Energy Dispersive X-ray Fluorescence Spectrometer EDX-7200

Application News

EDX-7200 New Primary Filter
- Quantitative Analysis of Titanium Dioxide in Antibacterial Coatings -

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User Benefits

◆ EDX-7200 can nondestructively analyze coatings.

◆ The amount of titanium dioxide can be determined by thin-film FP method.

◆ The new primary filter is effective for analyzing Ti to Co (energy range: 4~8 keV).

■ Introduction

With growing interest in hygienic products, various antibacterial products have recently been developed. Due to titanium dioxide's relative safety and photocatalytic capability, titanium dioxide coatings are being applied as antibacterial coatings in both indoor and outdoor products. However, there is also concern that the coating may come off as a result of rubbing or wiping with a solvent like alcohol.

Energy Dispersive X-ray Fluorescence Spectroscopy (EDX) is effective at measuring titanium dioxide coatings because it does not require cumbersome sample preparation and can analyze samples nondestructively. The newly launched EDX-7200 comes standard with a series of primary filters that are effective for trace element analysis, and the newly installed filter is especially effective for the analysis of Ti to Co.

In this application note, we present examples of titanium dioxide photocatalytic coatings applied on resins and subsequently analyzed for coating amount before and after wiping with chlorine disinfectant and alcohol.

■ New primary filter

The use of primary filters can reduce characteristic and continuous X-rays scattered from X-ray tubes. The filters are useful for analyzing trace elements because of their ability to increase signal to noise. Fig. 1 shows the profile comparison of trace elements in oil with and without the EDX-7200's new primary filter (#5).
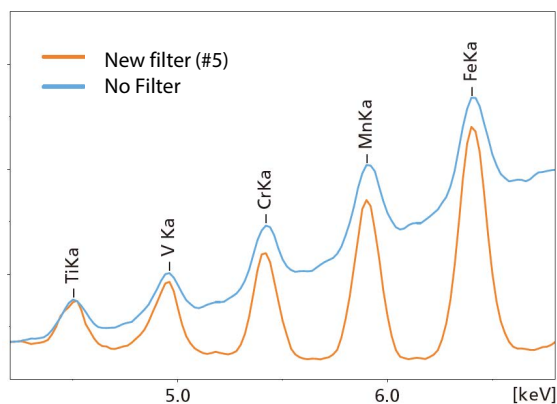
*Strength standardized for comparison.

Fig. 1 Profile Comparison of Trace Elements in Oil

■ Sample

Four samples were prepared as shown in Table 1. Fig. 2 shows the appearance of sample A.

Table 1 Sample Summary

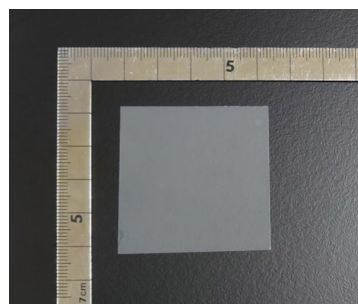| Sample name		Preprocessing content
Sample A	:	Titanium dioxide photocatalytic coatings applied to polypropylene (PP) sheets
Sample B	:	Wipe sample A with chlorine-based disinfectant
Sample C	:	Wipe sample A with ethanol
Uncoated sample	:	PP sheet only

Fig. 2 Appearance of Sample A

■ Sample

The samples were placed and measured in such a way that they were directly irradiated with X-rays on the coating surface without any pretreatment.

■ Ti profile

Samples A-C and uncoated sample were measured using the newly installed primary filter (#5). For comparison, we also performed measurements using a conventional filter (#2), which is also effective for analyzing trace amounts of Ti. Overlapped profiles are shown in Fig. 3 (a) and (b). It is evident that the signal to noise ratio of trace Ti is significantly higher on filter (#5) compared to filter (#2).
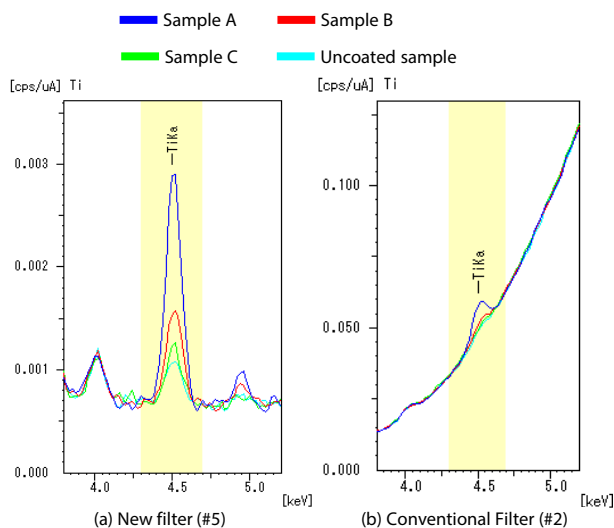

Fig. 3 Ti Kα Profile Overlapping

■ Detection Lower Limit

Table 2 shows the results of the coated sample measured 10 times using both the new filter (#5) and the conventional filter (#2). The titanium dioxide (TiO_2) coating amount was determined using the film FP method. From the standard deviations obtained, the detection limits for each filter are 0.0045 $\mu g/cm^2$ for the newly installed filter (#5) and 0.0132 $\mu g/cm^2$ for the conventional filter (#2). These results indicate that the newly installed filter (#5) is effective for measuring trace amounts of Ti.

Table 2 Repeatability of the uncoated sample

[$\mu g/cm^2$]

n	New filter (#5)	Conventional Filter (#2)
1	0.032	0.030
2	0.031	0.033
3	0.033	0.028
4	0.033	0.024
5	0.030	0.025
6	0.033	0.035
7	0.035	0.027
8	0.032	0.031
9	0.033	0.021
10	0.031	0.031
Mean	0.032	0.029
Standard deviation (σ)	0.0015	0.0044
Lower detection limit (3σ)	0.0045	0.0132

■ Quantitative analysis results

Quantitative analysis results of TiO_2 coating on samples A-C are shown in Table 3. The uncoated sample also contained trace amounts of Ti and therefore blank correction was performed to subtract the contribution from the uncoated sample. The results are shown in Table 3.

Table 3 Quantitative Analysis Results

[$\mu g/cm^2$]

Sample name	Adhesion amount of TiO_2
Sample A	0.121
Sample B	0.033
Sample C	0.005

■ Conclusion

Titanium dioxide photocatalytic coatings applied to PP sheets were evaluated by EDX for TiO_2 adhesion before and after wiping. From the results, it is clear that most of the coating material was removed by wiping.

Primary filters are effective for trace element analysis. The use of a new filter on the EDX-7200 allowed sensitive analysis of trace Ti before and after wiping.

■ Measurement conditions

Table 4 Quantitative Analysis Results

Equipment	:	EDX-7200
Element	:	$_{22}$Ti
Analysis group	:	Quantitative
Analytical method	:	Thin film FP method
Detector	:	SDD
X-ray tube ball	:	Rh target
Tube voltage	:	30 [kV]
Tube current	:	Auto [µA]
Collimator	:	10 [mmφ]
Primary filter	:	#5 (#2)
Atmosphere	:	Air
Integration time	:	300 sec
Deadtime	:	Up to 30 (%)

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