APPLICATION NOTE # XE-2012-3204

Analysis of Yellow Dyes with X-Calibur SDD EDXRF combined with professional Fundamental Parameter Software

ABSTRACT

Papilion dyes are used for coating of Polyester fibers. Such yellow dyes samples were analyzed by EX-Calibur SDD EDXRF. Quantitative elemental analysis was conducted by specialized Fundamental Parameter software, without the use of certified standards. The fraction (w/w%) of the polymer matrix (C H O) was also calculated by this software.

OBJECTIVES

1. Qualitative elemental analysis the yellow dyes
2. Analyze these samples quantitatively with specialized Fundamental Parameter Software
3. Calculate the % of the organic component, mostly the yellow dye compound, out of the overall sample weight

SAMPLES

Table 1: Sample list

#	sample ID	description
1	Dye 90S	Standard yellow dye
2	Dye 90D	Sample of yellow dye

BACKGROUND

EDXRF is an ideal analytical technique for qualitative and quantitative elemental analysis for industrial control purposes. This analytical techniques boasts of being extremely quick, noninvasive, and in most cases does not require any sample preparation. Using an "operator" user interface, the analysis can be automated to reduce the labor and minimize the staff training.

ANALYTICAL CONFIGURATION

Table 2: Analytical configuration of EX-Calibur SDD

Instrument	X-Calibur SDD EDXRF Analyzer
Excitation	Rh-Anode X-ray Tube, 50KV 50W
Detector	High Performance Silicon Drift Detector SDD.
Analysis Time	120 second
Type of analysis	Qualitative analysis of all elements
Environment	Vacuum, for detection of low atomic weight elements
Sample preparation	Samples were analyzed as obtained from the customer in X ray cups with Prolene thin film support
Acquisition conditions	High excitation energies without the use filters using 1mm (diameter) collimator inbuilt in instrument filter wheel.

Figure 1: X-Calibur SDD EDXRF analyzer

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December 2012

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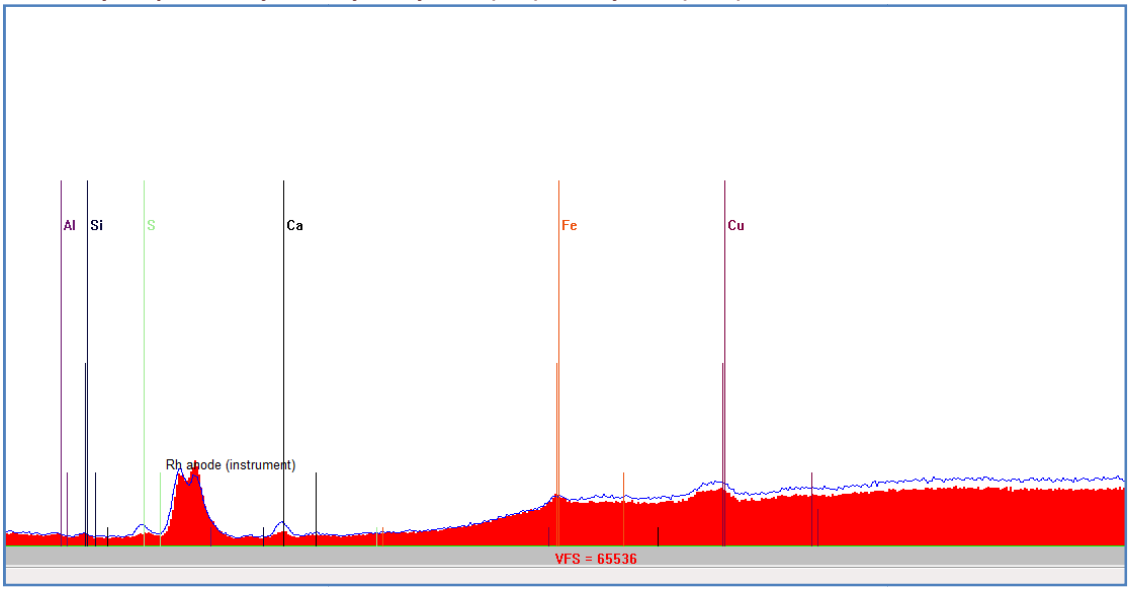
EXPERIMENTAL

Two yellow dyes, standard and sample (table 1), were analyzed qualitatively and quantitatively by X-Calibur SDD EDXRF. The spectra acquisition was performed with high excitation energies in vacuum in order to eliminate the oxygen in the X-ray beam path since otherwise the oxygen absorbs the low energy signal emitted by the low atomic weight elements. Collimator (D=1mm), inbuilt in the EX-Calibur analyzer filter wheel, was used during this analysis.
Quantitative elemental analysis was conducted with specialized Fundamental Parameter Software without the use of any standard. This software was also used for calculation of the fraction (w/w %) of the organic component (table 2).

RESULTS AND DISCUSSIONS

Figure 2 : Overlay of spectra of yellow dyes: Dye90D (red) and Dye90S (Blue)



Quantitative analysis in EDXRF is best done with regression analysis based on certified calibration standards of known concentrations and calibration curves per element of interest. Yet, in the absence of standards, Fundamental Parameter Software was used to calculate the element content and fraction of the organic matrix. This becomes necessary here, since the organic substances and yellow dye, occupies almost all the sample weight.
Both samples contain trace amounts of Al, Si, S, Ca, Fe and Cu; the amount of S and Ca in Dye90S is approximately three folds that of Dye90D.
In order to evaluate the sample homogeneity for the trace elements, 3 independent sample portions were analyzed for each Dye sample with proper statistical evaluation (Table 2).
The STD values obtained, indicate fairly homogenous distribution of all trace elements in the organic part of the sample.

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Table 2: Summary of quantitative results for Yellow Dyes Fundamental Parameter Method

Dye 90S

#	Filename	Filename Low MW elem. (C H O)	Al (w/w%)	Si (w/w%)	S (w/w%)	Ca (w/w%)	Fe (w/w%)	Cu (w/w%)
1	DYE-90S-1	99.933	0.025	0.014	0.011	0.014	0.003	0.0003
2	DYE-90S-2	99.915	0.037	0.021	0.011	0.013	0.003	0.0004
3	DYE-90S-3	99.923	0.03	0.021	0.009	0.012	0.004	0.0002
	average	**99.924**	**0.031**	**0.019**	**0.010**	**0.013**	**0.003**	**0.0003**
	STD	0.009	0.006	0.004	0.001	0.001	0.001	0.0001

Dye 90D

#	Filename	Filename Low MW elem. (C H O)	Al (w/w%)	Si (w/w%)	S (w/w%)	Ca (w/w%)	Fe (w/w%)	Cu (w/w%)
1	Dye-90D-1	99.936	0.028	0.024	0.003	0.005	0.003	0.0003
2	Dye 90D-2	99.946	0.022	0.019	0.003	0.006	0.003	0.0003
3	Dye 90D-3	99.941	0.020	0.030	0.003	0.004	0.003	0.0003
	average	**99.941**	**0.025**	**0.022**	**0.003**	**0.005**	**0.003**	**0.0003**
	STD	0.005	0.004	0.004	0.000	0.001	0.000	0.0000

CONCLUSIONS

This work demonstrates the excellent performance of Xenemetrix made EX-Calibur SDD EDXRF combined with Advanced Fundamental Parameter Software to analyze Papilion yellow dyes.
Quantitative elemental analysis was successfully conducted and the fraction of the polymer matrix was calculated. The X-Calibur SDD analyzer is capable of analyzing most inorganic elements in organic matrix (F to Fm) quickly and easily without any sample preparation; therefore it is the most suitable analytical technique for QC labs and reverse engineering study.

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