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Quality control of mineral impurities in industrial talcs

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ABSTRACT

A practical method to determine and qualify common mineral impurities in talc (chlorite) using Controlled transformation Rate Thermal Analysis (CRTA) has been investigated. The vacuum conditions and low rate of decomposition used in the present study has enabled to understand and quantify the decomposition in terms of the different brucite and talc sheets of the present mineral sample with chlorite impurity.

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1. INTRODUCTION

The talc is a tri-octahedral layered mineral composed of hydrated magnesium silicate with the chemical formula $Mg_3Si_4O_{10}(OH)_2$. The talc structure consists of two tetrahedral silicate SiO_4 sheets separated by an octahedral $MgO_4(OH)_2$ sheet, as is shown in figure 3. Each tetrahedron shares three corners with the adjacent tetrahedrons. Therefore, three oxygen atoms of each tetrahedron belong simultaneously to two silicon atoms while the fourth oxygen atom belongs to one tetrahedral only. The ratio of the silicon to oxygen atoms is 2:5, thus corresponding to the formula $[Si_4O_{10}]^{4-}$. The charge of the anion layers is counter-balanced by the appropriate numbers of magnesium cations. Talc has found a wide-spread application such as rubber, paper, pharmaceutical, cosmetic, ceramic refractory and ceramic porcelain industries[1-3]. Its thermal decomposition behaviour is of considerable interest.

The stoichiometric balance of the dehydroxylation of pure talc can be written by the following equation:

$$\underset{\text{Talc}}{Mg_3(Si_2O_5)_2(OH)_2} = 3MgSiO_3(s) + SiO_2(s) + H_2O(g) \qquad (1)$$

The three products of reaction are enstatite ($MgSiO_3$), amorphous silica (SiO_2) and gaseous water. However, industrial talc is found in the form of massive foliated or globular aggregates, 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 composite is an important issue consequently; the characterisation of the different talcs is necessary task for the fabrication of talc composites.

Intensive studies by conventional thermo analytical techniques (thermogravimetric analysis (TG), differential thermogravimetric analysis (DTG) and differential scanning calorimetry (DSC)) were carried out on the talc structure changes at various temperatures[5-30]. The analysed samples originated from locations throughout the world: U.S.S.R[5, 6,10,19,23] Germany[16-18], France[20,26], Spain[28], U.S.A[7, 8,12], Japan[11], Taiwan[15], Pakistan[21] and India[22]. This fact explains that the descriptions of the thermal decomposition of talc are conflicting.

Many factors can have a significant effect on the results obtained by thermoanalytical methods, first the different chemical composition of samples (it must be noted that talc minerals occurring in nature are rarely chemically pure substances) and the type of instrument influences the shape of the thermograms; for this it is problematic to compare the shape of two curves recorded under different experimental conditions (e.g., sample size, geometry of the sample holder, measuring and recording instruments) as well as assured high standardization and reproducible analytical conditions (e.g., furnace atmosphere, particle size of the mineral powder, technique of packing the sample, inert material, and heating rate). Furthermore conventional thermal analysis, which subjects samples to a linear heating rate, can have a major influence on the information content of the TG curve and the nature of the heat treated products.

To minimize as much as possible the influence of these parameters this study uses the method of Controlled transformation Rate Thermal Analysis (CRTA)[31,32]. This method enables a control of the reaction rate allowing a sufficient decrease in the rate to diminish the pressure and temperature gradients within the reaction sample. Rouquerol[31] has already shown the necessity to control not only the temperature, but also the water vapour pressure above the sample to obtain reproducible results and thus to a more reproducible experiment. The possibility of lowering the rate of transformation at will makes it possible to ensure the sample a more homogeneous temperature. Under these conditions all the grains constituting the sample react at the same time. This results in a better separation of intermediate stages in the thermal decomposition and a greater sensitivity of the detected phenomena.

The present investigation using CRTA was undertaken to clarify the dehydroxylation steps of two samples of talc. Several intermediate samples were immediately analysed by XRD, FTIR and MEB with the aim to understand the decomposition mechanisms.

TABLE 1: Chemical composition (Weight %), of talc-fr and talc-tn

Oxides	SiO_2	MgO	Al_2O_3	Fe_2O_3	CaO	P_2O_5	K_2O	TiO_2	MnO	Li_2O
talc-fr	54.83	33.6	2. 94	0.87	0.64	0.017	0.005	0.004	0.002	0.001
talc-tn	42.21	33.6	12.2	1.5	0.70	0.017	0.017	0.014	0.005	0.003

2. EXPERIMENTAL

Samples

Two talcs coming from the Luzenac Europe SA, France, (hereafter talc-fr) and the Tunisian central pharmacy, Tunisia, (hereafter talc-tn) were used as starting materials. Before to be used in this study, the two talc were characterized as purity and morphology. The chemical composition was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Liberty 200, Varian, Clayton South, Australia). The powder sample was dissolved by assisted acid treatment. Analytical wavelengths which showed minimum spectral interferences and maximum sensitivity were selected. The microstructure of the talc powders was investigated using a Philips XL 30 scanning electron microscope. Nitrogen gas adsorption-desorption measurements using a nitrogen adsorption porosimeter (Quanta-Chrome, Autosorb-1) were performed for talc powders to obtain their specific surface. Sample Controlled Thermal Analyses were recorded by using a TA Instrument, model Q500 and an apparatus home built[31] respectively. The totality of evolved gases is continuously analysed by a RIBER mass spectrometer coupled to the apparatus for Sample Controlled Thermal Analysis.

3. RESULTS AND DISCUSSION

3.1. Chemical analysis

The chemical composition of the two talcs analysed by ICP-AES is presented in TABLE 1.

The talcs contain chlorite as impurities. The amounts of chlorite are 16 % in the talc-fr and 66.3% in the talc-tn respectively. Some oxides as Cr_2O_3 and BaO are not present in the analyzed talcs.

3.2. Microstructural and morfological characterization

Both samples of the talc have practically the same specific surface, measured by adsorption of nitrogen in 77 K, equal to 2.45 $m^2 \cdot g^{-1}$ and the same grain size around 10 μm.

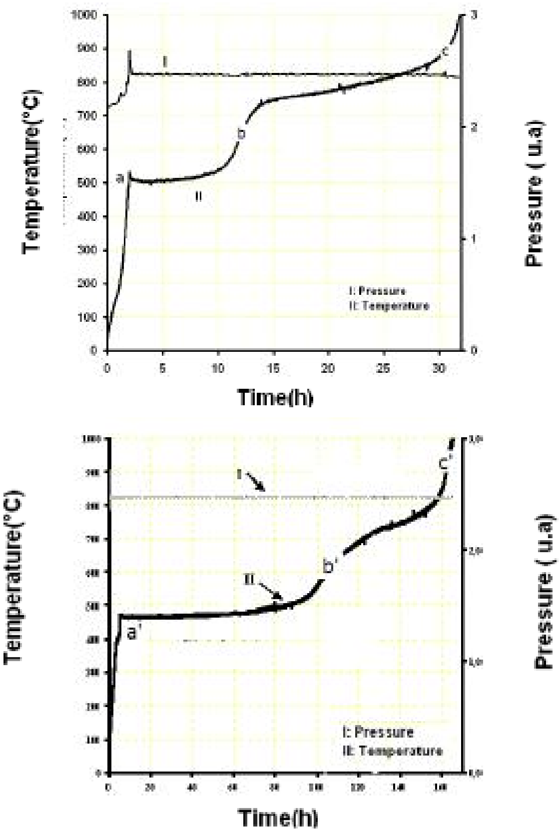

Figure 1(a): CRTA curve of talc-fr under $2\cdot10^{-2}$ mbar water vapour; (b): CRTA curve of talc-tn under $2\cdot10^{-2}$ mbar water vapour pressure

3.3. Controlled rate thermal analysis CRTA

The CRTA curves of the talc-fr and talc-tn samples, recorded under identical experimental conditions (residual pressure, mass, rate) are shown in figure1a and figure1b respectively.

In this method, production rate of the gaseous phase can be permanently controlled (adjusting the heating rate) so that the pressure level above the sample could be kept at a reference value of 2.10^{-2} mbar. The curve I shows the variation of the pressure and the curve II shows the variation of the sample temperature vs. time. According to major changes suggested by the shape of the CRTA diagram, we can divide the decomposition process of the talc-fr into two steps (Figure 1a). The first step occurs between 500 and 650^{0}C (segment ab on the curve II, figure 1a). The second step occurs between 650 and 850^{0}C (bc on the curve II, figure 1a). The CRTA curves of sample talc-tn (Figure 1b) revealed also two decomposition steps between 470-650°C (a'b') and 650-850^{0}C (b'c').

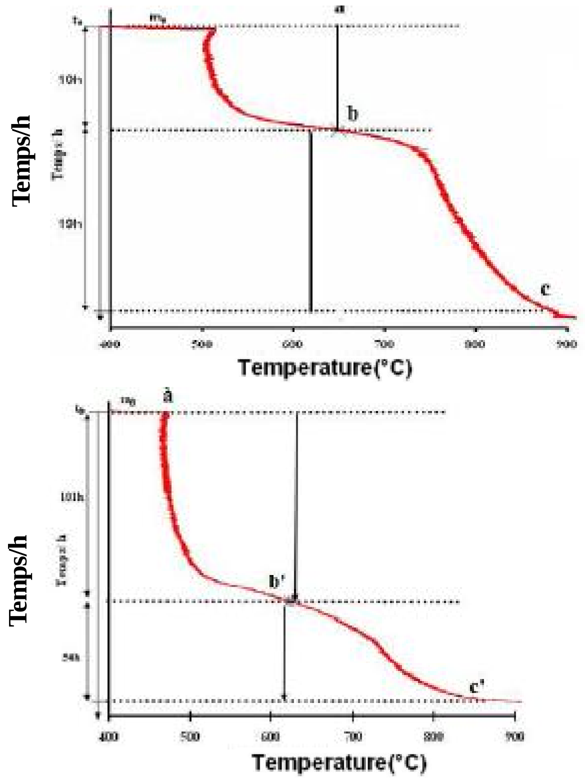

Figure 2(a): Thermogravimetric curve of talc-fr; (b): Thermogravimetric curve of talc-tn

In order to obtain more information on the thermal decomposition of In order to obtain more information on the thermal decomposition of talc samples, a continuous evolved gas analysis was carried out during CRTA. According to these results[39,40], the decompo sition steps noted on the CRTA curves correspond to the complete deshydroxylation of talc samples. Along the thermolysis, the sample is rounded by the water vapour generated by the talc decomposition. In this case, the gas composition above the sample does not greatly change. The vapour pressure is kept in a quasi-equilibrium state because its rate of production during the dehydration is maintained equal to its rate of elimination from the reactor. Under these conditions, the length of each dehydration step is proportionally with the mass of water lost. Thus, the CRTA curve as-obtained is analogue to a thermogravi metric curves (Figure 2a, 2b).

Intermediates samples obtained at the end of each decomposition step were characterized by XRD, FTIR, SEM and BET methods. In this aim, the CRTA experiment and the reaction were stopped at the temperatures determined from the initial curves and then

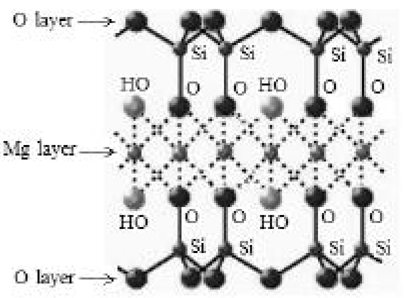

Figure 3: Crystalline structure of talc

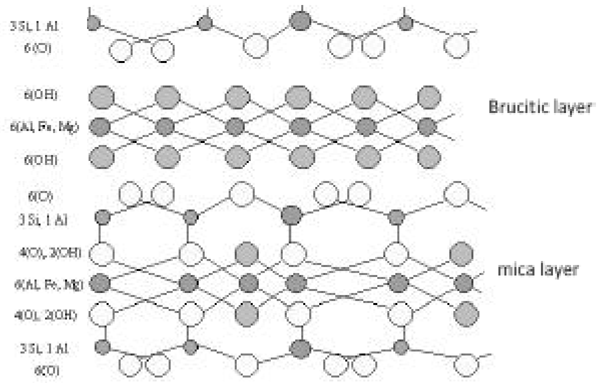

Figure 4: Crystalline structure of the chlorite

are restarted under identical conditions of rate and pressure. The results of the intermediates samples characterization showed that the first decomposition step can be attributed to the removal of the hydroxyl group from the brucite layer of chlorite and, the second step of the thermolysis can be attributed to the loss of the water from the talc and, the mica layer of chlorite[39, 40].

In order to explain the dehydroxylation of the talc it is necessary to take a closer look at the structure of both minerals (talc/chlorite) and also the arrangement of the hydroxyl groups. Both talc and chlorite are triocta hedric minerals with sheet structure and theoretical formula $Mg_3SiO_4O_{10}(OH)_2$ and $(Mg_{6-x-y}\ Fe_y\ Al_x)(Si_{4-x}Al_x)\ O_{10}(OH)_8$, respectively. The elemental layer of talc is composed of an octahedral plane of brucite ($Mg(OH)_2$) with therefore only one type of -OH site confined between two external tetrahedral silicate sheets[34,35]. Chlorite (Figure 4), however is composed of alternating talc (containing two hydroxyl (OH) groups separated by a bridging oxygen) and brucite layers (containing six hydroxyl (OH) groups arranged next to each other)[34-37].

The “talc” can release water from two OH groups. By heating the mineral, the water is removed in distinct steps due to the bonding energy differences.

Nevertheless a closer inspection of the relative length of the two steps in the CRTA curves reveals a 1:2 ratio for parts (ab) and (bc) respectively for the sample talc-fr (figure 2a) and a ratio of loss of mass of 2:1 for stage (a’b’) and (b’c’) respectively for the sample talc-tn(figure 2b). The chemical and XRD analysis of the initial product indicated a chlorite content of 16% in talc_fr and of 66,33% of talc-tn.

The CRTA curve (figure 2a) thus reflects 1:2 ratio allotted to:

- The first step is due to loss of water from the brucite sheets in chlorite. Thus 16%×6 (OH groups per brucite sheet (i.e. 96)).
- The second step is due to water loss from the talc layers in chlorite and in the talc itself (i.e. 16×2 -OH groups in chlorite in addition to 84%×2 OH groups in the talc (i.e. a total of 200)).

The CRTA curve (figure 2b) thus reflects this percentage and 2:1 ratio perfectly :

- The first stage due to the deshydroxylation of the layers brucitic of chlorite, corresponds at the beginning of a number of hydroxyl proportional to:
  $6\times(m_{chlorite})$, is $6\times6,63\ m_{total}$
- The second stage is due to the deshydroxylation of the layers mica (OH) of chlorite and of those of talc (OH), it corresponds at the beginning of a number of hydroxyl proportional to:
  $(2\times m_{chlorite})+(2\times m_{talc})=2\times6,63\ m_{total}+2\times3,37\ m_{total}$

The report/ratio of the water masses lost during the two stages is in concord with the percentage found by chemical analysis.

This study thus shows the possibility to characterise, via CRTA, a mineral talc sample in terms of talc decomposition and the quantification of impurities such as chlorite[39,40].

4. CONCLUSION

In the light of the results of this study we can understand at the moment the visible contradictions existing in the literature concerning the thermolyse of the talc. First of all, let us note that the biggest confusion comes from the fact that the word “talc” is used with two different meanings:

- This word indeed indicates a mineralogical space which

the composition and the structure are clearly defined;
- It also indicates the mineral of talc, is always impure.

The mineral talcs studied have properties which depend on their origin; this explains the dispersal of the results on their thermal behaviour. The thermal behaviour of the talcs shown two dehydration steps corresponding to deshydroxylation of brucitic and mica layer.

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