Quantification of alkylphenols ethoxylate in textiles by HPLC/MS/MS

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Introduction

Alkylphenols ethoxylate (APEO), part of the great alkylphenol's family, are compounds used in the textile industry as surfactant. The REACH program regulates the use of these compounds by including them in Annex XVII, thus prohibiting the commercialization of articles with a concentration greater than 0.1%. Moreover, they are known as endocrine disruptor for animals but also for humans. Therefore, following the publication of Greenpeace « Dirty Laundry », the problem of APEO's presence in textile has been brought into focus, forcing textile manufacturers to control their products further. Thereby, in order to meet the needs of industrial customers and also to have a reliable method of analysis, it was necessary for CTC to develop a test method for the quantification of APEO in textile.

Experimental methods

The method used was based on the Project Standard CEN/TC248/ N201. The global approach used was the Liquid Chromatography coupled with Mass Spectrometry as analytical method for the analysis of a mixture of two standard mixtures containing different APEO:
- One containing 15 OPEO (Octylphenols Ethoxylate)
- Other containing 17 NPEO (Nonylphenols Ethoxylate)

These solutions served only to make the optimization of the method by playing on the various parameters linked to the analysis and to make the calibration solution for the quantification.

After diverse assays, the final method's choice, in order to have the best sensitivity, was the SIM mode (Single Ion Monitoring) with a Capillary Voltage of 5000 V and different fragmentor values for the 32 ions. This value was determined by comparing the intensity of each peak for different values of fragmentor. An example of this comparison for one ion is shown in Figure 1.

And the elution condition for the chromatography section presents the following parameters:

-column:Poroshell 120 Bonus RP
-Length: 150 mm
-Internal Diameter: 4.6 mm
-Particle size: 2.7 µm
-Temperature of the column: 40°C
-Flow rate: 0.3 mL/min
-Injection Volume: 10 µL

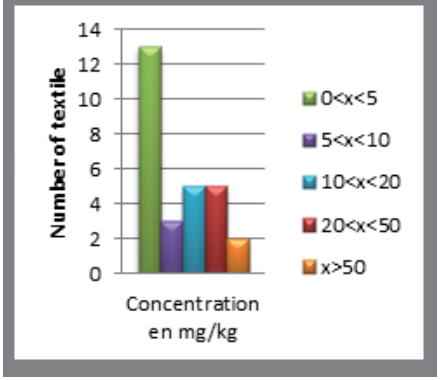
Time (min)	%A	%B
0	50	50
1	50	50
2	2	98
10	2	98

Table 1: Description of the gradient elution

See table 1 for the gradient elution used, with Ammonium Acetate pH 3.6 (A) and acetonitrile (B).

The instrumental method is now applied to 50 real samples to quantify APEO present in textile. First of all, compounds of interest are extracted with methanol by ultrasonic bath at 40°C for one hour. Then they are analyzed the by the LC/MS/MS method described above.

Figure 1 : Proportion of textiles according to their APEO composition



Results and discussion

As it can be seen from Figure 1, more than half of the samples contain APEOs. Moreover, all have a concentration less than 1000 mg/kg. So they are in compliance with the law. To conclude this method can be applied to real samples and allows the quantification of Alkylphenols ethoxylate in textile. In this way, CTC has a protocol for the analysis of these compounds.

Conclusion

The application of technical analysis to real samples was used to quantify the APEO present in textiles. This work will be continued to assess the repeatability and accuracy of the method. The company CTC has now a set of reliable data and information to help set an official standard for the study of APEO.

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