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## X-RAY PATTERNS OF HYDRATED CALCIUM SILICATES

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## ABSTRACT

X-ray powder diffraction data of 15 hydrated calcium silicates are reported as a supplement to a previously published report on the formation of these compounds. These data may be useful to workers on portland cement hydration, boiler scale, etc., for the identification of phases.

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## I. INTRODUCTION

Some years ago a report<sup>1</sup> was published on the hydrothermal synthesis of hydrated calcium silicates. In the course of that study the crystalline products of hydrothermal action were identified in part by means of X-ray powder patterns. While the patterns made then were satisfactory for the purpose, they were not considered sufficiently sharp and accurate to justify measuring and reproducing the resultant data in the paper.

Since that time, however, there has been considerable interest in the hydrated calcium silicates because of their occurrence in the hydration products of portland cement, in boiler scales, etc. Therefore, the patterns of the compounds were remade on more modern equipment and the data are given here.

## II. METHODS AND SOURCES OF MATERIALS

Of the 15 calcium silicate hydrates considered here seven occur as natural minerals, and these natural minerals were used to prepare the patterns. The other eight compounds were formed as described in the paper referred to above.<sup>2</sup>

Table 1 gives the source of the minerals and the starting material and subsequent treatment of the synthetic preparations. The experi-

<sup>1</sup> Einar P. Flint, Howard F. McMurdie, and Lansing S. Wells, *Formation of hydrated calcium silicates at elevated temperatures and pressures*, J. Research NBS **21**, 617 (1938) RP1147.

<sup>2</sup> In the original paper, table II, the compound  $10\text{CaO} \cdot 5\text{SiO}_2 \cdot 6\text{H}_2\text{O}$  is indicated as not being obtained synthetically. This is an error. It was made synthetically; in fact, it has not been found as a mineral.

ment numbers from the previous paper are also given and in that paper a complete discussion of the methods used is presented. Some of these phases were made at the time of the previous study, others were remade, and all were examined with the petrographic microscope to assure reasonable purity.

The patterns were registered with cameras of about 5.7-cm radius, using  $\text{CuK}\alpha$  radiation.

TABLE 1.—*Origin of materials used in making the X-ray patterns*

## A. MINERALS

Mineral	Composition	Place of origin
Okenite.....	$\text{CaO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ .....	Discos Island, Greenland.
Gyrolite.....	$2\text{CaO} \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ .....	Benkoelen, Sumatra, Netherlands East Indies.
Xonotlite.....	$5\text{CaO} \cdot 5\text{SiO}_2 \cdot \text{H}_2\text{O}$ .....	Isle Royale, Mich.
Crestmoreite.....	$2\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$ .....	Crestmore, Riverside County, Calif.
Foshagite.....	$5\text{CaO} \cdot 3\text{SiO}_2 \cdot 3\text{H}_2\text{O}$ .....	Do.
Afwillite.....	$3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$ .....	Dutoitspan Mine, Kimberley, South Africa.
Hillebrandite.....	$2\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ .....	Velardena District, Durango, Mexico.

## B. SYNTHETIC PREPARATIONS

Composition	Method of preparation				Experiment No. <sup>a</sup>
	Starting material	Temperature	Pressure	Time	
$4\text{CaO} \cdot 5\text{SiO}_2 \cdot 5\text{H}_2\text{O}$ .....	$0.80\text{CaO} \cdot \text{SiO}_2$ aq.....	$^{\circ}\text{C}$ 225	<i>atm</i> 25	14 days.....	20
$\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ .....	$\text{CaO} \cdot \text{SiO}_2$ aq.....	150	5	60 days.....	28
$6\text{CaO} \cdot 4\text{SiO}_2 \cdot 3\text{H}_2\text{O}$ .....	$\beta 2\text{CaO} \cdot \text{SiO}_2$ .....	25	1	10 years <sup>b</sup> .....	61
$2\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}^c$ .....	$2\text{CaO} \cdot \text{SiO}_2$ aq.....	225	25	44 days.....	63
$2\text{CaO} \cdot \text{OSiO}_2 \cdot \text{H}_2\text{O}^d$ .....	$\beta 2\text{CaO} \cdot \text{SiO}_2$ .....	250	39	15 days.....	65
$10\text{CaO} \cdot 5\text{SiO}_2 \cdot 6\text{H}_2\text{O}$ .....	$\beta 2\text{CaO} \cdot \text{SiO}_2$ .....	200	15	3 days.....	66
$6\text{CaO} \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ .....	$10\text{CaO} \cdot 5\text{SiO}_2 \cdot 6\text{H}_2\text{O}$ .....	450	400	7 days.....	70
$3\text{CaO} \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O}$ .....	$3\text{CaO} \cdot \text{SiO}_2$ .....	250	39	12 days.....	75

<sup>a</sup> Experiment numbers from previous paper (J. Research NBS 21, 617 (1938) RP1147).

<sup>b</sup> Later treated at 175° C in bomb for 41 days.

<sup>c</sup> Index of refraction = 1.60.

<sup>d</sup> Index of refraction = 1.64.

## III. RESULTS

Table 2 gives the interplanar spacings ( $d$ ) and estimated relative intensities for 15 calcium silicate hydrates. These compounds all have symmetry which is orthorhombic or lower; therefore, it is all but impossible to index the reflections.

TABLE 2.—Diffraction data of 15 hydrated calcium silicates

Estimated intensity	d	Estimated intensity	d	Estimated intensity	d	Estimated intensity	d
Okenite (CaO.2SiO <sub>2</sub> .2H <sub>2</sub> O)		Crestmoreite (2CaO.2SiO <sub>2</sub> .3H <sub>2</sub> O)		Afwillite 3CaO.2SiO <sub>2</sub> .3H <sub>2</sub> O—Con.		CaO.SiO <sub>2</sub> .H <sub>2</sub> C—Continued	
VS	A 8.91	M	A 14.9	VW	A 2.065	M	A 2.78
W	7.67	S	10.4	W	2.026	M	2.50
VW	6.63	W	5.5	W	1.988	S	2.23
VW	4.22	W	4.9	M	1.955	VW	2.116
M	3.59	VW	4.14	W	1.924	W	2.013
M	3.09	W	3.74	W	1.868	S	1.893
M	2.99	M	3.50	M	1.868	VW	1.868
S	2.96	VW	3.29	M	1.782	VW	1.808
S	2.83	VS	3.09	VW	1.735	M	1.772
VW	2.73	S	2.95	W	1.705	VW	1.632
VW	2.57	VS	2.84	W	1.679	W	1.601
VW	2.41	M	2.74	W	1.629	6CaO.4SiO <sub>2</sub> .3H <sub>2</sub> O	
W	2.16	W	2.66	M	1.606		
VW	1.897	VW	2.34	M	1.596		
M	1.823	W	2.26	W	1.564		
		VW	2.18				
		W	2.05			M	4.30
Gyrolite (2CaO.3SiO <sub>2</sub> .H <sub>2</sub> O)		M	1.959	Hillebrandite (2CaO.SiO <sub>2</sub> .H <sub>2</sub> O)		W	3.68
M	9.6	M	1.861	S	12.6	W	3.45
W	7.9	M	1.819	VW	4.77	VW	3.39
W	6.4	VW	1.792	VW	4.05	M	3.22
W	4.68	W	1.773	VW	3.52	VS	3.10
S	4.24	W	1.735	M	3.34	W	2.98
M	3.84	Foshagite (5CaO.3SiO <sub>2</sub> .3H <sub>2</sub> O)		VW	3.01	VW	2.94
W	3.54	M	9.7	VS	2.92	W	2.86
VS	3.36	M	8.0	VW	2.83	W	2.73
M	3.15	M	5.67	VW	2.75	W	2.64
VW	3.02	M	5.06	VW	2.68	W	2.58
VW	2.92	M	3.42	VW	2.62	S	2.253
S	2.85	S	3.36	VW	2.45	S	2.066
M	2.65	M	3.24	M	2.373	W	2.034
VW	2.58	W	3.09	M	2.247	VW	1.987
VW	2.52	S	2.93	W	2.056	M	1.943
W	2.45	S	2.81	M	2.247	VW	1.897
W	2.29	M	2.72	W	2.056	W	1.833
M	2.25	W	2.48	M	1.963	W	1.792
W	2.12	VS	2.31	W	1.928	W	1.756
W	2.065	S	2.16	M	1.864	S	1.699
VW	2.060	W	2.11	W	1.812	2CaO.SiO <sub>2</sub> .H <sub>2</sub> O (n=1.60)	
VW	1.990	W	2.075	W	1.747		
VW	1.940	W	2.040	4CaO.5SiO <sub>2</sub> .5H <sub>2</sub> O		W	4.83
S	1.884	W	1.950	S	4.22	M	4.26
M	1.813	VW	1.916	W	3.76	W	3.55
Xonotlite (5CaO.5SiO <sub>2</sub> .H <sub>2</sub> O)		W	1.827	M	3.14	W	3.38
M	7.10	W	1.776	S	3.05	W	3.28
S	4.22	S	1.745	W	2.85	S	3.05
VS	3.61	Afwillite (3CaO.2SiO <sub>2</sub> .3H <sub>2</sub> O)		W	2.67	VS	2.93
S	3.24	S	6.5	VW	2.52	VW	2.42
S	3.07	M	5.06	W	2.52	W	2.40
M	2.82	W	4.68	W	2.30	W	2.24
M	2.68	W	4.18	M	2.105	W	2.07
VW	2.62	VW	3.76	M	1.924	M	1.963
M	2.49	VS	3.17	W	1.877	M	1.875
W	2.41	VW	3.05	M	1.841	M	1.819
W	2.25	VS	2.85	CaO.SiO <sub>2</sub> .H <sub>2</sub> O		W	1.637
S	2.03	S	2.74	VW	6.13	2CaO.SiO <sub>2</sub> .H <sub>2</sub> O (n=1.64)	
S	1.940	W	2.66	W	4.52	W	4.22
M	1.826	W	2.59	W	4.24	VW	3.82
W	1.748	VW	2.44	VW	3.80	VS	3.04
M	1.705	M	2.36	W	3.58	M	2.84
W	1.679	VW	2.33	M	3.21		
		M	2.275	VS	3.01		
		M	2.159				

1S, strong; M, medium; W, weak; V, very; B, broad.

