Phenom World).

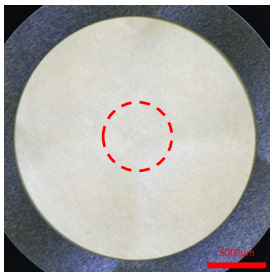


Fig. 5 Silver Membrane Filter after Collection

Table 2 Infrared Microscope Analysis Conditions

Instruments:	IRTracer™-100, AIM-9000
Resolution:	8 cm^{-1}
Number of Scans:	100
Apodization Function:	SqrTriangle
Wavenumber Range:	700 - 4000 cm^{-1}
Detector:	Liquid nitrogen-cooled MCT

■ Results and Discussion

Acquisition of Particle Images and Evaluation of Particle Size and Particle Concentration by Dynamic Image Analysis

Fig. 6 shows a portion of the particle images detected by the iSpect DIA-10. The first images listed when arranged in descending order of area-equivalent diameter are shown. As can be seen from the particle images, the largest particles had area-equivalent diameters of about 10 µm for the virgin material and about 40 µm for the recycled material. The images also show that particle shape and brightness vary from particle to particle. In general, the smaller the relative refractive index between the particle and dispersing medium, and the shorter the particle in the observation direction, the brighter the particle appears.

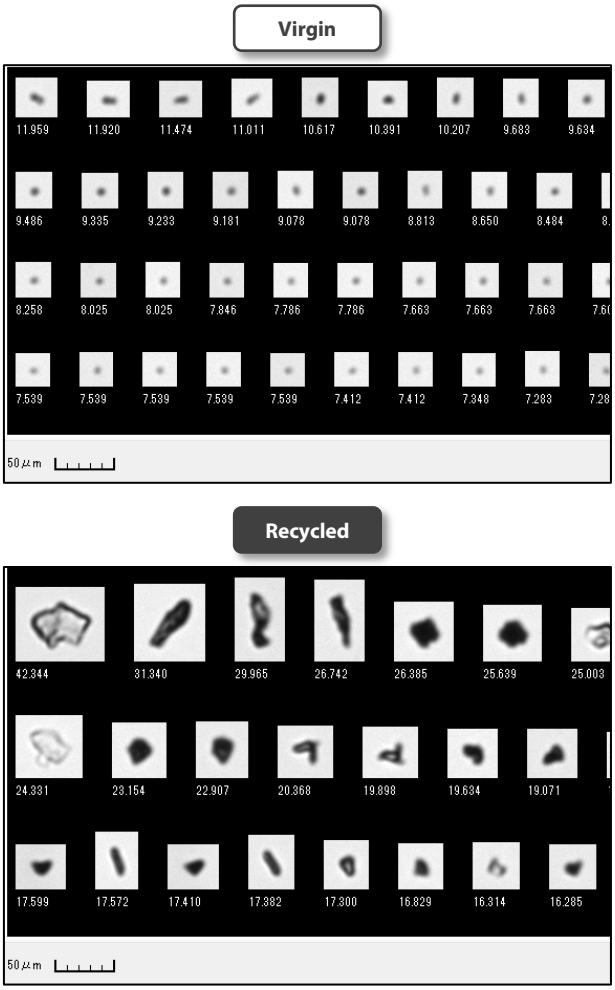


Fig. 6 Detected Particle Images
Excerpt from the top when arranged in descending order of area-based diameter.

Therefore, bright particles are presumed to be either organic materials with a small refractive index, glass fragments that transmit light, or plate-like particles (short in the observation direction), whereas dark particles are presumed to be inorganic or metallic particles or long dense particles (long in the observation direction). In this way, information on particle shape and brightness can be used to infer particle types.

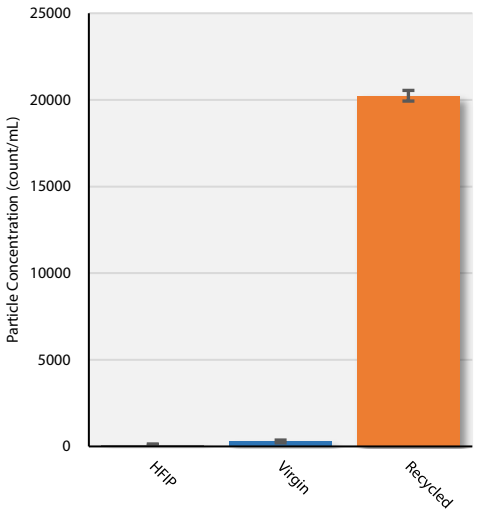


Fig. 7 Particle Concentration Measurement Results
Average of three measurements with standard deviation indicated by error bars.

Table 3 Particle Concentration by Size Range (count/mL)

Sample	Particle Concentration by Size Range (count/mL)					
	All	5-10 µm	10-25 µm	25-50 µm	50-100 µm	>100 µm
HFIP	104	91	12	0	0	0
Virgin	286	257	23	3	0	0
Recycled	20240	19523	699	18	0	0

Note: Average of three measurements.

Table 4 Number of Particles per Gram of Pellets by Size Range (count/g)

Sample	Number of Particles Gram of Pellets by Size Range (count/g)					
	All	5-10 µm	10-25 µm	25-50 µm	50-100 µm	>100 µm
Virgin	2860	2570	230	30	0	0
Recycled	202400	195230	6990	180	0	0

Note: Average of three measurements.
Note: Calculated from 100 mg/mL suspension concentration used for sample preparation.

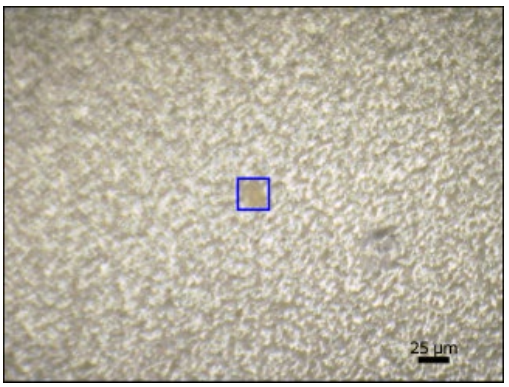
Morphology Observation and Composition Analysis of Microscopic Foreign Matter by Infrared Microscope and SEM-EDS

Selected results from analyzing the composition of the virgin material are shown in Figs. 8 and 9. Brown particles were observed in the infrared microscope images. Based on the IR spectra, they were presumed to be amide-type compounds or cellulose. These particles could have been introduced during handling/processing at the time of measurement, making it difficult to determine whether they originated from the sample itself.

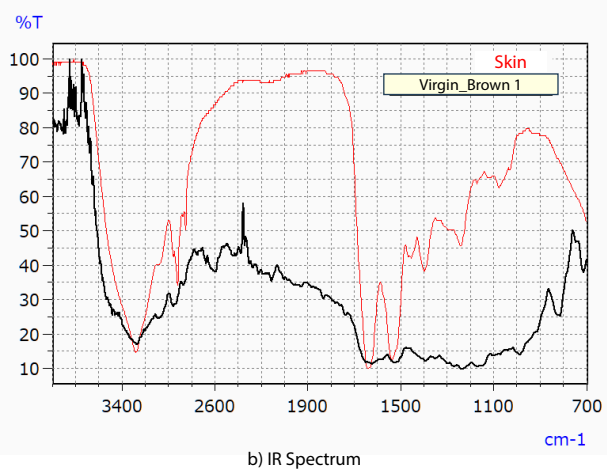
For the recycled material, the infrared microscope suggested the presence of particles that were not organic substances. SEM-EDS results for those particles are shown in Figs. 10 and 11. For the particle in Fig. 10, the EDS spectrum showed mainly C (carbon), Ca (calcium), and O (oxygen), suggesting a carbonate or similar substance. The Ag (silver) peak is from the filter substrate. For the particle in Fig. 11, a metallic luster was observed under an optical microscope and Al (aluminum) was detected in the EDS spectrum, indicating metallic aluminum.

Recycled materials may come into contact with various substances during the collection process. Therefore, composition information for foreign matter can be used to investigate the origin of foreign matter, evaluate measures for reducing foreign matter, and evaluate the impact of the foreign matter on final products.

Furthermore, the particle in Fig. 10 was confirmed to have a similar shape and major axis length to the particles shown in the second to fourth images from the left in the top row of iSpect DIA-10 particle images in Fig. 6. Based on the morphological similarity, iSpect DIA-10 particle images may also be useful for inferring particle composition.

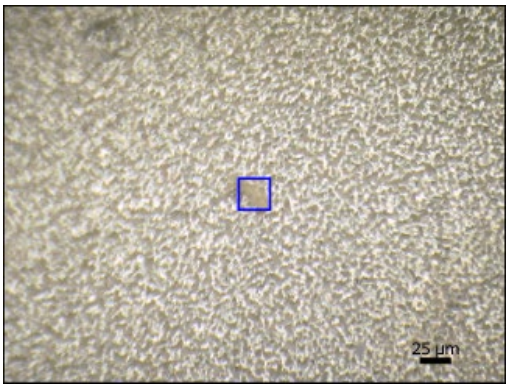


a) Observation Image by Infrared Microscope

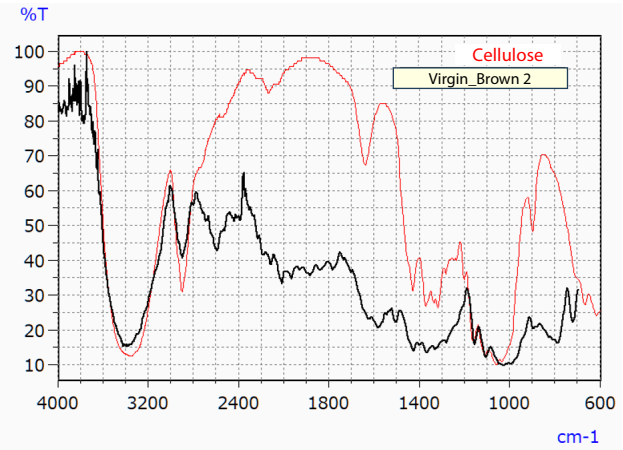


b) IR Spectrum

Fig. 8 Composition Analysis Result
Virgin Material: Human Skin-Derived Component

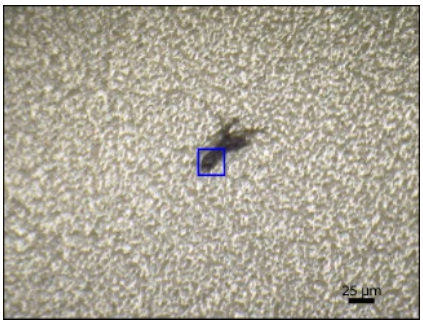


a) Infrared Microscope Observation Image

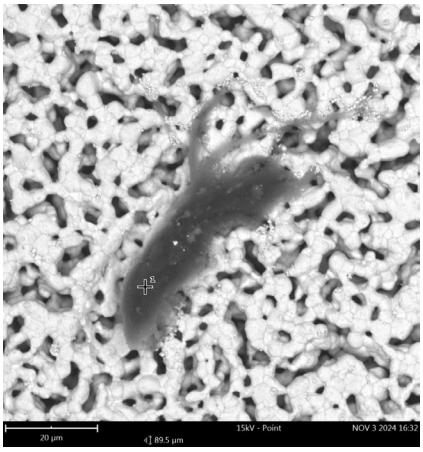


b) IR Spectrum

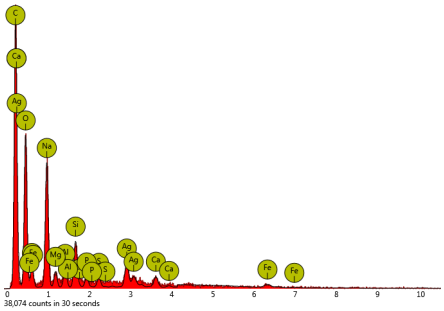
Fig. 9 Composition Analysis Result
Virgin Material: Cellulose



a) Infrared Microscope Observation Image

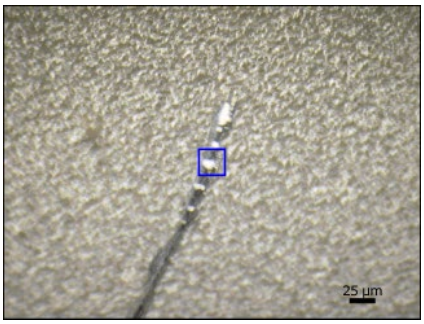


b) SEM Image

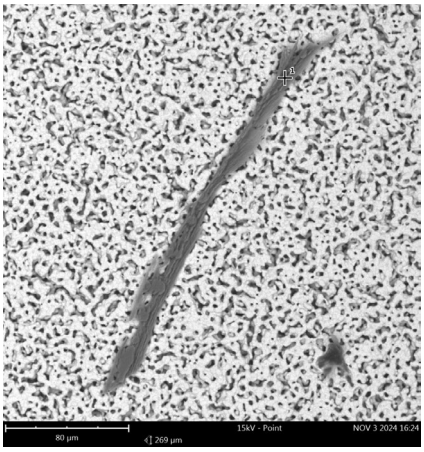


c) EDS Spectrum

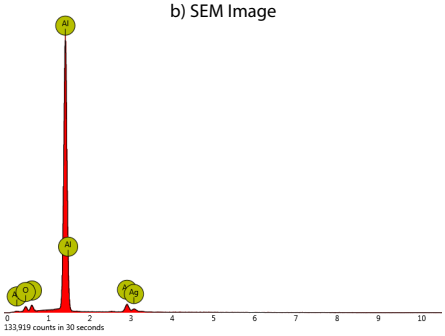
Fig. 10 Composition Analysis Result
Recycled Material: Particle Containing Mainly C, Ca, O



a) Infrared Microscope Observation Image



b) SEM Image



c) EDS Spectrum

Fig. 11 Composition Analysis Result
Recycled Material: Particle Containing Al

■ Conclusion

By using an iSpect DIA-10 system to measure suspensions prepared by dissolving two types of PET pellets (virgin and recycled) in HFIP, we obtained particle images of insoluble foreign matter and evaluated size and concentration of particles. That enabled quantitative comparison of particle size and count in microscopic foreign matter contained in virgin and recycled materials.

In addition, after diluting the pellet suspensions, the insoluble microscopic foreign matter in the diluted suspensions was collected on filters. The morphology and composition of the microscopic foreign matter were evaluated using an infrared microscope and SEM-EDS system. The results indicated that the suspensions contained organic substances, such as cellulose and amide-based compounds, and inorganic substances and metals that contain elements such as C, O, Ca, and Al (suggestive of carbonates and metallic aluminum).

Thus, the iSpect DIA-10 enables simple quantitative evaluation of the size and number of microscopic foreign particles in PET pellets using only small sample quantities. Furthermore, composition evaluation by infrared microscope and SEM-EDS systems provides useful information for investigating the origin of foreign matter, examining measures to reduce foreign matter, and evaluating impacts on final products. If similarities in size and shape are observed, particle images obtained with iSpect DIA-10 can also potentially be used to estimate particle composition.

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