Thermochimica Acta, 38 (1980) 329—334

Note

THE CORRELATION BETWEEN WEIGHT LOSS AND PHASE COMPOSITION OF THE CALCINATION PRODUCTS OF GYPSUM

ABDEL-AZIZ A. KHALIL

National Research Centre, Cairo (Egypt)

(Received 15 October 1979)

Various authors have tried to use the weight loss taking place on heating calcined gypsum at temperatures <300°C to assess the phase constitution of the products of calcination. Some investigators have postulated that the percentage weight loss could be used as representing the chemically-combined water attached to $CaSO_4$ in a general form $CaSO_4 \cdot n\ H_2O$ [1]. They tried to calculate the value of n corresponding to any weight loss data determined. Accordingly, they found that the values of n could be 2.0, 1.8. 1.2,···, 0.8,··· 0.3 moles, etc. These calculations meant that $CaSO_4$ could form a continuous solid solution with water. This is not true and we are familiar only with two forms of hydrated calcium sulphate, namely the di- and hemihydrate phases.

It has been published by many workers that the dehydration of gypsum is a continuous process and if the calcination temperature is over 90°C, gypsum, hemihydrate and anhydrite phases may be found together in different amounts, depending on the calcination conditions [2—6]. Moreover, we have mentioned previously that the change in weight could not be used, as a separate tool, for the assessment of the calcination products [7].

The present work aims at determining the relation between weight loss and the phase constitution of the calcined plaster. More light is thrown on the problem and a simplified formula for phase calculation is suggested. It was possible, using this formula, to correlate the weight loss with the actual amounts of gypsum, hemihydrate and anhydrite. In addition to this, previous knowledge of the amount of any of these phases helps in the deduction of the amounts of the remaining phases using the weight loss value.

DEHYDRATION OF GYPSUM

Gypsum ($CaSO_4 \cdot 2\ H_2O$) in its pure state gives, on heating, a weight loss value of 20.93%, corresponding to the evolution of the two molecules of water of crystallisation, while hemihydrate ($CaSO_4 \cdot 0.5\ H_2O$) shows a value of 6.2% due to the liberation of the half molecule of water. The dehydration of gypsum proceeds in the following way

$$CaSO_4 \cdot 2\ H_2O \rightarrow CaSO_4 \cdot 0.5\ H_2O + 1.5\ H_2O \quad (I)$$

$$CaSO_4 \cdot 0.5\ H_2O \rightarrow CaSO_4 + 0.5\ H_2O \quad (II)$$

If the dehydration of gypsum took place in the stepwise sequence shown above, i.e. reaction (I) going to completion then reaction (II) starting, it would be simple to find a direct relation between weight loss and phase composition of the calcination products. Unfortunately, the dehydration proceeds in a different manner; the calcined product always consists of a mixture of the three phases at the same time and a variation in the calcination conditions reveals a difference in the relative amounts of each phase [6,7]. In the presence of such mixtures it is difficult to assess the actual phases present using only the weight loss data because the same value could be achieved through various mixtures with different amounts of gypsum and hemihydrate, as the hydrous forms along with anhydrite. Table 1(A) shows a number of mixes capable of giving a weight loss value typical of the pure hemidhydrate phase, i.e. the weight loss value is identical with the stoichiometric value for pure hemihydrate. Generally, for any weight loss value there is an infinte number of mix compositions that could be postulated or deduced. As another example, Table 1(B) shows some mixtures that can give a 10% value. Accordingly, the real evaluation of the phase composition of calcined plaster could not be carefully done using these data as the sole tool.

Three different samples of gypsum plasters were prepared using a comparatively pure Egyptian raw material (98% $CaSO_4 \cdot 2\,H_2O$) to avoid the interference of impurities. The chemical analysis of the raw material is shown in Table 2. The preparation conditions of the plasters and their

TABLE 1

Arbitrary mixes giving a total weight loss of (A) 6.2% and (B) 10%

Mix composition (%) [a]			Potential phase wt. loss (%)	
G	*H*	*A*	W_g	W_h
(A)				
	100.0			6.20
5	83.12	12.88	1.05	5.15
10	66.24	23.76	2.093	4.11
20	33.87	46.13	4.19	2.10
22.22	25.0	52.78	4.65	1.55
23.70	20.00	56.30	4.96	1.24
25.00	15.48	59.52	5.24	0.96
28.14	5.00	66.86	5.89	0.31
29.63		70.37	6.20	
(B)				
46.30	5.00	48.70	9.69	0.31
44.80	10.00	45.20	9.38	0.62
43.33	15.00	41.67	9.07	0.93
41.85	20.00	38.15	8.76	1.24
40.37	25.00	34.63	8.45	1.55
33.00	50.00	17.00	6.90	3.10

[a] *G* = gypsum; *H* = hemihydrate; *A* = anhydrite.

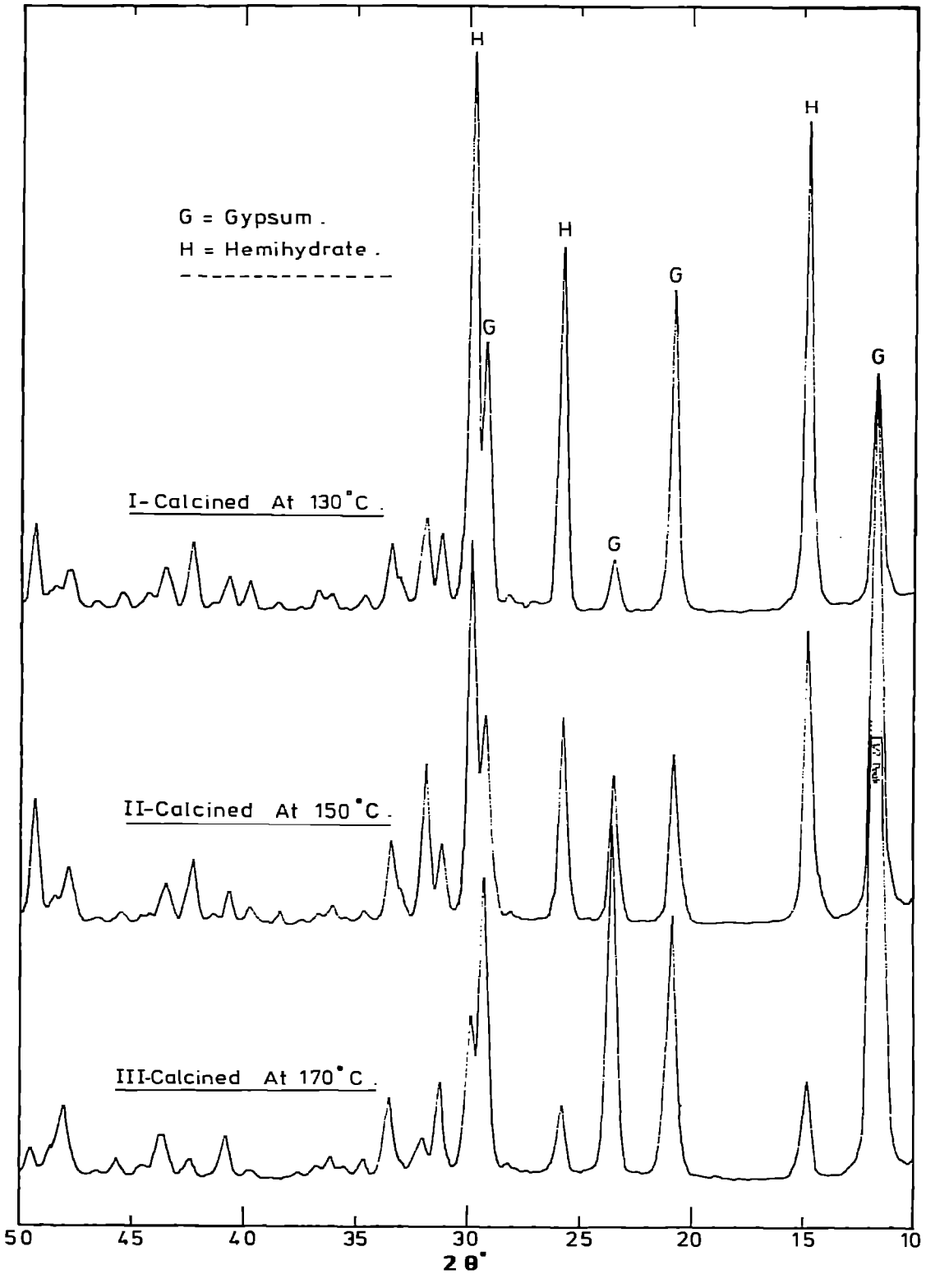

Fig. 1. X-Ray diffraction patterns of the prepared plasters.

TABLE 2

Chemical analysis of the raw material (%)

SiO_2 and insol. matter	R_2O_3	CaO	MgO	SO_3	H_2O	CO_2	NaCl	Total
0.20	0.24	32.78	0.06	46.21	20.40	Traces	0.14	100.03

TABLE 3

Conditions of preparation and weight losses of the plasters

Conditions	Weight loss (%)
Gypsum lumps (2 in.) heated at 130°C	6.80
Gypsum lumps (2 in.) heated at 150°C	6.73
Gypsum powder (150 μm) heated at 170°C	6.72

weight losses are tabulated in Table 3. The constitution of the prepared plasters was assessed by X-ray and their diffraction patterns are given in Fig. 1. From Table 3 it is clear that the prepared plasters gave nearly the same weight loss value, i.e. they retained the same amount of their water of crystallisation. Despite this empirical knowledge the three samples show different compositions. Although they reveal the presence of gypsum and hemihydrate a marked difference in their relative amounts has been observed. As the calcination temperature was increased the gypsum content increased, with the subsequent decrease of the hemihydrate, and vice versa. However, the presence of anhydrite in such plaster samples is almost expected but the great similarity in the (d) spacings of the hemihydrate and γ-anhydrite makes the interpretation of the latter difficult. Moreover, the preparation conditions of these plasters do not favour the formation of β-anhydrite [8], therefore the probability of its formation has been omitted.

WEIGHT LOSS AND CONSTITUTION

Since the constitution of the calcined plaster, in the normal case, shows the presence of gypsum, hemihydrate and anhydrite phases, it is possible to follow the thermogravimetry of the mixture as follows.

Let us have a mixture containing (X%) gypsum and (Y%) hemihydrate. The anhydrite content will be $100 - (X + Y)$. Since the weight loss is a contribution of both gypsum and hemihydrate

$$W_t = W_g + W_h$$

where W_t = weight loss of the tested sample (mixture), W_g = weight loss due to gypsum content, W_h = weight loss due to hemihydrate content, W_g = $(2\ H_2O/CaSO_4 \cdot 2\ H_2O)(X/100) = 20.93\ X$, $W_h = (0.5\ H_2O/CaSO_4 \cdot 0.5\ H_2O)$

$(Y/100) = 6.2\ Y$, $W_t = 20.93\ X + 6.2\ Y$ = measured value, and $X + Y \leqslant 1$.

This simple equation relates the weight loss to both gypsum and hemihydrate contents; accordingly, the percentage of either of them must be known, using another tool, prior to any assessment based on weight loss.

Let us consider an example of a sample consisting of 45% gypsum giving rise to 11% weight loss. The percentages of the hemihydrate and anhydrite phases may be calculated as follows. Applying the formula

$$11 = 20.93\ X + 6.2\ Y; X = 0.45$$

solve for Y ($Y = 25.5\%$). Therefore, anhydrite content = 29.5%, and the examined sample consists of 45% gypsum, 25.5% hemihydrate and 29.5% anhydrite.

GENERAL CASE

Let us consider the case when the anhydrite content is negligible, i.e. when the calcination conditions are carefully controlled so as to eliminate any anhydrite formation. The anhydrite content is supposed to be checked using another tool of assessment. In the case of the anhydrite content being equal to zero

$$X + Y = 1$$

In such a case the weight loss value can tell us directly the relative amounts of both gypsum and hemihydrate without any prior determination of either of them.

Suppose we have, as an example, calcined plaster that gives a weight loss value of 18% and we are sure of the complete absence of anhydrite in this sample. Let us find the amounts of gypsum and hemihydrate phases as follows

$$20.93(1 - Y) + 6.2\ Y = 18, \text{ where } X = 1 - Y$$

therefore, $Y = 19.892\%$ and the sample consists of 19.89% hemihydrate and 80.11% gypsum.

This deduction leads us to apply this formula in a general form if the amounts of anhydrite are available. In other words, if the anhydrite content is known it will be easy to calculate the amounts of gypsum and hemihydrate. Let us take the example when the calcined plaster gives 10% weight loss and the anhydrite content is measured as 17%. Applying the formula, it is easy to determine the amounts of gypsum and hemihydrate in this sample to be 33 and 50%, respectively. Similarly, if the amounts of either gypsum or hemihydrate are available the calculation of the remaining phases can be achieved.

It can be concluded that if the amount of any of these three phases is known, it will be possible to use the weight loss data satisfactorily for the real assessment of gypsum plasters.

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