Detection of 23 sensitizing and carcinogenic dyes in textiles by LC-MS/MS

Liu Qing[1],Xiong[2], Xengxing[1], Yang Zong[1], Liu Bingjie[1], Guo Lihai[1]
[1]SCIEX Asia Pacific Application Support Center, Shanghai, China; [2]Food inspection and testing, Jiangxi province, China

ABSTRACT

A method for the detection of 23 allergenic and carcinogenic dyes using both positive and negative ionization mode was developed. The method was applied to the measurement of the 23 dye compounds in 6 textiles representing several different categories. Compared with the LC-UV method specified in the standardized method, this procedure is faster and easier, saving time and effort.

INTRODUCTION

Carcinogenic and allergenic dyes can induce cancer and allergic reaction in humans. These dyes are thought to potentially cause cancer, or induce allergy in skin, respiratory tract or mucous membrane in humans and animals. At present, methods for detection of carcinogenic and sensitizing dyes mainly use LC with UV detection. The detection limits for LC-UV are generally high, and there can be significant matrix interferences which potentially impact data quality. In contrast, high performance liquid chromatography coupled with triple quadrupole mass spectrometry (LC-MS/MS) has greater sensitivity and selectivity. Triple quadrupole mass spectrometry mainly scans specific precursor and fragment ion pairs through multiple reaction monitoring (MRM) mode, which greatly reduces interference and provides accurate qualitative and quantitative analysis.[1]

MATERIALS AND METHODS

Sample preparation: All samples were cut into pieces of approximately 5 g. Samples were extracted using a solution of 80% acetonitrile/water (v/v).

HPLC conditions: A Shimadzu Prominence LC system was used with a Waters HSS T3 C18 column (2.1 x 100 mm, 1.7 µm, 100 Å, column oven at 40°C). The mobile phases were A) 5 mM ammonium acetate with 0.1% formic acid in water, and B) acetonitrile. The flow rate was 300 µL/min and the injection volume was set to 10 µL.

MS/MS conditions: A SCIEX Triple Quad 4500 system with Turbo V ion source and electrospray ionization (ESI) probe was used. The MS source conditions were as follows: curtain gas (CUR), 40 psi; collision gas (CAD), medium; nebulizing gas (GS1), 40 psi; heater gas (GS2), 40 psi; ion spray voltage (IS), 5500V and -4500V in positive and negative mode, respectively; source temperature, 550°C. (Figure 1).

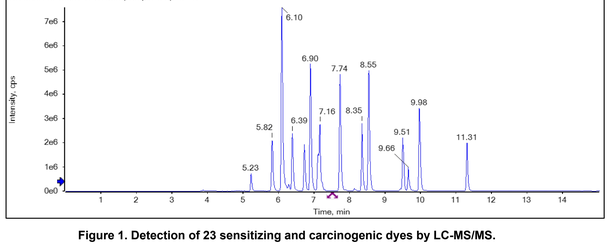

Figure 1. Detection of 23 sensitizing and carcinogenic dyes by LC-MS/MS.

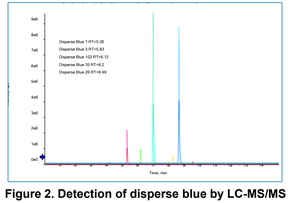

Figure 2. Detection of disperse blue by LC-MS/MS

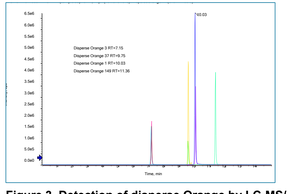

Figure 3. Detection of disperse Orange by LC-MS/MS.

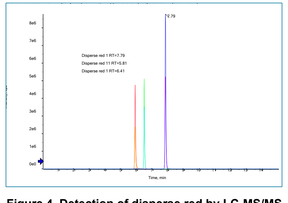

Figure 4. Detection of disperse red by LC-MS/MS.

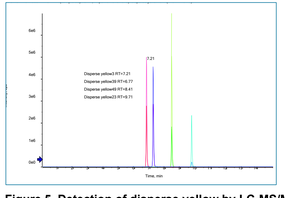

Figure 5. Detection of disperse yellow by LC-MS/MS.

RESULTS

The 23 allergenic and carcinogenic compounds were detected with a single run using LC-MS/MS, comprising 4 unique classes of sensitizing and carcinogenic dyes such as disperse red, disperse blue, disperse green, and disperse yellow **(Figure 2- 5)**. This method was used to analyze 6 textiles from different categories. The LOQ, RSD (n=6) and linear dynamic range of sensitizing and carcinogenic dyes was shown to meet the needs of textile testing **(Table 1, 2)**.

Table 1. LOQ, RSD (n=6) and linear dynamic range of sensitizing and carcinogenic dyes.

Compound	LOQ (µg/kg)	Linear Range (µg/kg)	RSD(n=6)
Disperse Blue	1	1-100	2.15
Disperse Blue 3	0.1	0.1-100	1.67
Disperse Blue 7	0.1	0.1-100	2.31
Disperse Blue 26	0.1	0.1-100	2.12
Disperse Blue 35	0.5	0.5-100	2.23
Disperse Blue 102	0.1	0.1-100	1.67
Disperse Blue 106	0.1	0.1-100	1.89
Disperse Blue 124	0.1	0.1-100	1.98
Disperse orange 1	0.1	0.1-100	2.01
Disperse orange 3	0.1	0.1-100	2.32
Disperse orange 37/76/59	0.1	0.1-100	1.99
Disperse red1	0.1	0.1-100	2.05

Table 2. LOQ ,RSD (n=6) and linear dynamic range of sensitizing and carcinogenic dyes.

Compound	LOQ (µg/kg)	Linear Range (µg/kg)	RSD(n=6)
Disperse red11	10	0.1-100	2.02
Disperse red17	10	0.1-100	1.23
Disperse yellow1	10	0.1-100	2.05
Disperse yellow3	10	0.1-100	1.78
Disperse yellow9	10	0.1-100	1.03
Disperse yellow39	10	0.1-100	2.32
Disperse yellow49	1	0.1-100	2.13
Disperse brown1	5	0.1-100	1.97
Disperse orange149	5	0.1-100	1.95
Disperse yellow23	5	0.1-100	1.45
Malachite Green	1	0.1-100	1.52

Optimized chromatography provided good separation for the 23 allergenic and carcinogenic dyes, acquired using both positive and negative ionization modes **(Figure 6).** The linearity of the standard curves was good (R^2> 0.999) **(Figure 7).**

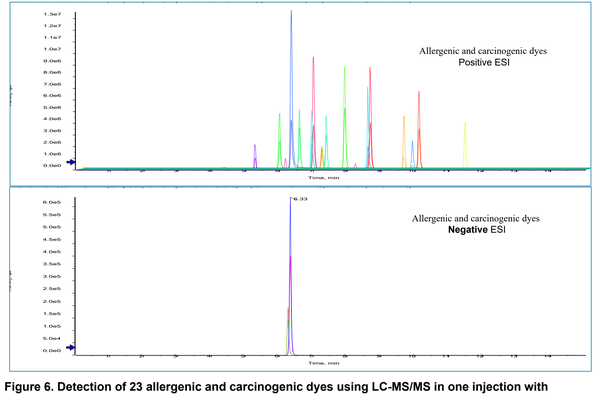

Figure 6. Detection of 23 allergenic and carcinogenic dyes using LC-MS/MS in one injection with positive and negative ion polarity.

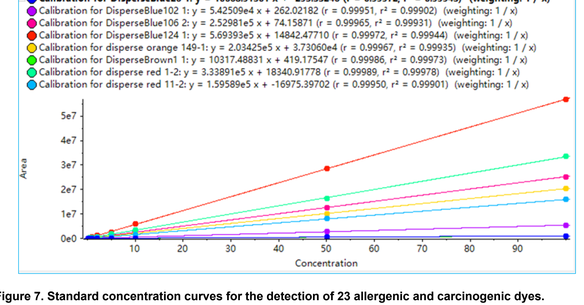

Figure 7. Standard concentration curves for the detection of 23 allergenic and carcinogenic dyes.

To monitor recovery, 2.5 ng of the 23 analytes was spiked onto blank textiles samples. The recovery rate was found to be between 91.2 and 110.1% across all compounds **(Figure 8).**

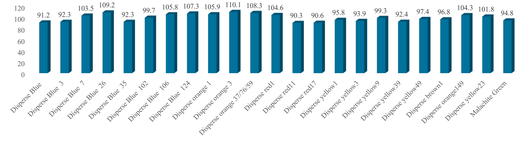

Figure 8. Recovery rate for 23 sensitized carcinogenic dyes was between 91.2 to 110.1%.

CONCLUSIONS

The developed method was been successfully applied for the accurate quantification of 23 allergenic and carcinogenic compounds in 6 textiles from different categories. Compared with the LC-UV technique specified in the standard method, 23 kinds of allergenic and carcinogenic dyes were detected across positive and negative modes, saving time and resources. In addition, this method analyzed more compounds with higher sensitivity and precision.

This method fully meets the testing requirements of GB/T 18885-2009 Technical Requirements for Ecological Textiles.[2]

REFERENCES

1. Simultaneous qualitative and quantitative determination of Disperse Yellow 3 in dyes by HPLC-MS/MS-LIQ method .Printing and Dyeing（2021 No.3.）
2. China national standard: Technical specifications of ecological textiles - GB/T 18885-2009.

TRADEMARKS/LICENSING

To receive a copy of this poster:

- Scan the code with your phone camera
- Complete the form