# DETERMINATION OF NON HALOGENATED SOLVENTS IN INDUSTRIAL WASTEWATER USING SOLID PHASE MICROEXTRACTION (SPME) AND GC-MS

V. LÓPEZ-GRIMAU*, J.M. GUADAYOL**, J.A. GRIERA* and M.C. GUTIÉRREZ*

*INTEXTER (Institut d'Investigació Tèxtil), Universitat Politècnica de Catalunya, Colom 15, 08222 Terrassa, Spain. lopez@etp.upc.es

**Dpt.d'Enginyeria Química, Universitat. Politècnica de Catalunya,.Colom 1, 08222 Terrassa, Spain. jose.maria.guadayol@upc.es

*Abstract*— **Solid phase microextraction (SPME) was applied to the analysis of a selected group of non halogenated solvents -all of them classified as volatile organic compounds (VOCs)- that may be present in diverse industrial wastewaters. Two different fibres (100-PDMS and 85-PA) were selected as SPME adsorbents. The extracted volatile organic compounds were analysed by high resolution gas chromatography coupled to mass spectrometry (GC-MS), following the EPA method 624. Calibration curves and method detection limits obtained for these solvents using the two SPME fibres are reported. The 100-PDMS fibre provided better sensitivity, but the saturation was reached at a lower concentration than 85-PA fibre.**

**The first fibre (100-PDMS) was applied to the analysis of several textile wastewaters. The method detection limits obtained with this fibre were: 0.1-0.3 µg/L for ethylbenzene, m-xylene, p-xylene, toluene and diisobutyl ketone. In addition, SPME and Headspace (HS) detection limits were compared.**

*Keywords*— **Solid phase microextraction (SPME), gas chromatography-mass spectrometry (GC-MS), wastewater, industrial solvents, VOCs.**

## I. INTRODUCTION

In recent years, there has been a remarkable growth in the number of organic compounds used as solvents for several industrial activities, such as synthesis, extractions, cleaning and protection of surfaces, etc. In general, the industrial processes requiring the use of these compounds include recovery of the used solvents. However, this recovery is hardly ever complete, so part of the solvent is incorporated into the process effluents.

Solvents can be divided into two classes according to their chemical composition: halogenated and non halogenated. In general, the use of halogenated solvents is severely restricted by the regulations of different countries and also by the European Community regulations (Council Directive 89/678/EEC, 1989; Council and European Parliament Directive 94/60/EEC, 1994; Commission Directive 96/55/EC, 1996), since they represent a danger to health and environment.

Non halogenated solvents are increasingly used in industry because of their lower toxicity. However, non halogenated solvents also involve toxicity problems and for this reason, their content in wastewater is also restricted. Moreover, it is important to identify these solvents in industrial wastewater because they could alter the operation of biological treatment plants. Most of these solvents have harmful effects on the environment and are considered priority pollutants by the US Environmental Protection Agency (US-EPA, 2005). They are generally referred as volatile organic compounds (VOCs), which are defined, from a chemical point of view, as organic compounds that have a boiling point ≤100 ºC and/or a vapour pressure >1 mm Hg at 25 ºC. However, the most accepted environmental definition was formulated by the United Nations Commission in 1991 and presented VOCs as organic compounds, different from methane, able to produce photochemical oxidants, mainly ozone, by reaction with nitrogen oxides in the presence of solar light. This category of compounds includes thousands of pollutants; which may have a variety of harmful health effects. At high levels of exposure, many VOCs can cause central nervous system depression. All of them can be irritating upon contact with the skin, or to the mucous membranes when they are inhaled (Jennings and Sneed, 1996).

The most common non halogenated solvents and their main industrial applications are reported in Table 1. Most of them are used as solvents for coatings, resins, paints, plastics, adhesives, printing inks, cosmetics. In the textile industry, they are intermediates in dye synthesis and may also appear as traces or additives in dyes, dye carriers, raw materials and wet textile processing. In pharmaceutical industry, they are mainly employed for synthesis and extraction processes. Another group of compounds is used in the manufacture of plastic fibres, synthetic rubber, artificial leather, photographic films, flavours, perfumes, insecticides, etc (National Safety Council,2004; Spectrum Laboratories Inc., 2004).

In the present work, diverse textile effluents treated in different biological plants were analysed to establish their content in non halogenated solvents. High resolution gas chromatography coupled to mass spectrometry (GC-MS) was employed, following EPA method 624.