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TA-MS INVESTIGATIONS OF COAL WESTERHOLT AND ITS RELATED ASPHALTENE AND ASPHALTOLE

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ABSTRACT

For the investigation of coal westerholt (39,7 % v.m.d.a.f) and its related asphaltene and asphaltole, quasi static air was applied as well as dynamic argon atmosphere. The flow rate was adjusted to the leak rate of the ceramic orifice system.
The simultaneous TA-MS spectrometric investigations yielded the complete oxidation of the samples producing (CO_2, H_2O, SO_X, NO_X) in quasi static air. Applying dynamic argon mass fragments (m/z > 64 amu) were detected. The asphaltene and asphaltole samples degradate into mass fragments in the lower mass range (m/z $\leq$ 44 amu).

INTRODUCTION

Asphaltenes and asphaltoles are products of the coal-liquification process, which can be distinguished by their soloubility in different solvents (1,2).
The products were characterized performing traditional analytical- and spectroscopic methods (4 - 7).
Only a few puplications are dealing with the investigation of the thermal behavior of these materials. We have been the first who have carried out simultaneous TA - MS experiments on coal samples.

EXPERIMENTAL

The experimental procedure and instrumentarly equipment has been described previously (8 - 12). The samples, as mentioned before, have been additionally investigated as a 1:1 mixture with magnesium oxide in aluminium oxide crucibles as well as on an aluminium oxide plate, wich results in a greater surface distribution of the sample material

RESULTS AND DISCUSSION

Thermoanalytical investigations

Performing the TA-MS experiments in air an increase of mass of each sample have been observed, followed by the oxidative degradation (see fig. 1).
This effect of mass increase can be explained by incorporation of oxygen into the molecular structure of the samples without evolution of volatiles. This

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This hypothesis can be supported by comparing the results of the investigations applying dynamic argon, which shows only normal decomposition of the samples (see fig. 2)

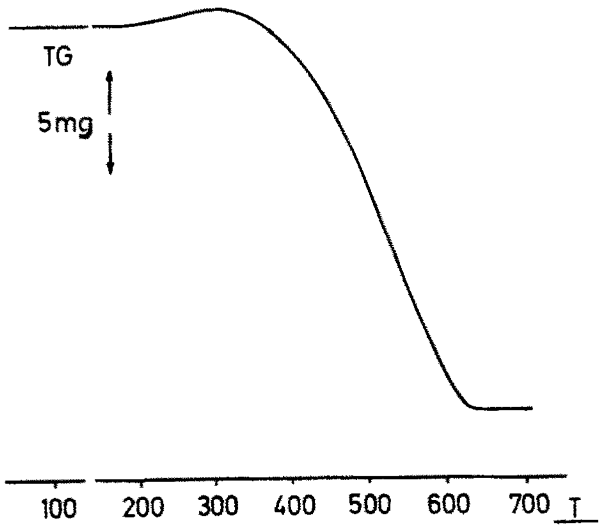

Fig. 1: TG - curve of coal Westerholt in quasi static air atmosphere

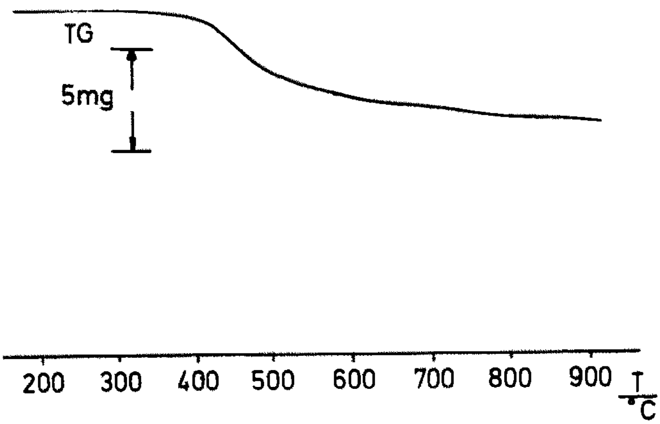

Fig. 2: TG - curve of coal Westerholt in dynamic argon atmosphere

Heating the coal samples in air atmosphere leads to different mass changes, ranging from 90 % to 95 % depending on the additional amount of magnesium oxide and on the surface distribution. The mass loss of the asphaltene and asphaltole fractions have been 100 % performing the same experimental conditions. This is due to the absence of the mineral matter of the coal.

The investigations applying dynamic argon atmosphere show clear differences in decomposition mechanism. The decomposition process of coal and asphaltene occurs by a two stage mass loss. The first step is relatively sharp and leads to a mass loss of 25 % (coal) e.g. 40 % (asphaltene). The decomposition of the asphaltole occurs nearly monotonous, accompanied by a very weak DTG - peak. The degradation in argon is comparable with the oxidative decomposition.

Evaluating the DTA - curve shows that during these meassurements oxigen is absent, no exothermic peak was recorded.

The TA-MS experiments applying oxidative atmosphere yield out fragments in the lower mass range up to 64 amu. The samples will be completely oxidized to H_2O, CO_2, NO_x and SO_x. Mixing the samples with MgO the mass fragment 46 (NO_x) could not be detected, but SO_2 (64 amu) is produced in high concentration showing two maxima. The first maximum of ion current intensity corresponds with the normal oxidativ decomposition, the second maximum, which appears at higher temperatures, can be attributet to decomposition of $MgSO_4$ formed during thermal treatment. Table 1 shows the maxima of the evolved Nitrogendioxide and Sulphurdioxide.

TABLE 1

Maxima of NO_2 and SO_2 emission

		m/z = 46	m/z = 64	
coal Westerholt	(crucible)	860 K	700 K	
	(plate)	770 K	730 K	
	(with MgO)	-----	700 K	920 K
asphaltole	(crucible)	900 K	900 K	
	(plate)	680 K	680 K	
	(with MgO)	-----	760 K	1130 K
asphaltene	(crucible)	960 K	740 K	
	(plate)	850 K	810 K	
	(with MgO)	-----	1050 K	1130 K

Applying dynamic argon atmosphere, the coal Westerholt only yield mass fragments greater then 64 amu. The fragments of asphaltene and asphaltole ar less or equal 44 amu. In fig. 3 the mass spectra of coal Westerholt, asphaltene and asphaltole are exhibited.

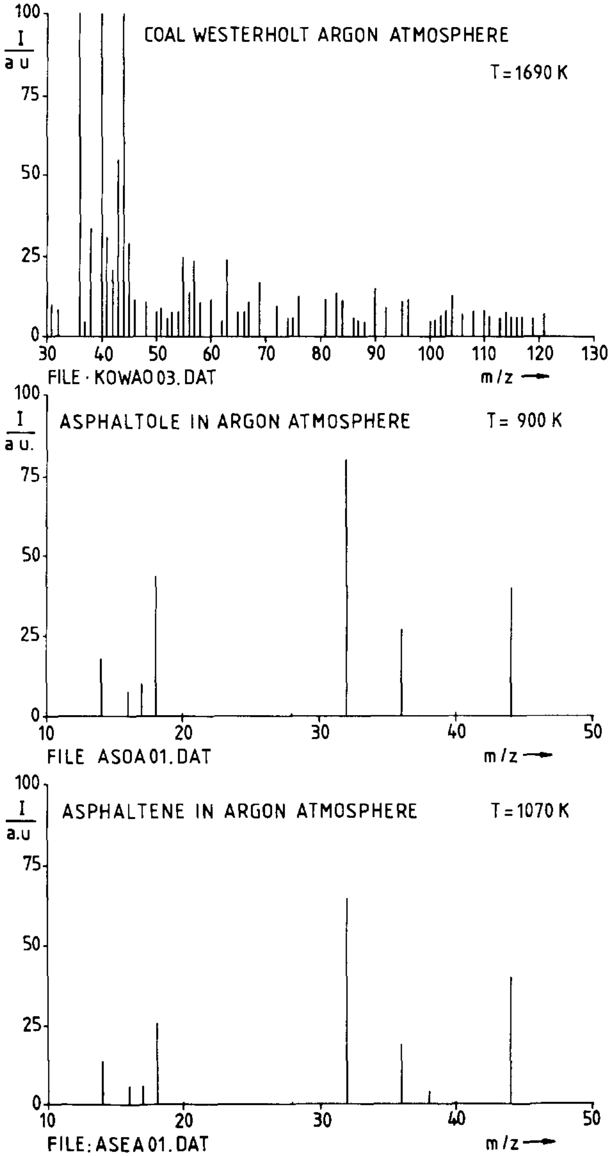

Fig. 3: mass spectra of the samples in argon

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