

Quality control of mineral impurities in industrial talcs by thermogravimetric analysis

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Abstract

A practical method to determine and quantify common mineral impurities in talc (carbonate and chlorite) using thermogravimetric analysis has been investigated. Six varieties of commercial talc were studied by thermogravimetry whilst X-ray diffraction measurements were used to identify the impurities and to assess the validity of the thermal analysis. Consistent and accurate results for impurity levels were obtained, which may allow performance of routine industrial quality control analysis.

INTRODUCTION

Incorporation of talc as a reinforcing filler is common practice in the plastic and ceramic industries. The mineral improves the physical properties of the original compounds and aids the material in tailoring for optimum performance as well as decreasing the cost of the final product [1–3].

Industrial talcs are found in the form of massive foliated or globular aggregates. They are associated with minor amounts of chlorites, dolomites, calcites, magnesites and quartz [4], in compositions and concentrations which depend on the paragenesis of the mineral.

The influence of these mineral impurities in the mechanical, thermal and electrical behaviour of the talc composites is an important issue. Consequently, the characterization of the different talcs is a necessary task for the fabrication of talc composites.

This report attempts to develop a method for industrial control of talc impurities based on thermogravimetric analysis.

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EXPERIMENTAL

Materials

Six commercial talcs were supplied by the S.E.T. (Leon, Spain). The average particle size in all the samples was 20 μm (analysed with Malvern equipment). We have adopted the supplier denomination to distinguish them as talcs C, PE, Q, S, SS, and SU.

Apparatus and procedure

TG and DTG measurements were carried out using a thermobalance (Perkin-Elmer model 3600) under a flow of dry air (25 ml min^{-1}). The heating rate of the furnace was fixed at 10 $^{\circ}\text{C min}^{-1}$. All the samples had previously been dried at 150 $^{\circ}\text{C}$ for six hours and were kept in a dry atmosphere until measurement. The sample mass was 10 mg and the temperature range selected was between 100 and 950 $^{\circ}\text{C}$.

X-ray diffraction measurements (XRD) for all talc samples were performed at room temperature using specimens previously heated to 20, 500, 600 and 700 $^{\circ}\text{C}$ (heating time of four hours). The diagrams were recorded in a 3–60 degree range, using nickel filtered Cu $K\alpha$ radiation using Phillips PW-1877 equipment.

RESULTS

TG data

TG plots for the samples are given in Fig. 1. This figure shows that below the decomposition temperature of the talc (approx. 950 $^{\circ}\text{C}$), the samples have suffered at least one mass loss due to the presence of impurities.

In particular, the first loss appeared in samples Q and S within the range 500–600 $^{\circ}\text{C}$, while a second was found between 600 and 750 $^{\circ}\text{C}$ for talcs C, PE, Q and SS. The talc SU did not suffer any significant loss below its decomposition temperature.

XRD patterns of the samples

The talc impurities identified in XRD studies on comparison with diffraction standards [5] of the talcs are summarized in Table 1. Quartz (talcs C, Q and S), calcite (C, PE, Q and SS), dolomite (C, PE, Q and SS) and chlorite (Q and S) were all impurities consistently found in the samples. The talc SU was virtually free from impurities.

XRD patterns showed that up to 500 $^{\circ}\text{C}$ there was no indication of impurity decomposition. Nevertheless, at 600 $^{\circ}\text{C}$, XRD patterns showed a

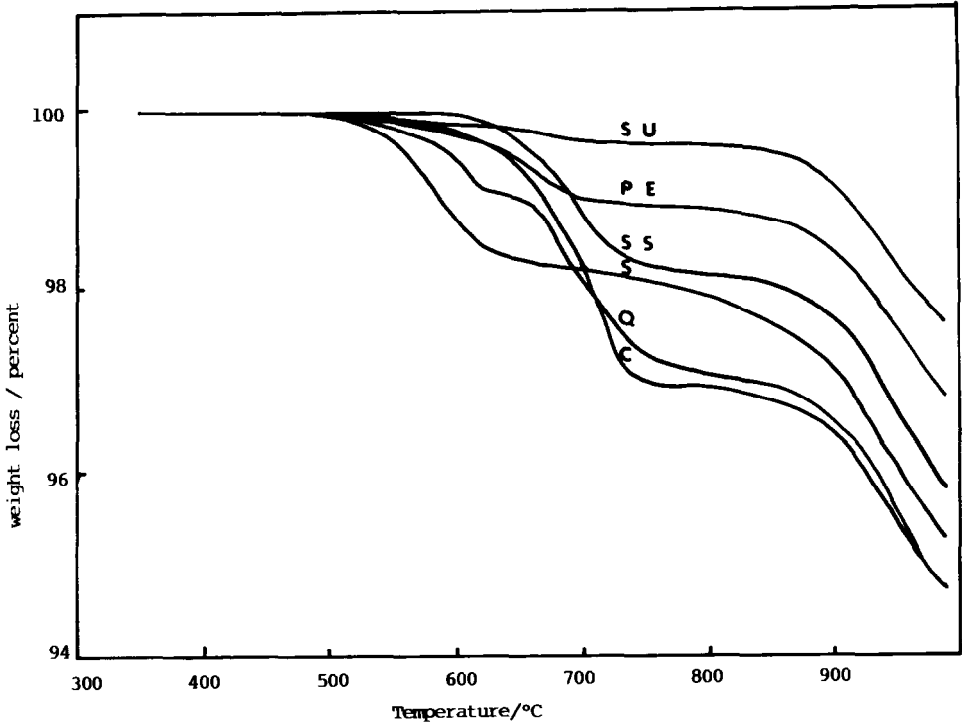


Fig. 1. TG curves of the different talc samples.

lack of calcite, dolomite and chlorite components with the XRD pattern recorded at 700°C showing no significant difference to this (Table 1). Talc and quartz remained unchanged at temperatures of 500, 600 and 700°C.

DISCUSSION

The impurities detected by XRD were those expected from the genesis of this mineral. Naturally occurring talc is formed during the reaction of quartz and dolomite, which also produces calcite and possibly chlorite, magnesite, serpentines, feldspars, etc., depending on geological conditions [4].

The talc impurities found, with the exception of quartz, are eliminated by decomposition in the temperature range 500–600°C. However, there is no change in the talc composition when temperatures are as high as 700°C. The stability of talc within this range of temperature allows measurement by TG of the amount of impurities present.

The steps of mass losses found by TG were identified using the XRD pattern of these minerals.

SU, a talc containing no impurities as detected by XRD, only showed a mass loss at 900°C (Fig. 1) which can only be attributed to talc decomposi-

TABLE 1

Impurity identification by XRD in talcs treated at different temperatures

Sample	Temp. (°C)	Dolomite	Calcite	Chlorite	Quartz
C	20	+	+	-	+
	500	+	+	-	+
	600	-	-	-	+
	700	-	-	-	+
PE	20	+	+	-	-
	500	+	+	-	-
	600	-	-	-	-
	700	-	-	-	-
Q	20	+	+	+	+
	500	+	+	+	+
	600	-	-	-	+
	700	-	-	-	+
S	20	*	*	+	+
	500	*	*	+	+
	600	-	-	-	+
	700	-	-	-	+
SS	20	+	+	-	-
	500	+	+	-	-
	600	-	-	-	-
	700	-	-	-	-
SU	20	-	-	-	-
	500	-	-	-	-
	600	-	-	-	-
	700	-	-	-	-

+, certain identification; *, uncertain identification; -, not present. Powder patterns of these products were compared with the following JCPDS reference patterns: talc (13-558), magnesite (8-479), dolomite (11-78), calcite (8-586), quartz (5-490).

tion. The same mass loss was found in all the other talcs and was therefore considered to be produced by thermal decomposition of the talc.

C, PE and SS which were proved to contain solely carbonates from calcites and dolomites (quartz was not considered in the study because it does not decompose in this temperature range), presented a maximum in the DTG curve between 600 and 750 °C which corresponds to decomposition of a normal carbonate group in TG analysis [6]. A broad range of decarbonation was observed due to the different origins of the carbonates (one from calcite and two from dolomite) [7,8] with distinct, but close, decomposition temperatures.

On the other hand, sample S did not contain significant amounts of dolomite or calcite, but showed the presence of chlorite (Table 1) in XRD analysis. Therefore, the mass loss found in the corresponding TG curve can be attributed to the latter mineral which, in accordance with the literature data, appears at a lower temperature than that of carbonates [7], with a

TABLE 2
Percentage of impurities in each sample

	C	PE	Q	S	SS	SU
Carbonates	3.95	1.13	2.92	–	2.46	0.30
Chlorite	–	–	3.96	12.79	–	–

DTG maximum at 550 °C. This step is produced by the dehydroxylation of the brucitic layer in the chlorite. There is a second dehydroxylation of the chlorite whose temperature range coincides with that of talc decomposition, so it cannot be used to calculate the percentage of this impurity.

In XRD analysis the Q sample showed impurities of both carbonates (calcite and dolomite) and chlorite (Table 1). Accordingly, two mass losses were obtained by TG at the given temperatures for chlorites and carbonates. The remaining chlorite would decompose simultaneously with the talc.

The TG data can be used to quantify the percentage of impurities in the sample of talcs. The mass loss in the decomposition corresponding to carbonate was assigned to carbon dioxide (CO₂) and can be related to total carbonates (dolomites and calcites). The mass loss of chlorites was attributed to water formed from the six hydroxyl groups of the brucitic layer. The molecular stoichiometry of the chlorites is complex. XRD patterns and formulae for different chlorites can be found in the literature [5]. The molecular weight used in the calculation was that of the chlorite with an XRD pattern similar to those obtained in this work. All these results are summarized in Table 2.

The objective of the present study was to characterize the level of impurities in the talcs by a quantitative method suitable for use by industry in rapid quality control of talcs. It has been shown that TG and DTG provide a suitable method to achieve this purpose.

The dolomite/calcite ratio, which is found in the XRD analysis, is difficult to obtain by calorimetric methods. However, this ratio is of little interest for the industrial applications. For the C, PE, Q and SS samples this relation was estimated by comparing the relative intensities of the (104) Bragg reflections of both impurities with those observed on patterns of known dolomite and calcite mixtures. These values were found to be C = 3 : 1, PE = 10 : 3, Q = 3 : 1, SS = 7 : 1.

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