Development of a new micro reaction sampler for pyrolysis-GC/MS system facilitating on-line analytical chemolysis of intractable condensation polymers

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Abstract:

A new micro reaction sampler is developed to facilitate the thermally assisted hydrolysis and methylation (THM) reaction of intractable condensation polymers, at an elevated temperature in a sealed glass capsule under high pressure. The sampler is integrated with a glass capsule and a crushing metal rod, and it is air-tightly mounted inside a vertical micro-furnace pyrolyzer of a pyrolysis (Py)-GC/MS system. The developed sampler is firstly applied for THM-GC/MS measurements of polycarbonate (PC) whose structural characterization using tetramethylammonium hydroxide (TMAH) as a reagent has been reported in detail previously, to confirm the applicability of the developed sampler. Two peaks are clearly observed in the pyrogram and they are assigned to methyl derivatives of a main chain unit (bisphenol A) and a terminal group (*tert*-butyl phenol) which are the THM reaction products of PC. The area ratio of these two peaks is almost the same as that obtained by the conventional THM-GC/MS, suggesting the effectiveness of the developed reaction sampler. Further, the developed sampler is applied for THM of nylon 6.6 which has been hardly analyzed by conventional THM-GC/MS using TMAH as a reagent in the open system where samples are exposed to the ambient gas in a pyrolyzer. Using the developed method, measured pyrogram clearly shows several peaks with fairly high yields that are assigned to methyl derivatives of monomer units of nylon 6.6, that is, adipic acid and hexamethylenediamine, and a hybrid dimer of each monomer. Therefore, it can be said that the developed sampler based on the closed system, sealed glass capsule, is quite useful for the pyrolysis analysis of intractable condensation polymers such as nylon 6.6, compared to the conventional THM-GC/MS using the open system.

Frontier Labs products used:

Multi-Shot Pyrolyzer, On-line micro-reaction sampler, UA$^+$-5