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THE STUDY OF COAL BY THERMAL ANALYSIS - THE EFFECT OF MEASURING CONDITIONS ON THERMAL ANALYSIS RESULTS

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ABSTRACT

The article presents a brief review on the use of thermal analysis in coal investigations. Thermal analysis curves obtained by various authors have been compared and it has been shown that there are great divergences in the results. In the present study DTA, TG and DTG curves of different amounts of ground coal with different grain sizes were recorded. The shapes of the DTA curves, the exact locations of the various exothermic peak maxima, the total weight loss and the temperature with the maximum rate of weight loss, depended on the degree of grinding (grain size) and on the size of the sample employed. It was concluded that it would be necessary to adopt a standard methodology for the characterization of coal by thermal analysis studies.

INTRODUCTION AND LITERATURE SURVEY

Thermal analysis is widely used for the study of inorganic and organic materials. In general, DTA curves of inorganic substances are more clear than those of organic substances, including solid fuels, and the various peaks are interpretable. Hollings and Cobb were pioneers in the field of investigation of solid fuels by DTA and their publications from 1914 and 1915 are cited in ref [1]. By carrying out thermal analysis in an inert atmosphere (under nitrogen) they were able to determine the exact stage of coal decomposition.

A relationship can be observed between the degree of coalification and the shape of the DTA curves. Glass [2, 3] carried

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out extensive studies with the purpose of characterizing the degree of coalification by DTA. Some examples of his DTA curves are shown in Fig. 1(A). A correlation between the shape of the curves and the well known coking properties of the samples can be used to give the information as to what degree the coal has undergone carbonization. He also found a relationship between the value of coal plasticity

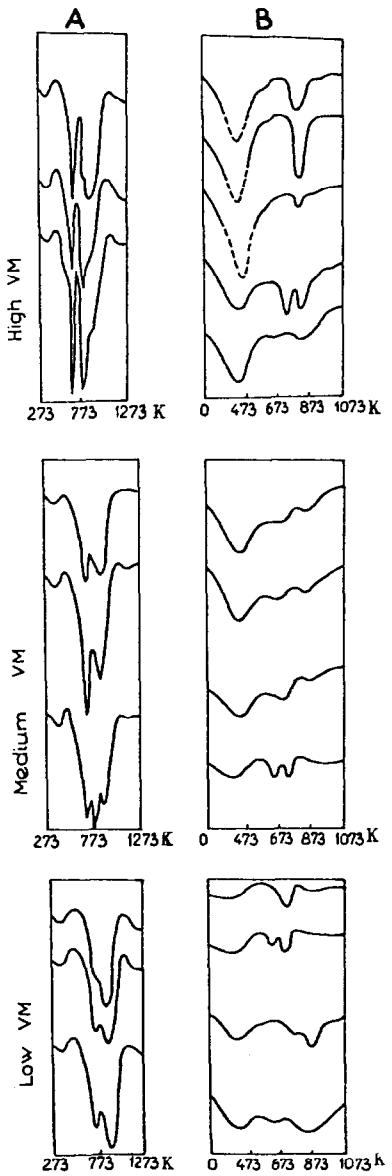

Fig. 1. Comparison between DTA curves of bituminous coal obtained by different investigators,
(A) - after Glass [2, 3];
(B) - after Heilpern [12].

(according to Giesseler) and the characteristic peaks of DTA curves. Similar investigations were carried out by Boyer and Payen [4]. According to Kessler and Romovackova [5] it is possible to distinguish between the following grades of coal: lignite, brown coal, bituminous coal and anthracite. A thermal analysis investigation of brown coals and their components was carried out by Martyniuk and Tomkow [6].

In Fig. 2 DTA curves of some Polish brown coals obtained by several investigators, are compared. The thermal decompositions of brown coals mined in different regions are similar. Ruscev and Dragostinov [7] compared the DTA curves and infrared spectra of the coals. Some DTA curves obtained by Ruscev et al. [8, 9] are shown in Fig. 2(A). Khemchandani and Sarkar [10] studied different types of coal using DTA combined with thermogravimetry (Fig.2(C) and Table 1). They concluded that the maximum decomposition rate of the organic substance V_m expressed in percentage per degrees centigrade is dependent on the content of volatile matter and that the higher the volatile matter content, the higher will be the maximum decomposition rate. However, when analyzing Table 1 one may disagree with these authors since not all the data support this conclusion, showing that the decomposition process is rather complicated.

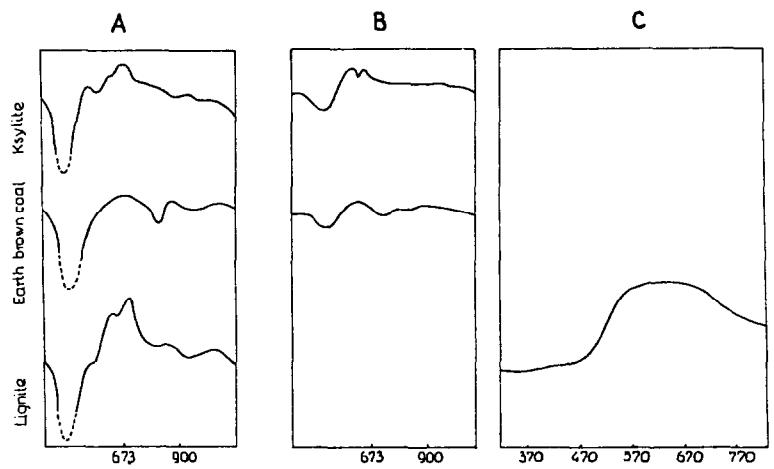

Fig. 2. Comparison between DTA curves of brown coals obtained by different investigators, (A) - after Ruscev [8, 9];(B) - after Martyniuk and Tomkow [6]; (C) - after Khemchandi and Sarkar [10].

TABLE 1
Dependence of the maximum decomposition rate on volatile matter content in coal (after Khemchandi and Sarkar [10]).

Volatile matter	Maximum rate of decomposition	Temperature range of decomposition reaction
V_{daf}	%-deg^{-1}	deg
37.9	0.082	88
46.8	0.328	43
48.0	0.132	68
48.1	0.248	28
51.2	0.156	68
66.1	0.472	49
66.7	0.272	58

Cypres et al. [11] constructed a special DTA apparatus for investigating coals. The apparatus operates under pressure by using different gases. The effects of pressure and atmosphere on the nature of the DTA curves are shown in Fig 3.

Heilpern [12] used thermal analysis to investigate Polish bituminous coals embracing the whole coalification degree ranging from 31 to 38 (according to Polish (further) classification). Some representative DTA curves are shown in Fig 1(B). There is a correlation between the coalification degree and the temperature of the maximum DTA effect, the latter shifts to higher temperatures with increasing degree of coalification. Heilpern attempted to relate the results of thermal analysis with the plastic properties and approximate analysis of the coals.

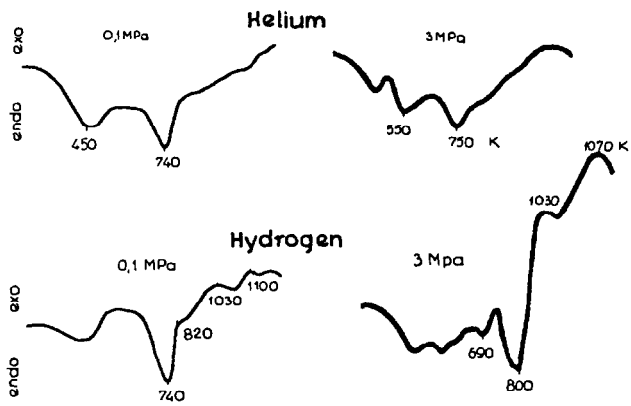

Fig. 3. DTA curves of coal recorded under helium and hydrogen atmospheres (after Cypres et al. [11]).

From the above quoted references it seems that the different curves obtained for identical coals may be due to differences in the measuring conditions such as the heating rate, the sample size, the kind of holder, the manner of filling the holder, the rate and kind of flowing gas. In the article published in the Pre-Congress Proceedings of 9 ICTA in Jerusalem [13] it was shown that different

DTA curves of identical coal samples were obtained by using different instruments and by changing the atmosphere. That work was enlarged and in the present article we are going to show that additional factors play an important role in affecting the nature of the DTA curves of coals. These are the size of the sample, as well as the degree of grinding, which is shown by the size of the grains of the coal.

EXPERIMENTAL

The investigation was carried out with a Polish brown coal from Lubstow Coal Mine. The nature and properties of the coal were previously described [13]. The ground coal was separated into three different mixtures, according to particle size, these were below 0.2 mm, between 0.2 and 0.5 mm and between 0.5 and 1.0 mm. DTA, TG and DTG curves were obtained by using the derivatograph OD-102 (MOM Budapest). Sizes of samples used for each run were 300, 400 and 500 mg. The amount of coal employed in each run was limited due to the size and geometry of the ceramic sample holder.

RESULTS AND DISCUSSION

Taking the above into consideration an attempt was made to study the effect of coal grinding (particle size) and the size of the sample on the nature and shapes of the DTA, TG and DTG curves. All DTA curves recorded under a dynamic air atmosphere show exothermic reactions. Fig. 4 shows that the shapes of the DTA curves and the exact locations of the various exothermic peaks depend on the degree of grinding and on the size of the sample employed. Total weight losses (obtained from TG), and the temperatures of the most intense peaks in the DTA and DTG curves (T_m) are given in Table 2 as a function of the degree of grinding and the size of the sample. From the table it follows that with relatively large amounts the

TABLE 2
Dependence of the maximum temperature of the first exothermic peak in the DTA curves of coal on the sample size and on the degree of grinding (particle size)

Degree of grinding (particle size)	Sample size	Weight loss	Exothermic peak temperature T_m
mm	**mg**	**wt.%**	**K**
below 0.2	300	90.0	578
	400	81.2	573
	500	79.0	563
0.2-0.5	300	90.0	598
	400	86.2	563
	500	77.0	553
0.5-1.0	300	90.1	578
	400	77.5	579
	500	75.4	578

reaction which occurs between air and coal is not complete and consequently there is a decrease in total weight loss. Peak temperatures are dependent on size of the samples with particles below 0.2 or between 0.2 and 0.5 mm, but not with larger particles.

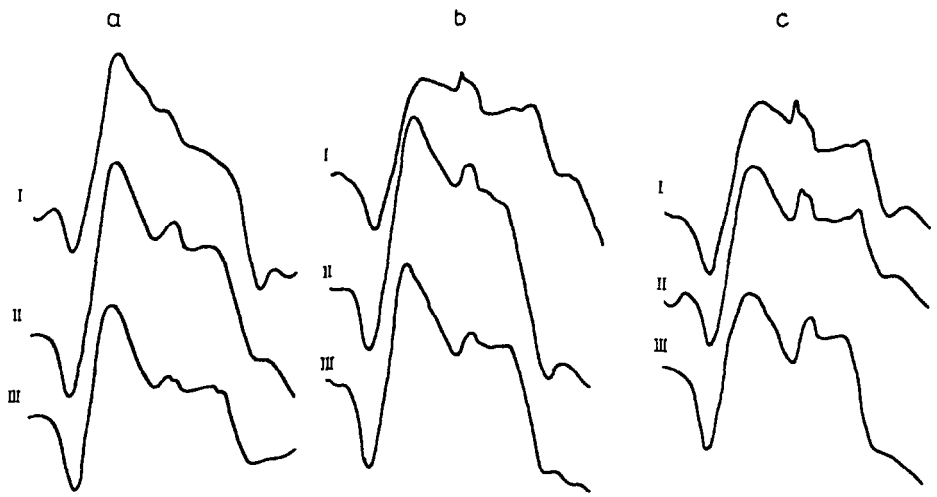

Fig. 4. Influence of the sample size and the degree of grinding (particle size) on the DTA curves of Polish brown coal samples from Lubstow Coal Mine. Sample size: I, 300 mg; II, 400 mg; III, 500 mg coal. Particle size: (a) below 0.2 mm; (b) 0.2-0.5 mm; (c) 0.5-1.0 mm.

It is well known that the methods which are commonly used to characterize coal, such as moisture, ash content, volatile matter, coke after Gray-King, etc., are in general independent of the experimental conditions. This makes it possible to compare coals from different origins. Thermal analysis, on the other hand, should be carried out under similar conditions in order to enable comparison between different coals.

CONCLUSIONS

It has been demonstrated that the measuring conditions have an effect on the nature of thermal analysis curves. In order to compare results and to reach conclusions from thermal analysis curves it is necessary to adopt a standard methodology of measurements in which parameter such as grain size, amount of coal, heating rate, atmosphere and rate of flowing gas are uniform.

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