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CHARACTERIZATION OF CARBONACEOUS PRODUCTS BY TG AND DTA

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Abstract

Thermogravimetry and differential thermal analysis, using only a combustion step with oxygen, were used to characterize carbonaceous products. Binary composition of alumina and wood charcoal, coke, carbon black, petroleum coke or carbon graphite were prepared containing 5 wt% of each. They were characterized by the burnout onset and DTA and DTG peak temperatures, which range from 476°C for wood charcoal to 790°C for carbon graphite.

Complementary characterization of each product was also performed by estimating the ash content from the TG curves, and the calorific value from the DTA curves. The results indicate that these thermal analysis techniques, with only one oxygen burnout step, can be applied both for quality control of each raw material, and to determine the appropriate processing temperatures of the ceramic compositions in which they are used.

Keywords: carbonaceous products, DTA, material characterization, TG

Introduction

The major commercial use of coal nowadays is for power generation, with minor uses as a raw material for ceramic compositions and coke in metallurgy [1]. Its characterization by thermal analysis techniques such as TG and DTA has received special attention [2], and many articles demonstrate that the experimental parameters have a great effect on the resulting thermal analysis curves [3–5].

DTA was the first thermoanalytical technique used to study the thermal behavior of coal (by Hollings and Cobb in 1914) [6]. TG was used first by Klevelen [7] in a coal burnout study.

Carbonaceous materials have usually been characterized by proximate TG analysis, using an initially inert gas flow to determine the moisture and volatile contents [8], followed by an oxygen burnout step to determine the fixed carbon and ash contents. The goal of this research was to characterize different carbonaceous products applied in ceramic compositions by means of TG and DTA, using only a combustion step with oxygen.

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Experimental procedure

Samples of binary compositions of an ALCOA A-16SG alpha-alumina and Brazilian wood charcoal, coke, carbon black, petroleum coke or carbon graphite were prepared containing only 5 wt% of each carbonaceous product in order to have better operating conditions for the combustion. Ternary compositions containing alpha-alumina, 5% of coke and 5% of one of the other products were also analysed.

Samples of 20 mg of each composition were used, in a Perkin-Elmer Thermogravimetric Analyzer TGA 7 and Differential Thermal Analyzer DTA 7, with a 20°C min^{-1} heating rate and a 25 cm^3 min^{-1} oxygen flow.

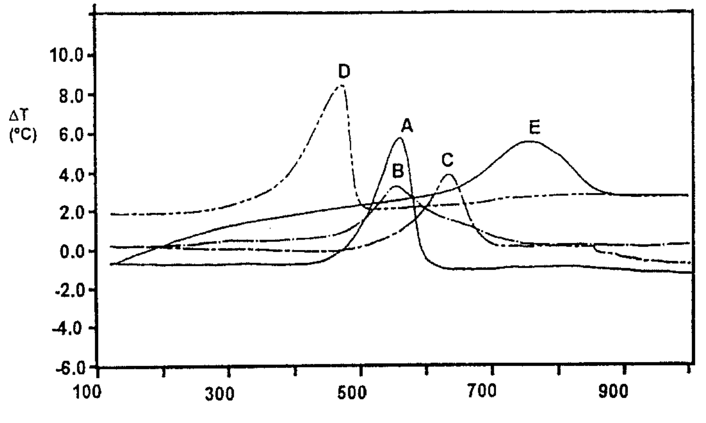

Fig. 1 DTA curves of the carbonaceous products. A – carbon black; B – coke, C – petroleum coke; D – wood charcoal; E – graphite; oxygen flow: 25 cm^3 min^{-1}, heating rate: 20°C min^{-1}

Results and discussion

All the DTA curves for samples containing only one carbonaceous product reflect exothermal reactions represented by a unique peak, as shown in Fig. 1. The characterization involved the onset and peak temperatures of the combustion. It is important to note that the area of the exothermal peak is in each case proportional to the energy released.

Figure 2 shows the TG and DTG curves obtained for the same samples and for a sample containing only alpha-alumina. As the alumina TG curve does not indicate any mass loss, the mass losses observed in the other TG curves are due to the carbonaceous product burnout and to some humidity due to the initial adsorbed water content. The ash contents were calculated from the respective residual mass percentage and are listed in Table 1.

The DTA and DTG curves used to study the burnout behavior and to characterize each product display burnout peaks in opposite directions, with similar shapes for the same material. The DTG curves afford better resolution than the

Table 1 Thermal analysis characterization of the carbonaceous products

Products	Results from DTA curves			Results from DTG curves		Results from TG curves	
	onset temp./	peak temp./	estimated calorific	onset temp./	peak temp./	ash content/	moisture content/
	°C		value/cal g^{-1}	°C		wt%	
Carbon black	489	565	8.07	497	570	6.72	1.12
Coke	489	558	6.38	481	564	36.14	0
Petroleum coke	569	638	5.49	550	639	24.10	2.12
Wood charcoal	370	476	9.19	411	471	19.66	0.74
Graphite	651	760	7.00	655	790	10.02	0.60

DTA curves; the respective observed peak temperatures are again listed in Table 1. It can be seen that only for carbon graphite is there a significant temperature difference, of about 30°C.

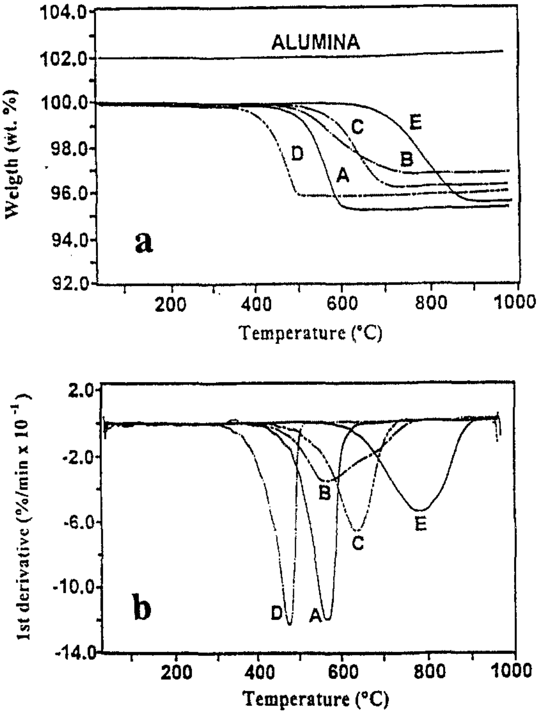

Fig. 2 TG curves of the carbonaceous products (a), corresponding DTG curves (b); A – carbon black; B – coke; C – petroleum coke; D – wood charcoal; E – graphite; oxygen flow: 25 cm^3 min^{-1}, heating rate: 20°C min^{-1}

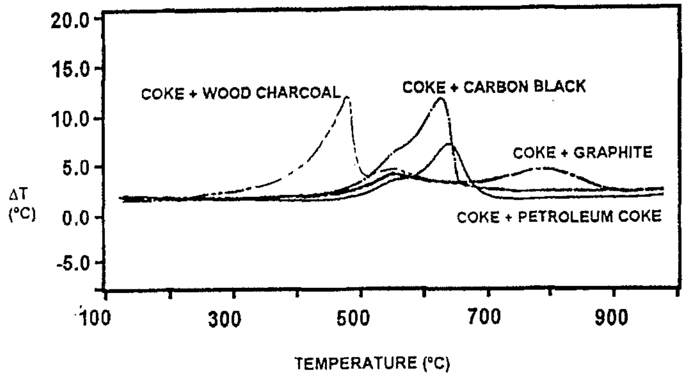

Fig. 3 DTA curves of samples containing two carbonaceous products; oxygen flow: 20 cm^3 min^{-1}, heating rate: 20°C min–[1]

The DTA curves for mixtures of coke and the other carbonaceous products with alumina show that in these cases the original DTA curves are overlapped, and the respective peaks can be shifted to higher temperatures, as occurred for carbon black (629°C) and carbon graphite (798°C), as shown in Fig. 3.

Since the heat of combustion of pure carbon graphite is 94.05 cal g^{-1} mol^{-1} [9] and the carbon graphite sample has a 10.02% ash content and contains 0.6% of adsorbed water, its estimated calorific value is 7.00 cal g^{-1}. As the DTA operating parameters were exactly the same for all the samples, the calorific values for the other carbonaceous products (Table 1) were estimated from the ratio between the respective DTA combustion peak area and that for carbon graphite.

Conclusion

Carbonaceous products can be characterized by TG and DTA using only an oxygen flow.

From the DTA and DTG curves, the onset and peak temperatures of their combustion can be determined. The adsorbed water and ash contents can be measured from the TG curves, and the calorific value can be estimated from the DTA curve area.

Thermal analysis techniques with only one oxygen burnout step can be applied both for quality control of each material, and to determine the appropriate processing temperatures of the ceramic compositions in which they are used.

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