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APPLICATION OF COMBINED THERMAL ANALYSIS - MASS SPECTROMETRY IN THE ANALYSIS OF FOSSIL FUELS AND POLYMERS

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ABSTRACT

Several standard coal samples of different coal rank have been investigated by means of simultaneous Thermogravimetry - Derivative Thermogravimetry-Differential Thermal Analysis - Mass Spectrometry (TA-MS). Additionally the thermal degradation of polyvinyl chloride and polymethane foam has been examined exemplarily with regard to the evolution of toxic compounds.

INTRODUCTION

Thermal analysis of fossil fuels in combination with mass spectrometry has been proved to yield a lot of informations which can be useful not only with respect to coal liquefaction and coal gasification as a resource of energy but to the composition of their devolitilization products with regard to environmental pollution.

Some main objectives concerning with TA-MS analysis have been described in our previous papers [1-3]. This paper deals with the results of our current studies investigating mineral coal samples.

The PU and PVC samples have been chosen to test the applicapability of the TA-MS method in the field of polymer degradation processes.

EXPERIMENTAL

The TA-MS experiments have been performed using a Netzsch STA 429 Thermal Analyzer coupled with a Balzers QMG 511 Quadrupole Mass Spectrometer. Detailed information about the instrumental equipment and experimental procedure has been reported [3-5]. The standard coal samples of different coal rank have been made available for us by the 'Coal mining Research Institute at Essen' F.R.G.

The PVC materials has been an original industrial product while the PU foam has been prepared especially for our investigations in a chemical plant.

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RESULTS AND DISCUSSION

The coal samples ranging from an anthracite up to typical gas flame coals with nearly 40 % volatile matter content dry and ash free have been heated up to final temperatures of 1500 K applying different gas atmospheres at standard pressure. The slope of the TG-DTG-DTA curves is strongly influenced by the sample size as well as their characteristic temperatures. Sample sizes below 0.1 mm diameter have been proved very useful avoiding spontaneous exploding of the particles which results in non reproducible TA data. The mass spectra show no differences correlating with coal rank when the experiments have been carried out in air or oxygen containing gas mixtures especially in the higher mass range. High vacuum operation yields more reproducibility of the data and high ion yields of compounds like the permanent gases as well as derivates of typical hydrocarbons series (phenoles, benzenes, naphtalines, dihydroxy benzenes, anthracenes, diphenyles etc.).

The simultaneously recorded intensities of the ion currents corresponding with mass fragments released from the coal samples show significant dependence of magnesia oxide added in different amounts to dilute the sample material. From the TA-MS experiments it can be drawn during one simultaneous run that PVC decomposes in a four stage degradation mechanism. The predominant portion of hydrogen chloride is formed at very low temperatures which can be ensured by detection of the ^{35}Cl, ^{37}Cl isotopes in $H^{35}Cl$ and $H^{37}Cl$ in correlation of their natural abundance as a 3:1 ratio.

PU-foam containing the flame retardant tetrakis (2-chloroethyl) diphosphate decomposes in oxidative atmosphere at standard pressure in one rapid reaction. Several high toxic species are formed and detected by the mass spectrometer. The most striking fact is the very early generating of vinyl chloride from the flame retardant, while hydrogen cyanide is produced at much higher temperatures.

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