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Thermogravimetric analysis of a talc mixture

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Abstract

A commercial talc also containing chlorite, dolomite and magnesite was subjected to thermogravimetric analysis to determine its mineral composition. Knowledge of the reactions and the decomposition temperatures, as well as the crystallochemical formulae of the phases present, has permitted the application of a system of equations that has provided mineral composition results in good agreement with standard analysis obtained via elemental chemical analysis.

INTRODUCTION

Beneficiation processes (leaching, flotation, magnetic separation, selective milling, etc.) designed to concentrate the desired components of an ore call for constant support from the analytical chemist for the characterization of concentrates and tailings. Although, in most cases, the standard calculation of amounts of minerals from the chemical analysis is theoretically possible using conditional matrix regression [1], this requires (1) the titration of numerous elements, (2) a reliable determination of structural water, (3) a precise knowledge of the mineral chemistry, involving electron microprobe and X-ray analysis, and (4) an investment in time. Thermogravimetry provides an alternative method of analysis. In spite of apparent disadvantages [2], such as the overlap of phenomena, shifts of temperature according to dilution effects and according to particle size distribution or chemical composition, and the difficult choice of analytical conditions when the components show contrasting thermal behaviour, thermogravimetry is easy to carry out, gives immediate results and is precise enough for the control of a beneficiation process. This paper explains a method of thermogravimetric analysis applied to an industrial

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talc ore containing mainly talc, chlorite, magnesite and dolomite [3, 4], plus about 2% of other minerals including magnetite and traces of sulphides. Beneficiation of the ore whose mineralogical composition has to be determined is performed to obtain a talc concentrate as low as possible in chlorite and carbonates.

The four minerals present decompose on heating, water being released in the case of the phyllosilicates (talc and chlorite), and carbon dioxide where the carbonates (magnesite and dolomite) are concerned. Consequently, knowing the formula weights and hence the weight losses—as well as the temperature range within which each component decomposes, of course—the quantities of the various minerals in the mixture can be determined. The main difficulties in quantitative determination lie in knowing the exact formula weight and in superimposing weight-loss curves when the thermogram is being executed.

SAMPLE

The sample studied comes from the Brusada Deposit at Valmalenco (Sondrio, Italy). The talc in this deposit derives from the alteration of serpentine [5]. Samples representative of five industrial talcs containing different quantities of talc, chlorite, dolomite and magnesite have been subjected to thermogravimetric analysis. A sixth sample (No. 6) was obtained by leaching Sample No. 2 with HCl (3 wt%) so as to remove most of the dolomite and obtain a sample containing only phyllosilicates. The samples for thermogravimetric analysis were ground to <30 μm, which is the size at which the talc is subsequently to be treated. It is necessary to grind as fine as this in order to attain a reasonable degree of liberation, considering the small dimensions of the talc grains present in the ore (15–30 μm). However, this fine grain size also favours the homogeneity of the sample for thermogravimetric analysis. The moisture content of the samples was around 0.3%.

THERMAL APPARATUS

The tests were performed using a Stanton Redcroft Model 1500 thermobalance having a sensitivity of 1 μg, temperature range 20–1500°C and allowed heating rates 0.1–50°C min^{-1}. The furnace winding is made of rhodium–platinum and the thermocouple is located under the rhodium–platinum crucible containing the sample. The heating rate in the tests was 20°C min^{-1} from 25 to 500°C, and 10°C min^{-1} from 500 to 1000°C in a stream of air. The weight of the sample was about 50 mg in each test and the scale factor (SF) was between 0.04 and 0.75 mg mm^{-1}.

THEORETICAL BEHAVIOUR OF MINERALS PRESENT IN THE ORE

The reactions and decomposition temperatures of the minerals present are [6]

Talc	$Mg_3Si_4O_{10}(OH)_2 \rightarrow 3MgSiO_3 + SiO_2 + H_2O$	900–1000°C
Dolomite	$(Ca, Mg)(CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2$ $CaCO_3 \rightarrow CaO + CO_2$	790–940°C
Magnesite	$MgCO_3 \rightarrow MgO + CO_2$	660–690°C

In general, the chlorite global decomposition reaction can be represented in the following manner:

$$\text{Chlorite } \underset{\text{mica}}{(Si_3Al)O_{10}(Mg_3)(OH)_2}\underset{\text{brucite}}{(Mg_2Al)(OH)} \xrightarrow[900°C\text{ (mica)}]{600°C\text{ (brucite)}}$$

$$\underset{\text{spinel}}{MgAl_2O_4} + \underset{\text{enstatite}}{2MgSiO_3} + \underset{\text{forsterite}}{Mg_2SiO_4} + \underset{\text{water}}{4H_2O} \qquad (1)$$

The structure of chlorite (Fig. 1) has been extensively studied [7–11]. The chlorite sheet is made up of two components, a brucite-like layer and a mica-like one. The former comprises an octahedral sheet and the latter is formed by a second octahedral sheet occurring between two tetrahedral

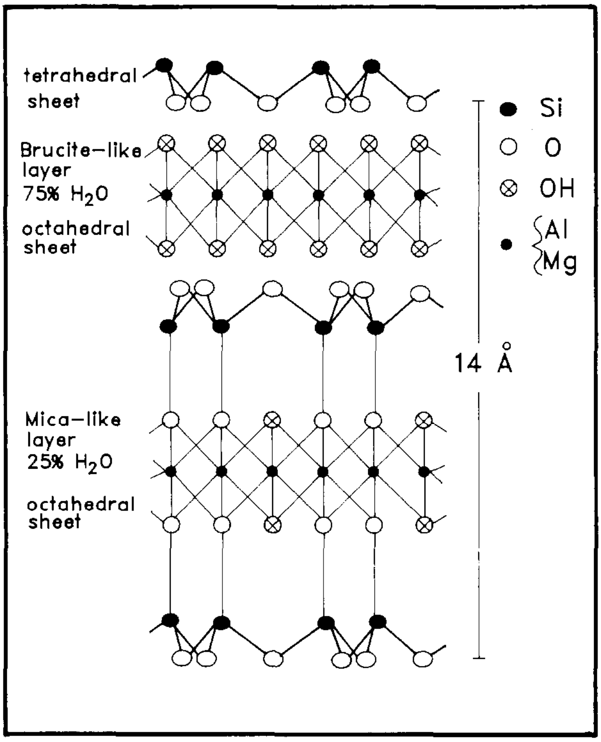

Fig. 1. Chlorite structure.

sheets, the package forming the mica-like layer. The dehydroxylation of the brucite-like layer (at ≈600°C, 75% of the structure water) leads to the formation of an intermediate compound that itself decomposes (at ≈800°C, 25% of the structure water) giving spinel, enstatite and forsterite after recrystallization, as described by global reaction (1).

The formula which correlates weight loss with the quantity of mineral in a mixture can be expressed as follows

$$\%\text{Phase} = \frac{(L \times SF)}{(TW \times W)} \times 10^4$$

where L = length of thermogram step corresponding to weight loss of mineral considered (mm); SF = scale factor (mg mm^{-1}), TW = theoretical weight loss just for the mineral to be determined (%); W = original weight of sample (mg).

RESULTS

As the theoretical weight loss of a mineral (TW) depends on its formula weight, its exact crystallochemical formula must be known in order to make an accurate estimate. Thus information is particularly necessary for chlorite, the weight loss of which can vary from 9.11 to 14.98% depending on its composition [12].

Microprobe analyses have therefore been performed to establish the structural formulae of the minerals affected by the thermal decomposition. These formulae are given in Table 1, together with their formula (weight FW) and the percentage of volatile substances ($\Delta W\%$) liberated during the decomposition of each of them, which causes the weight loss on heating. Figure 2 illustrates the thermogram obtained for sample No. 5. Except for the length of the three zones — which depends on composition — the thermogram resembles those obtained on the other samples.

The decomposition temperatures of the minerals present appear to be lower (Table 2) than the temperatures reported in the literature for the same minerals considered to be in the pure state. This might be explained

TABLE 1

Crystallochemical formulae of minerals present in talc subjected to thermogravimetric analysis

Mineral	Formula	FW	$\Delta W\%$
Talc	$(Si_4)(Mg_{2.78}Fe_{0.14}Ni_{0.01})_{2.93}O_{10}(OH)_{2.07}$	389.3	4.70
Chlorite	$(Si_{3.13}Al_{0.87})_4O_{10}(OH)_8(Mg_{4.47}Fe_{0.53}Al_{0.64}Cr_{0.12})_{5.77}$	563.1	12.78
Magnesite	$(Mg_{0.88}Ca_{0.01}Fe_{0.11})CO_3$	87.7	50.20
Dolomite	$(Mg_{0.92}CaFe_{0.08})_2(CO_3)_2$	186.6	47.20

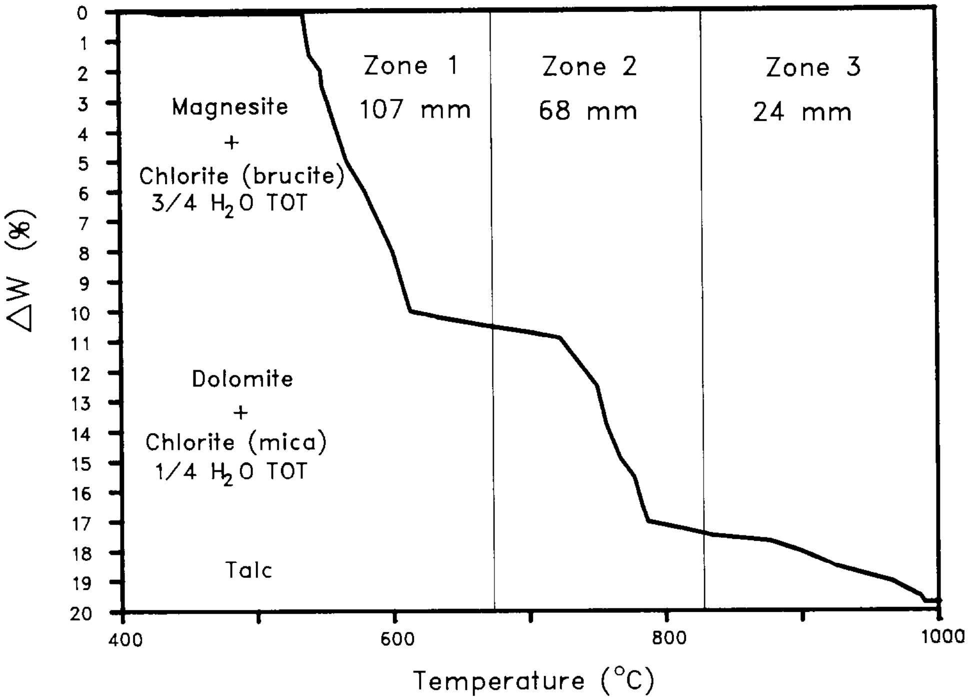

Fig. 2. Thermogram executed on sample 5 of the talc mixture containing talc, chlorite, dolomite and magnesite. Samples ground to <30 μm. Heating rate: 20°C $\min^{-1}$ from 25 to 500°C, and 10°C $\min^{-1}$ from 500 to 1000°C. Performed in a stream of air. Sample weight: 49.97 mg. Thermobalance: Stanton Redcroft Model 1500. Scale factor: 0.05 mg mm^{-1}.

by the fine grain size of the sample or by a difference in the heating rate; besides, in air, the decomposition temperatures are lower than in a self-generated atmosphere; there is also a dilution effect, and the decomposition temperature depends on the quantity of reactant in a given matrix. It is assumed, however, that the succession of the decomposition

TABLE 2

Comparison of experimental decomposition temperatures and decomposition temperature of pure minerals

	Chlorite (brucite) + magnesite (1st step)	Chlorite (mica) + dolomite (2nd step)	Talc (3rd step)
Experimental decomposition temperature	540–670°C	670–810°C	810–990°C
Theoretical decomposition temperature	600–690°C	790–940°C	900–1000°C

temperatures for the various minerals under test remains the same as for the pure minerals.

(1) Decomposition of magnesite and chlorite (brucite-like layer), $T = 540\text{–}670°\text{C}$;
(2) Decomposition of dolomite and chlorite (mica-like layer), $T = 670\text{–}810°\text{C}$;
(3) Decomposition of talc, $T = 810\text{–}990°\text{C}$.

The two weight losses of dolomite should thus be consecutive.

The problem of the overlapping weight losses of magnesite, chlorite and dolomite has been tackled by means of a system of five equations with five unknowns.

$$\left\{\begin{array}{l}
(L_1 - \frac{3}{4}L_c) \times \dfrac{SF}{W \times WT_{MA}} \times 10^4 = MA \\[2ex]
(L_2 - \frac{1}{4}L_c) \times \dfrac{SF}{W \times WT_{DO}} \times 10^4 = DO \\[2ex]
L_3 \times \dfrac{SF}{W \times WT_{TA}} \times 10^4 = TA \\[1ex]
L_c \times \dfrac{SF}{W \times WT_{CH}} \times 10^4 = CH \\[2ex]
TA + CH + DO + MA = 100
\end{array}\right\}$$

where the symbols have the following meanings: L_1 = first step of thermogram (mm); L_2 = second step of thermogram (mm); L_3 = third step of thermogram (mm); L_c = sum of contributions of steps $L1$ and $L2$ of the chlorite weight losses (mm); TA = wt.% of talc in sample analysed; DO = wt.% of dolomite in sample; MA = wt.% of magnesite in sample; CH = wt.% of chlorite in sample; SF = scale factor (mg mm^{-1}); W = sample weight (mg); WT_{CH} = weight loss of chlorite (%); WT_{MA} = weight loss of magnesite (%); WT_{DO} = weight loss of dolomite (%); WT_{TA} = weight loss of talc (%).

The unknowns are TA, CH, DO, MA and L_c. The sum of the four minerals is taken to be 100%.

The system of the five equations can also be written in the following matrix form

$$\begin{pmatrix} L_1 \\ L_2 \\ L_3 \\ 100 \end{pmatrix} = \begin{pmatrix} L_{MA} & \frac{3}{4}L_{CH} & 0 & 0 \\ 0 & \frac{1}{4}L_{CH} & L_{DO} & 0 \\ 0 & 0 & 0 & L_{TA} \\ 1 & 1 & 1 & 1 \end{pmatrix} \times \begin{pmatrix} MA \\ CH \\ DO \\ TA \end{pmatrix}$$

TABLE 3

Results (in %) obtained by thermogravimetric analysis of the six talc samples compared with the results obtained by chemical standard analysis

Mineral	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5		Sample 6	
	TGA	Chem	TGA	Chem	TGA	Chem	TGA	Chem	TGA	Chem	TGA	Chem
Talc	67.0	64.7	44.7	44.4	51.1	52.1	52.7	54.2	49.0	49.2	80.1	77.6
Chlorite	23.1	22.4	12.6	9.6	18.8	18.0	23.0	19.3	20.6	23.2	19.8	19.4
Dolomite	10.2	12.9	43.0	46.0	1.4	2.2	5.1	7.2	13.0	14.1	1.5	3.0
Magnesite	0	0	0	0	28.7	27.7	19.2	19.3	17.4	13.5	0	0
Total	100.3	100	100.3	100	100	100	100	100	100	100	101.4	100

where the symbols L_i assume the previous meanings and L_{MA}, L_{CH}, L_{DO} and L_{TA} are the theoretical weight losses of the pure minerals expressed in mm.

Table 3 sets out the results obtained for the six samples analysed, together with the mineralogical estimate performed for the standard calculation starting from the elemental chemical analyses, which was utilized as reference.

The results obtained for talc indicate that there is good agreement between the thermogravimetric and chemical methods except in the case of samples 1 and 6. Where the latter is concerned, it is observed that the sum of the minerals present amounts to 101.4%, because the system of five equations gave a mathematical value of −1.4% for magnesite, satisfying all the equations included the fifth one. But as −1.4% has no physical meaning the magnesite content has been taken as zero and as a consequence the sum of the four minerals becomes 101.4%. Similar behaviour occurred with samples 1 and 2, which gave a value of −0.3% for magnesite. The results obtained for chlorite are also in good accord except for samples 2 and 4. Magnesite, too, gives comparable values except for sample 5. The values obtained from the thermogram for dolomite, which is the mineral with the most variable content among those examined, show good agreement between the thermogravimetric and chemical analyses. It is also observed that the thermogravimetric values for the two phyllosilicates are generally higher than the corresponding ones obtained via the standard analysis, while the opposite is the case where dolomite is concerned. The results provided by the thermal analysis therefore follow the compositions of the samples analysed very well, even though no consideration was given to factors such as the oxidation of the iron present as a substituted element within the minerals present and as a free oxide in the ore (albeit in only small amounts). However, any correction that might be considered for iron oxidation would certainly be difficult to determine, and would not greatly improve the results obtained. One final point in favour of the system investigated is that the analyses did not have to be calibrated by a calibration line obtained using pure standards; which are very difficult to find in the deposit.

CONCLUSIONS

Thermogravimetric analysis of the mineral content of six samples of industrial talc having various talc, chlorite, dolomite and magnesite contents has given results in good agreement with those obtained via standard analysis following elemental chemical analysis. Microprobe analysis of the ore has indicated the expected weight loss on heating for each of the minerals present, and the adoption of a system of equations has avoided the need for matching weight-loss curves as a function of temperature obtained from the thermogram.

Thermogravimetric analysis thus provides a quicker method of mineral analysis for this type of ore than does chemical analysis, especially as the instrumentation now available permits thermograms to be obtained on up to 19 samples simultaneously.

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REFERENCES

1 J. Yvon, J. Baudracco, J.M. Cases and J. Weiss, in A. Decarreau (Ed.), Matériaux Argileux, Structure, Propriétés et Applications, Part IV, SFMC, GFA, Paris, 1990, Chap. 1, pp. 473–489.
2 R.C. Mackenzie, The Differential Thermal Investigation of Clays, Mineralogical Society, London, 1957, pp. 18–20.
3 L. Piga and J. Yvon, In Terra Abstracts Vol. 3, No. 1, Sixth Meeting of the European Union of Geosciences, Strasbourg, 1991, p. 412.
4 L. Piga, EEC contract MA1M0035, 1990, p. 17.
5 P. Beaulieu, Contribution a l'etude geologique des gisements de talc du Valmalenco, Doctoral Thésis, Université de Franche-Comté, 1983, p. 25.
6 R.C. Mackenzie, Differential Thermal Analysis, Academic Press, London, 1970, pp. 313, 321, 503, 524.
7 R.C. Mackenzie, Differential Thermal Analysis, Academic Press, London, 1970, p. 498.
8 G.W. Brindley, Acta Crystallogr., 3 (1950) 408.
9 G.W. Brindley and S.Z. Ali, Acta Crystallogr., 3 (1950) 25.
10 B.E. Brown and S.W. Bailey, Am. Miner., 47(7–8) (1962) 819.
11 H. Steinfink, Acta Crystallogr., 11 (1958) 191.
12 W.A. Deer, R.A. Howie and J. Zussman, Rock-Forming Minerals, Longmans Green, London, 1961, pp. 138–145.