 Quantitative Determination of Volatiles in Polymers and Quality Control of Recycled Materials by Static Headspace Techniques

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Abstract A presentation is given of headspace (HS) extraction and headspace solid-phase microextraction (HS-SPME) techniques and their combination with multiple headspace (MHS) extraction to enable quantitative determination of volatiles in solid polymer matrices. As an example, the development of HS, HS-SPME, and MHS-SPME methods for...
extraction of volatiles from thermo-oxidized and/or recycled polyamide 6.6 is reviewed with special focus on the problems encountered when extracting analytes from solid-sample matrixes including excessively long equilibrium times and adsorption of analytes to the sample matrix. Examples are also given of the application of HS-SPME in quality control of recycled materials, in durability assessment of polymeric materials and in degradation studies.

**Keywords** Degradation · Multiple headspace extraction · Quality control · Recycling · Solid-phase microextraction

**Abbreviations**

ASTM American Society for Testing Materials  
BTEX Benzene, toluene, ethylbenzene, xylene isomers  
CAR Carboxen  
CW Carbowax  
DSC Differential scanning calorimetry  
DVB Divinylbenzene  
DMF Dimethyl formamide  
FTIR Fourier transform infrared spectroscopy  
GC Gas chromatography  
HS Headspace  
IR Infrared spectroscopy  
LC Liquid chromatography  
MAE Microwave-assisted extraction  
MHE Multiple headspace extraction  
MHS Multiple headspace  
NMR Nuclear magnetic resonance spectroscopy  
PA Polycrylate  
PA 6 Polyamide 6  
PDMS Polydimethylsiloxane  
PP Polypropylene  
RSD Relative standard deviation  
SDME Single-drop microextraction  
SPME Solid-phase microextraction  
SFE Supercritical fluid extraction  
TGA Thermogravimetric analysis  
TVOC Total volatile organic content  
USE Ultrasound extraction  
UV Ultraviolet spectroscopy

1 **Introduction**

Plastic products contain residual monomers, by-products from polymerization, additives and degradation products. The low-molecular-weight compounds will in time migrate from the polymers into the surrounding environment. Thus, the knowledge of the amounts and identities of these compounds