Portland Cement Hydration

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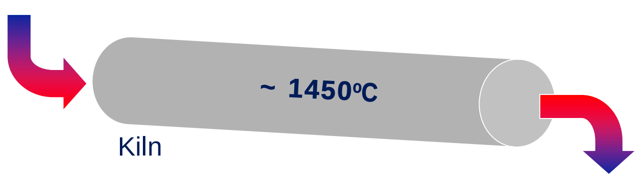
Cement Composition

$CaCO_3$ (limestone)
$2SiO_2 \cdot Al_2O_3$ (clay, shale)
Fe_2O_3 (iron oxide)
SiO_2 (silica sand)

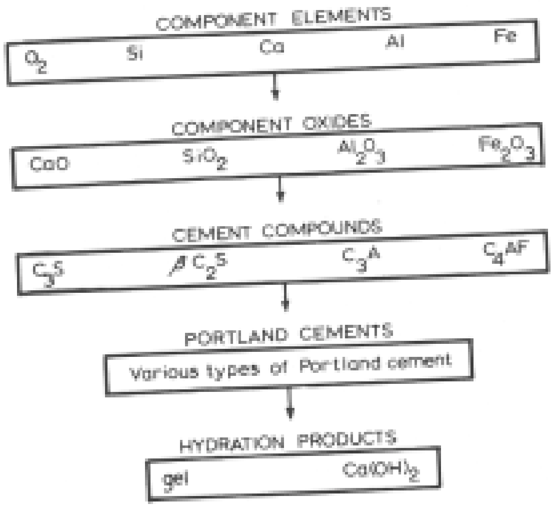


$3CaO \cdot SiO_2$
$2CaO \cdot SiO_2$
$3CaO \cdot Al_2O_3$
$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$

$CaO \cdot SO_3 \cdot 2H_2O$
Gypsum + Clinker

interground → Finished cement

Cement Composition



+ impurities (K, Na, Mg, etc)

- the presence and substitution (e.g., Na^+ for Ca^{2+}) of impurities can lead to polymorphism, structural "holes", and greater reactivity

Cement Hydration

<u>Hydration</u> - chemical combination of cement and water

Two primary mechanisms:

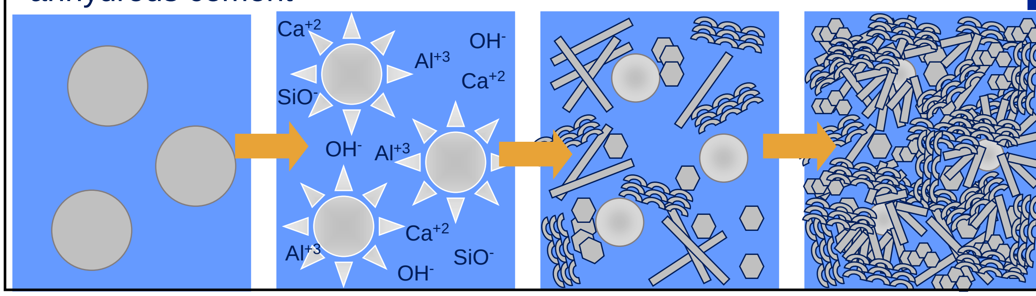
<u>Through solution</u> - involves dissolution of anhydrous compounds to their ionic constituents, formation of hydrates in solution, and eventual precipitation due to their low solubility

<u>Topochemical</u> - or solid-state hydration - reactions take place directly at the surface of the anhydrous cement compounds without going into solution

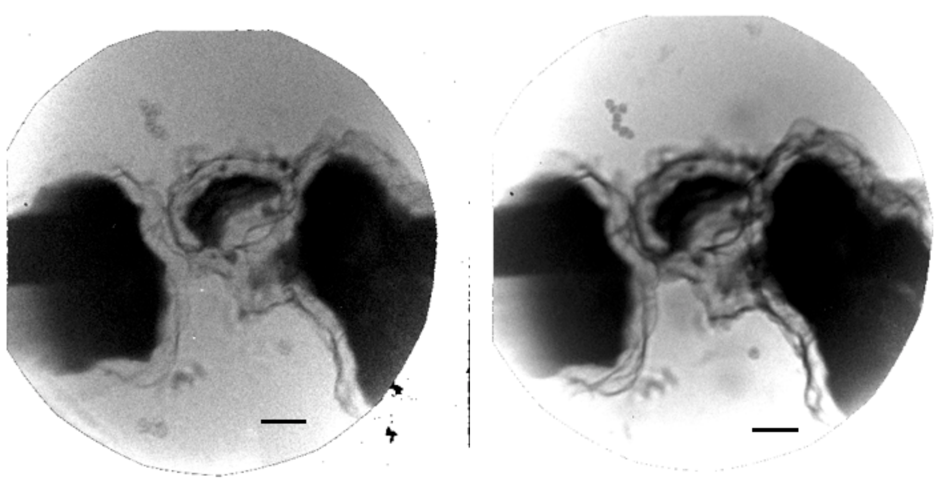
Cement Hydration

When water is added to cement, what happens?

- Dissolution of cement grains
- Growing ionic concentration in “water” (now a solution)
- Formation of compounds in solution
- After reaching a saturation concentration, compounds precipitate out as solids (“hydration products”)
- In later stages, products form on or very near the surface of the anhydrous cement



Cement Hydration



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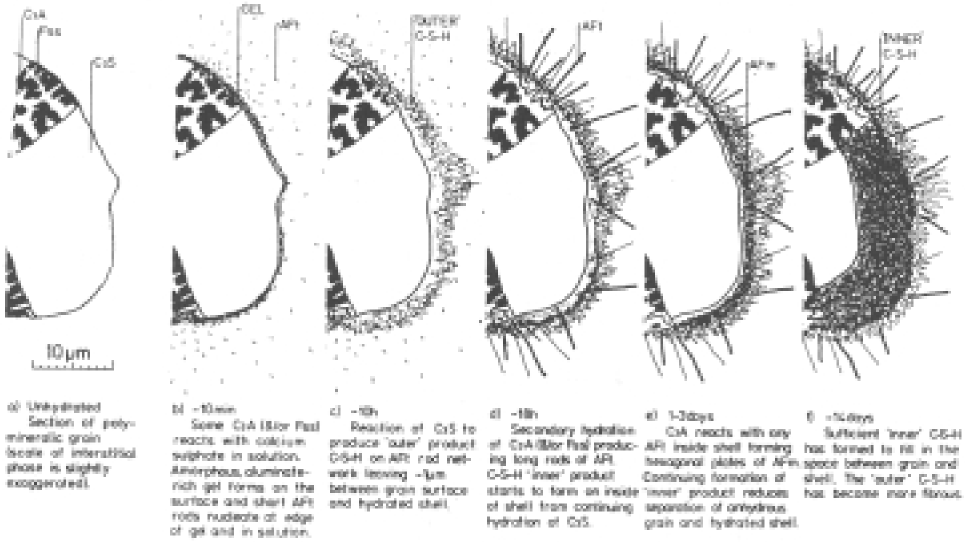
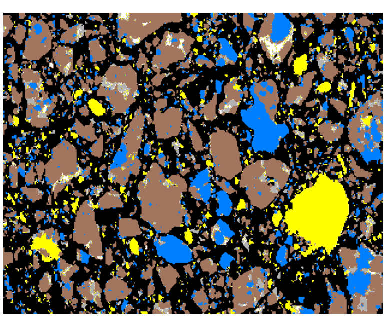
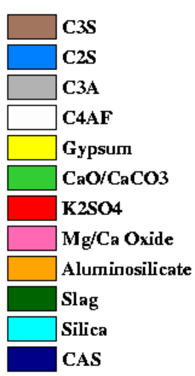


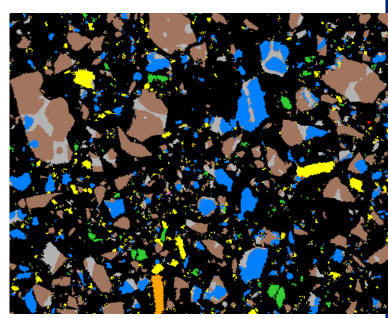
Fig. 7.3 *Development of microstructure during the hydration of Portland cement. From S34.*

Taylor, Cement Chemistry

Cement Hydration







C_3S	54%
C_2S	32%
C_3A	7%
C_4AF	4%

C_3S	52%
C_2S	30%
C_3A	17%
C_4AF	0%

Image Credit: Bentz at NIST

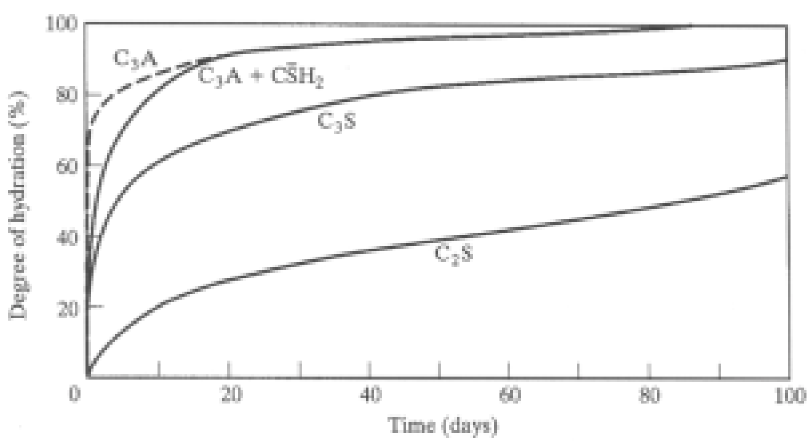
Cement Hydration

Because the hydration rates of the 4 key phases vary considerably, properties like

- time to stiffening
- setting time
- hardening rate

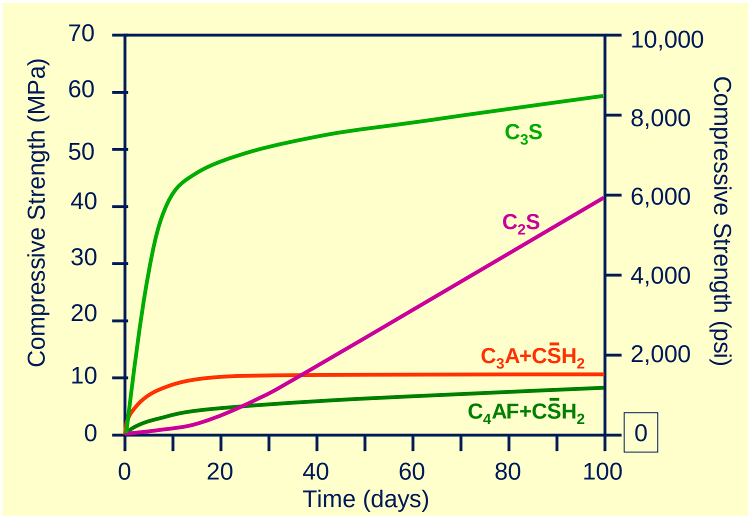
will vary with cement composition.

Cement Hydration



Taylor, Cement Chemistry

Cement Hydration



Compressive Strength development in pastes of pure cement compounds (*Mindess et al, 2003*)

Properties of Hydrated Cement Compounds

C_3S	Tricalcium silicate (alite)	Hydrates & hardens rapidly Responsible for initial set and early strength

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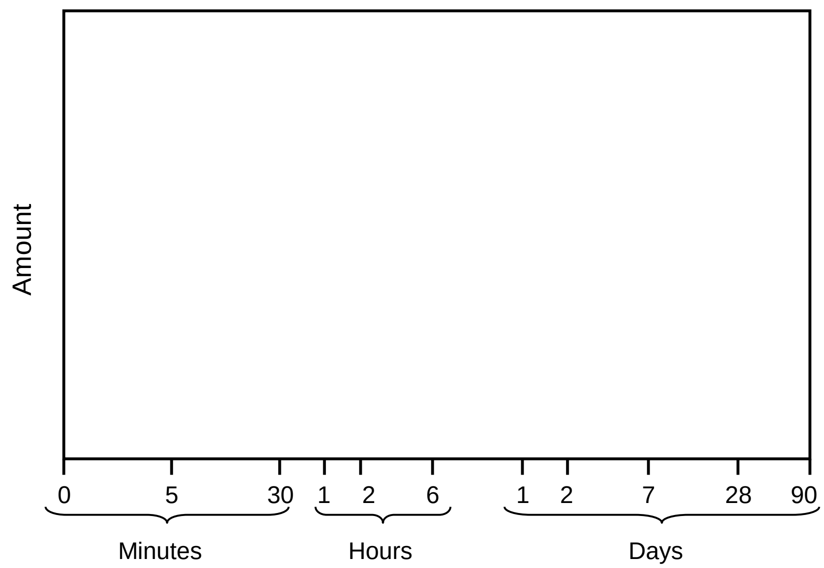
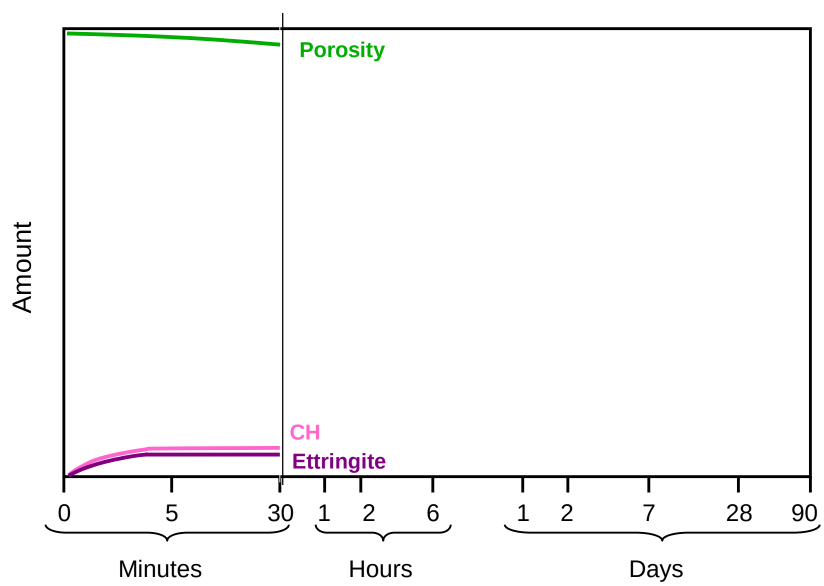
C_4AF	Tetracalcium aluminoferrite (ferrite)	Reduces clinkering temperature Hydrates rapidly but contributes little to strength Colour of hydrated cement (gray) due to ferrite hydrates

TABLE 3.4 Characteristics of Hydration of the Cement Compounds

Compounds	*Reaction Rate*	*Amount of Heat Liberated*	*Contribution to Cement Strength*	*Contribution to Cement Heat Liberation*
C_3S	Moderate	Moderate	High	High
C_2S	Slow	Low	Low initially, high later	Low
$C_3A + C\bar{S}H_2$	Fast	Very high	Low	Very high
$C_4AF + C\bar{S}H_2$	Moderate	Moderate	Low	Moderate


Amount
0
5
30
1
2
6
1
2
7
28
90
Minutes
Hours
Days


Porosity
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Amount
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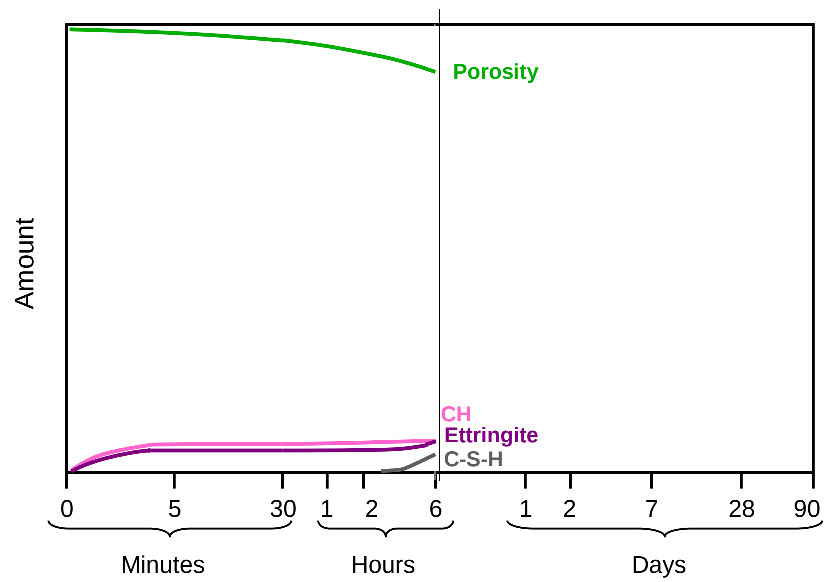
Porosity
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C-S-H
Amount
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1
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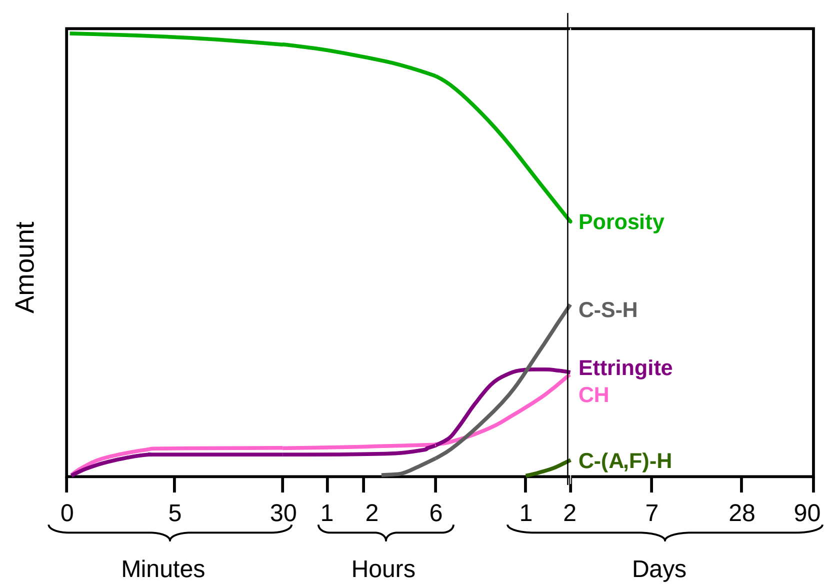
Porosity
C-S-H
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Amount
0
5
30
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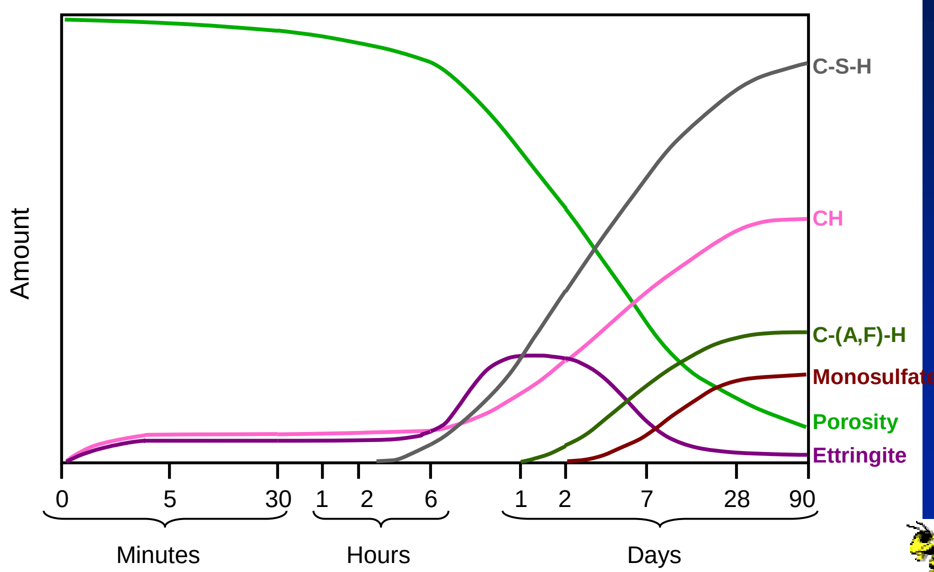


Cement Hydration

- Is the chemical combination of cement and water to form hydration products
- Takes time
- May not proceed to 100% completion

Formation of hydration products over time leads to:

- Stiffening (loss of workability)
- Setting (solidification)
- Hardening (strength gain)

Let’s look at the hydration reactions in more detail…

Hydration of the Calcium Silicates

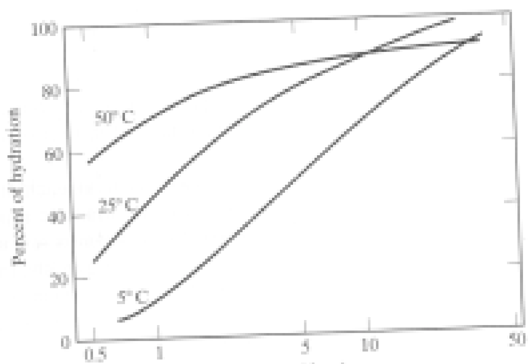
$2C_3S + 7H \rightarrow C_3S_2H_8 + 3CH$ ΔH=-500J/g

$2C_2S + 7H \rightarrow C_3S_2H_8 + CH$ ΔH=-250J/g

- Both produce C-S-H and CH as reaction products
- C_2S produces less CH (important for durability in sulfate rich environments)
- More heat is evolved during C_3S hydration
- C_3S hydration is more rapid, contributing to early age strength (2-3h to 14 days)
- C_2S hydration occurs more slowly to contributing to strength after ~7-14 days.

Hydration of the Calcium Silicates

Like most chemical reactions, the rate of cement hydration is influenced by temperature.



C-S-H

- Calcium silicate hydrate
- C/S varies between 1.1-2; ~1.5 is typical
- H is even more variable
- Structure ranges from poorly crystalline to amorphous - highly variable and poorly understood
- Occupies 50-60% of the solid volume of the hydrated cement paste (hcp)
- Huge surface area (100-700 m^2/g)
- Strength due to covalent/ionic bonding (~65%) and Van der Waals bonding (~35%) within the complex structure
- Primary strength-giving phase in portland cement concrete

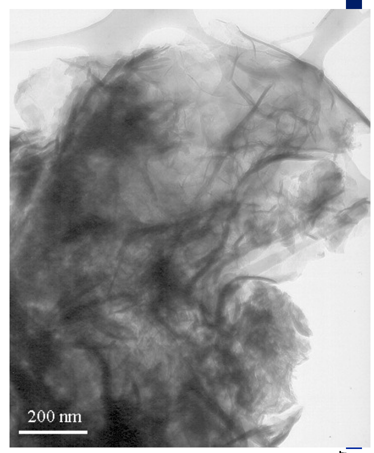
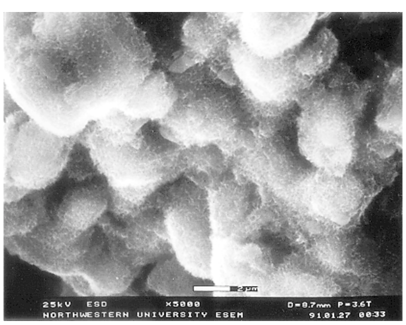


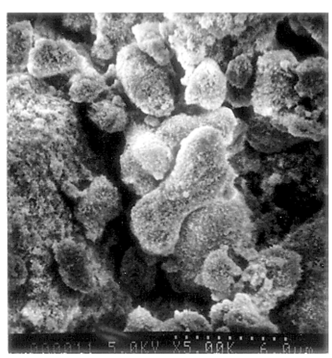
image credit: Dr. Eric Lachowski, S.Y. Hong, and F.P. Glasser via Concrete Microscopy Library at UIUC

C-S-H

The structure of C-S-H is poorly understood.



wet



dry

16 hr. C_3S paste

Variations in surface area, depending on technique used

$$\left\{ \begin{array}{ll} S_{H2O} & = 200\ m^2/g \\ S_{N2} & = 5\text{-}50\ m^2/g \\ S_{neutrons} & = 50\ m^2/g \end{array} \right.$$

C-S-H

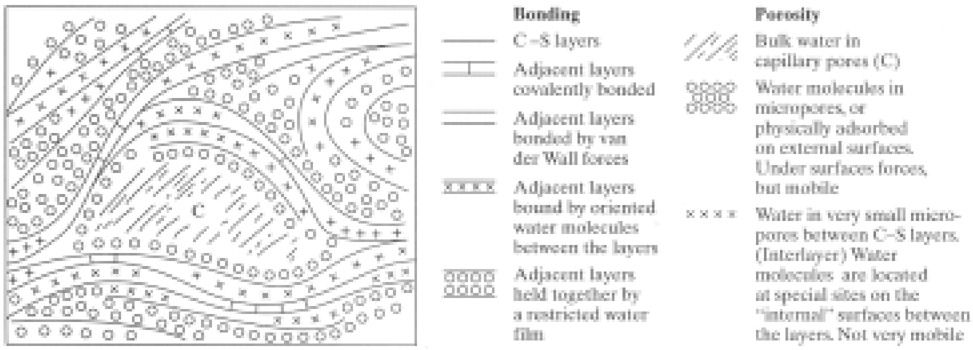
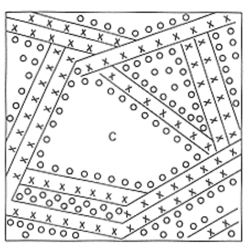


Figure 11.8
Schematic description of the structure of C—S—H.

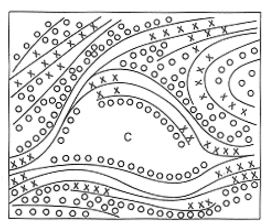
Figure credit: M&M text

Some other models for C-S-H

Adapted from Powers (1960's)

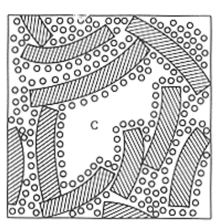


(a)



(b)

Feldman and Sereda (1970)



(c)

Munich model, Wittman (1979)

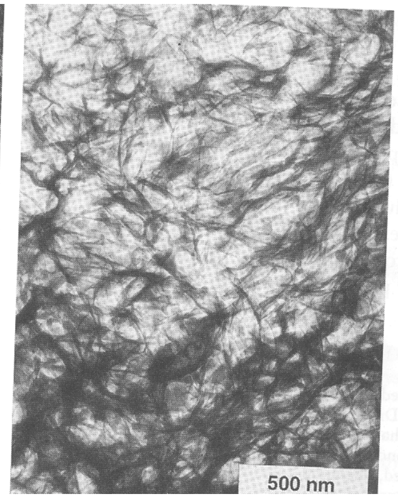
Colloid model

x Water in interlayer regions
o Water adsorbed on surfaces
C Capillary pore
C-S-H sheets
C-S-H particles (no designated structure)

Figure credit: M&M text

Microscopical Evidence

Fine, fibrillar structure, similar to Power's model (typical of Ca/Si>1.5)



Foil-like structure, similar to Feldman-Sereda model (typical of lower Ca/Si)

Figure credit: Richardson, in Structure and Performance of Cements, Bensted and Barnes (Eds), Spon 2002.

Some other models for C-S-H

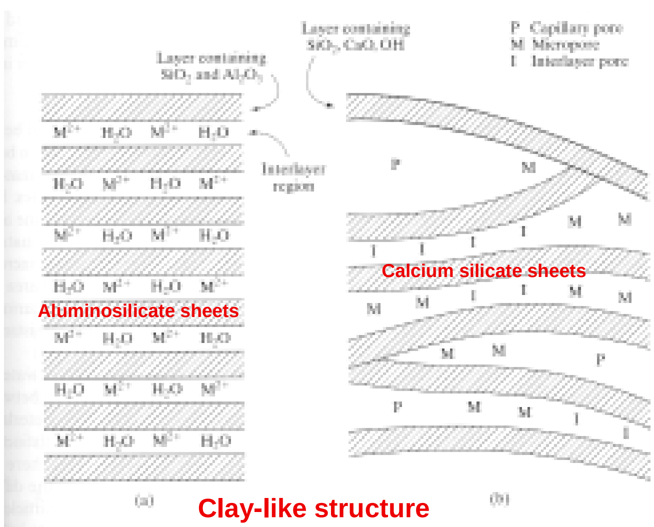
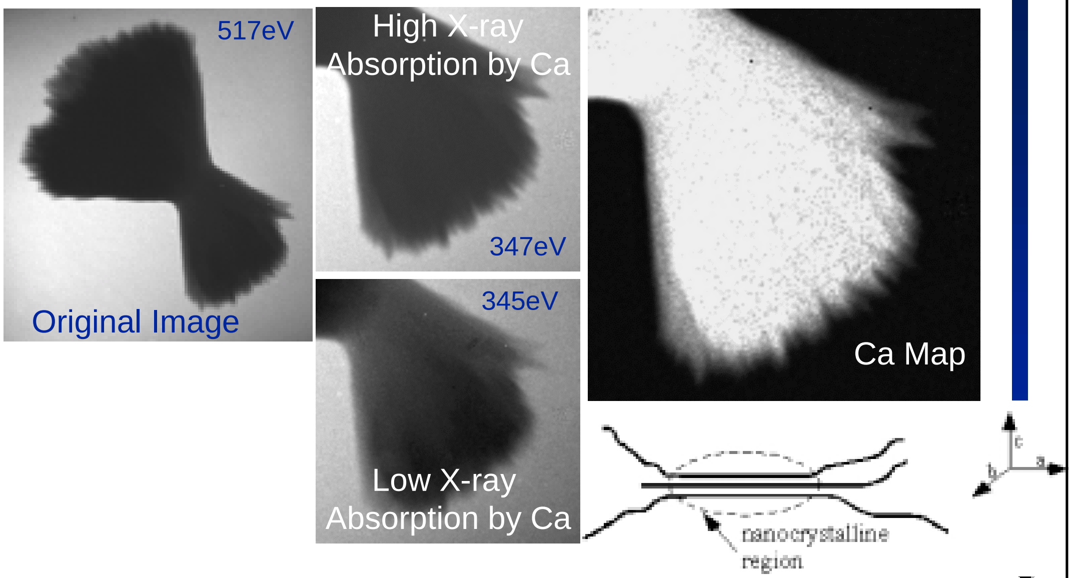


FIGURE 4.5

Schematic model of C-S-H in cement paste: (a) well-crystallized clay mineral; (b) poorly crystallized C-S-H.

Some other models for C-S-H



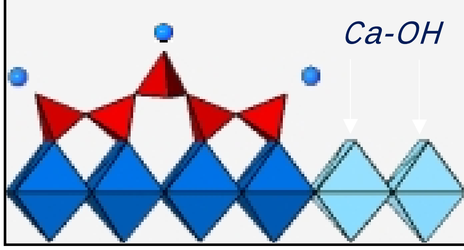
E. Gartner, K.E. Kurtis, and P.J.M. Monteiro, *Cement and Concrete Research*, May 2000, V30 (5):817-822.

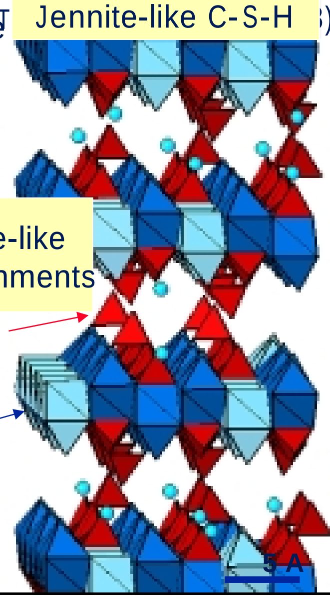
Some other models for C-S-H

At low C/S, C-S-H resembles tobermorite
At high C/S, C-S-H resembles jennite

Higher C/S ratios are obtained by 3 principle mechanisms.

3 Replacement of silicate chains with hydroxyls, producing Ca-OH.





Summary of Models for C-S-H

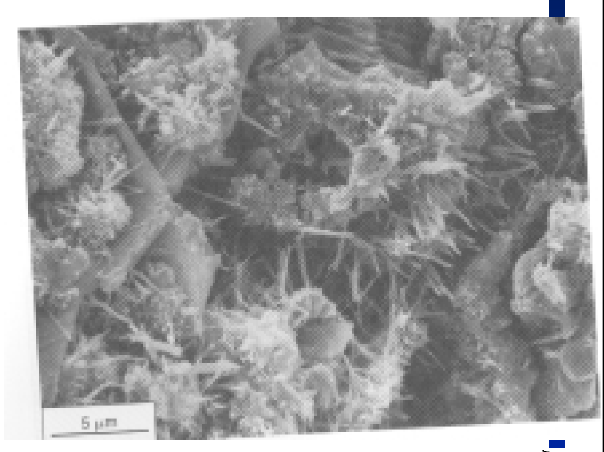
Table 6: Summary of models for the structure of C-S-H.

Name of Model	Primary Experimental Basis	Type of Model	Selected Characteristics of Model
Powers	Water sorption Volume of pores	Colloid	All products are gel Particle radius, 5 nm Gel pore volume, 28%
Taylor	X-ray TGA	Imperfect Tobomerite Jennite	Atomic structure of C-S-H
Brunauer	Water Sorption	2-3 layers	Structure changes upon drying
Feldman-Sereda	Nitrogen sorption Length vs. RH Modulus vs. RH Weight vs. RH	Layers	Crumpled and folded layers with interlayer water reversibly removed upon drying
Wittmann	Modulus vs. RH	Colloid	Structure not defined
Jennings	Density vs. RH Composition vs. RH Surface area	Colloid	Fractal: density and surface area depend on length scale

Inner vs. Outer Product C-S-H

Outer product (early) C-S-H/ groundmass

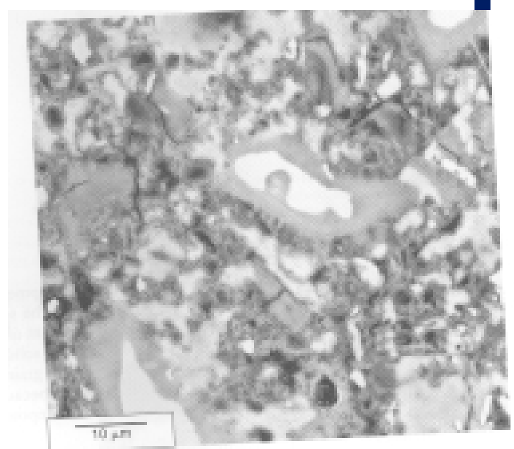
- forms during early hydration
- C-S-H forms away from the cement particle surface, filling water-filled space
- higher porosity
- contains high levels of impurities
- probably admixed with nano-scale $C_4A\check{S}H_{12}$



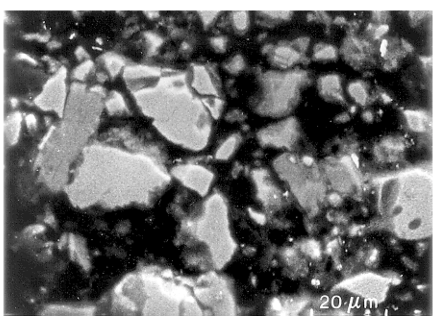
Inner vs. Outer Product C-S-H

Inner product (late) C-S-H/ phenograins

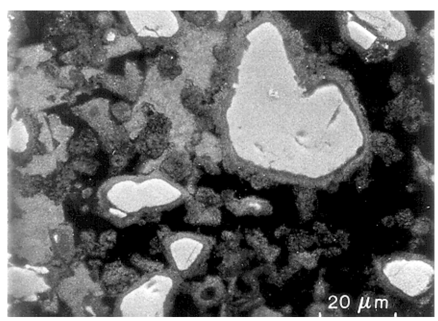
- forms during later hydration, when the process is diffusion controlled
- C-S-H grows inwards and outwards from the C-S-H “barrier”
- C-S-H formed takes shape of cement grains
- lower porosity, more dense
- fewer impurities
- more resistant to physical change on drying
- more abundant as hydration ↑ or as w/c ↓



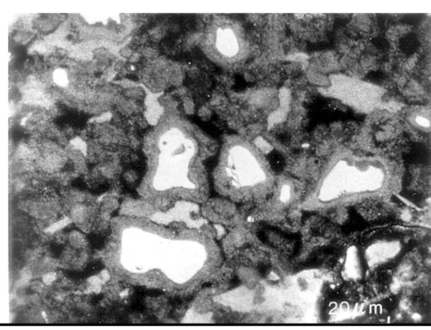
C_3S Hydration



1 Day



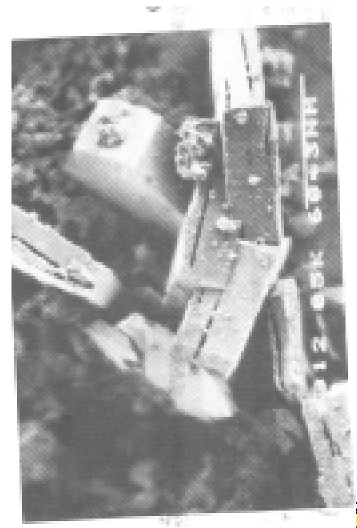
3 Days



28 Days

CH

- Calcium hydroxide or $Ca(OH)_2$
- Definite stoichiometry
- Variable morphology - from large, hexagonal prisms to thin, elongated crystals
- Size of the crystals depends on the amount of space available
- Occupies 20-25% of the solid volume in the hcp
- Much lower surface area than C-S-H
- Does not contribute much to strength
- Keeps the pore solution alkaline (pH 12.4-13.5)



Hydration of the Calcium Aluminates

- Reaction of C_3A with water occurs *very* quickly and liberates much heat - "Flash Set"
- Gypsum ($C\check{S}H_2$) is added to the cement to control the hydration of C_3A

$C_3A + 3C\check{S}H_2 + 26H \rightarrow C_6A\check{S}_3H_{32}$ $\qquad \Delta H = -1350 J/g$

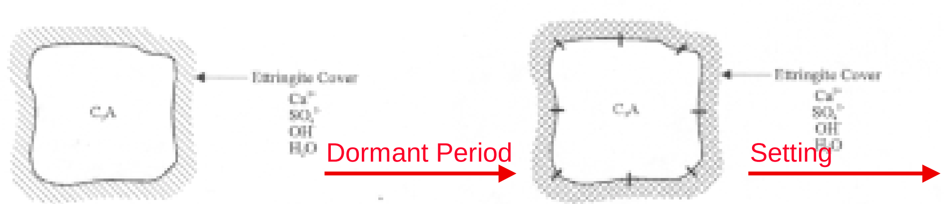


Figure 1: First stage - formation of a thin cover of ettringite on the C_3A surface

Figure 2: Second stage - a further amount of ettringite is formed on the C_3A surface

Role of Gypsum

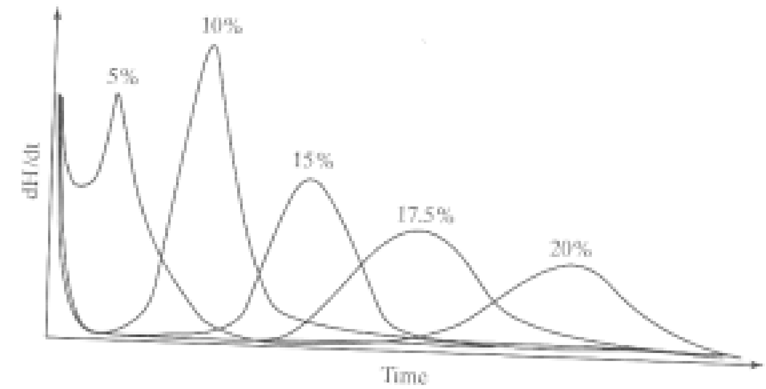


FIGURE 4.3

Rate of heat evolution during hydration of tricalcium aluminate with gypsum. (Wt. % of gypsum added is given for each curve.)

Role of Gypsum

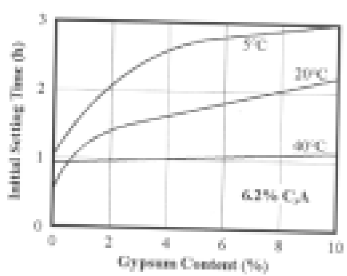


Figure 3: Variation of setting time with gypsum content (6.2% C_3A)

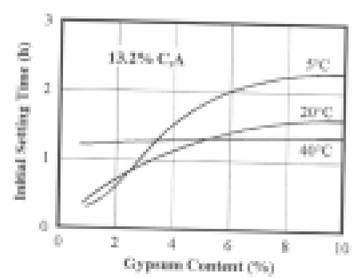


Figure 4: Variation of setting time with gypsum content (13.2% C_3A)

Hydration of the Calcium Aluminates

When more C_3A remains,

$C_6A\check{S}_3H_{32} + 2C_3A + 4H \rightarrow 3C_4A\check{S}H_{12}$

- Reaction of C_4AF occurs more slowly

$C_4AF + 2CH + 14H \rightarrow C_4(A,F)\ H_{13} + (A,F)H_3$

Hydration of the Calcium Aluminates

- Reaction of C_4AF (ferrite) phase are slower and evolve less heat than C_3A
- Also heavily retarded by gypsum

$C_4AF + 3C\check{S}H_2 + 21H \rightarrow C_6(A,F)\check{S}_3H_{32} + (F,A)H_3$

$C_4AF + C_6(A,F)\ \check{S}_3H_{32} \rightarrow 3C_4(A,F)\check{S}H_{12} + (F,A)H_3$

- Products of C_4AF are more resistant to sulfate attack than those of C_3A hydration

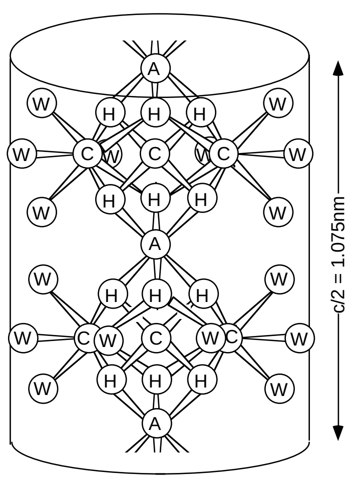
Hydration of the Calcium Aluminates

$C_6A\check{S}_3H_{32}$ (Ettringite, A_{ft})

- Needle-like morphology
- Needles interlock, take up much water
- contributes to stiffening of mixture
- some early strength

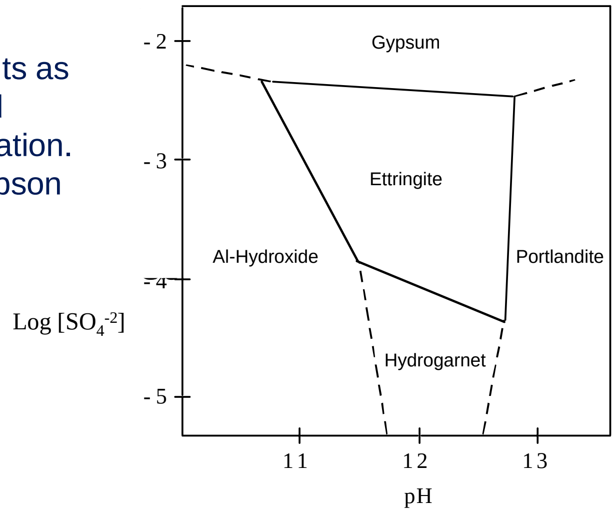
Hydration of the Calcium Aluminates

Ettringite crystal structure as part of a single column projection where A=Al, C=Ca, H=O of an OH group, W=O of an H_2O molecule. Hydrogen atoms have been omitted, as are the H_2O molecules attached to the calcium atoms lying in the central vertical line of the figure. (based on Taylor, 1997)



Hydration of the Calcium Aluminates

Ettringite stability in alkaline environments as a function of pH and sulfate ion concentration. (adapted from Hampson and Bailey, 1982)



Hydration of the Calcium Aluminates

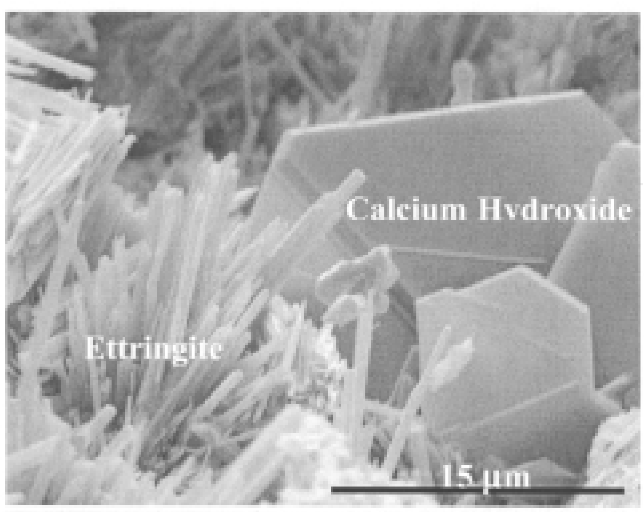
$C_4A\check{S}H_{12}$ (monosulfate, A_{fm})

- hexagonal plate morphology arranged in “rosettes” during early hydration
- become more “platey” with continued hydration
- can contain impurities
- vulnerable to sulfate attack

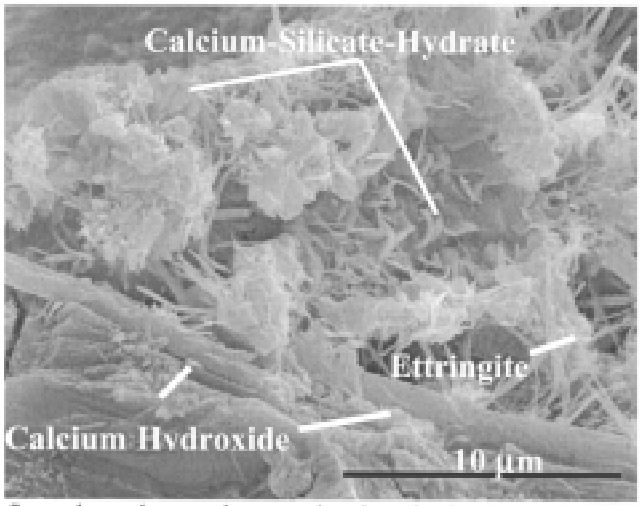
Calcium Aluminates and Calcium Sulfoaluminates

- Includes ettringite, monosulfate hydration, calcium aluminate hydrates, and ferric-aluminum hydroxide gels
- Comprise 15-20% of solid volume of hcp
- Do not contribute much to strength
- Formation of ettringite, in particular, does influence setting time
- High heat of hydration for C_3A can be favorable or unfavorable, depending upon application

Hydrated Cement Paste (hcp)



Hydrated Cement Paste (hcp)



Hydrated Cement Paste (hcp)

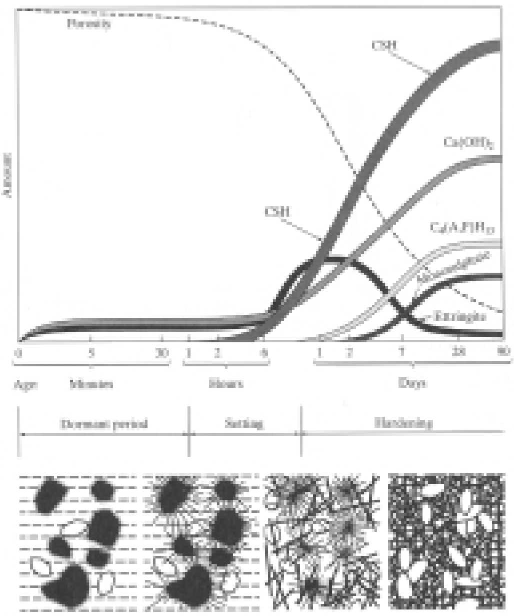
TABLE 4.4 Summary of Properties of the Hydration Products of Portland Cement Compounds

Compound	*Specific Gravity*	*Crystallinity*	*Morphology in Pastes*	*Typical Crystal Dimensions in Pastes*	*Resolved by*[a]
C–S–H	2.3–2.6[b]	Very poor	Spines: Unresolved morphology	1 × 0.1 μm (Less than 0.01 μm thick)	SEM, TEM
CH	2.24	Very good	Nonporous striated material	0.01–0.1 mm	OM, SEM
Ettringite	~1.75	Good	Long slender prismatic needles	10 × 0.5 μm	OM, SEM
Monosulfo-aluminate	1.95	Fair–good	Thin hexagonal plates; irregular "rosettes"	1 × 1 × 0.1 μm	SEM

[a]OM, optical microscopy; SEM, scanning electron microscopy; TEM, transmission electron microscopy.

Formation of hydration products over time leads to:

- Stiffening (loss of workability
- Setting (solidification)
- Hardening (strength gain)



Cement Hydration

TABLE 11.4 Influence of Cement Compounds on Concrete Properties

Property	Compound	Remarks
Setting behavior	C_3S	Controls normal setting
	C_3A	Can cause premature stiffening
Temperature rise during hydration	C_3S	
	C_3A	
Strength development	C_3S	Responsible for early strength
	C_2S	Contributes to long-term strength
Creep and shrinkage	C_3S, C_2S	Major contributions
	C_3A, C_4AF	Minor effects
Durability	C_3S	Leaching of $Ca(OH)_2$, sulfate attack
	C_3A	Sulfate attack

Heat of Hydration

- Cement hydration is exothermic
- Concrete is an insulator

Heat of hydration can be:

- detrimental (thermal gradients --> cracking)

- helpful (heat provides activation energy when concreting in cold weather; higher early strength)

Heat of Hydration

Heat evolution can be used to map the progress of hydration:

(1) Initial dissolution of solids (increasing ionic concentration)

(2) Induction period

(3) Acceleration

(4) Deceleration

(5) Steady state

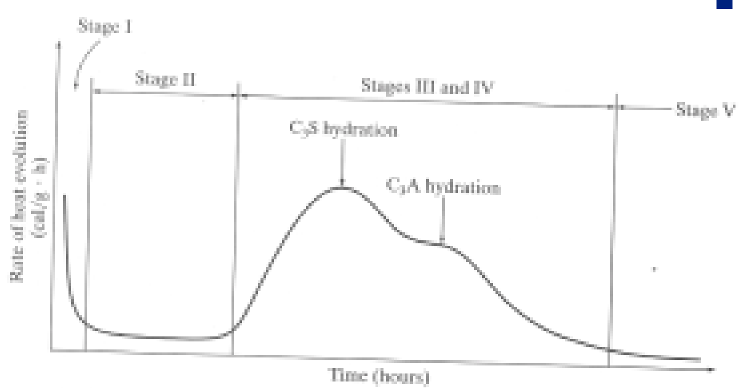


Figure 11.4
Rate of heat evolution during the hydration of portland cement (after S. Mindess and J. F. Young, *Concrete*, Prentice Hall, 1981, Fig. 4.4, p. 85).

Cement Hydration: Avrami Model*

- Popular model for describing hydration during the acceleration periods (Stages 2&3)

$$-\ln(1-\alpha)=[k(t-t_o)]^m$$

or when α is small, $\alpha=kt^m$

Where α is degree of hydration

t is time of hydration, where t_o corresponds to the length of induction period

k is a rate constant for a nucleation-controlled process

m = [(p/s)+q], where p= 1 for1D growth (needles/fibers)

m ~ 1-3 for C_3S — 2 for 2D (sheets/plates)

3 for 3D isotropic growth (sphere)

s=1 for interface or phase-boundary-controlled growth

=2 for diffusion-controlled growth

and q=0 for no nucleation (nucleation saturation)

1 for continuous nucleation at a constant rate

* Avrami, M. *J. Phys. Chem.*, 7, 1103 (1938), 8, 212 (1940).

Cement Hydration: Avrami Model

- k, then, is a combined rate constant, accounting for rate of nucleation, rate of product growth, and other factors not accounted for (e.g., changing diffusion coefficients)
- Can calculate the rate constant k from calorimetry data and the Avrami equation:

$$-\ln(1-\alpha)=[k(t-t_o)]^m$$

- when modeling as a function of time rather than degree of hydration:

$$d\alpha/dt=Amk^m(t-t_o)^{m-1}\exp\{-[k(t-t_o)]^m\}$$

Where A is a preexponential factor.

Thomas and Jennings, *Chem. Mat.*, 11:1907-14, 1999.

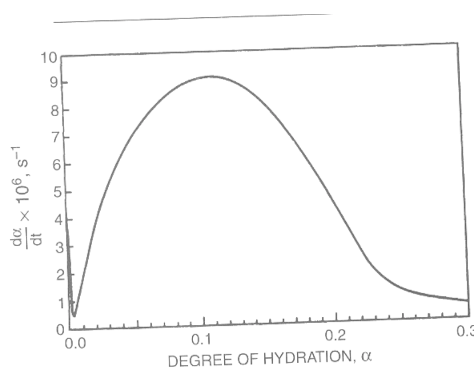


Figure credit: Gartner et al, in Structure and Performance of Cements, Bensted and Barnes (Eds), Spon 2002.

Cement Hydration: Avrami Model

- Can also determine the activation energy (E_a) for the reaction, which can be used to assess the temperature-dependence of the reaction:

$k(T)=A \exp(-E_a/RT)$

Where T is absolute temperature (K), R is gas constant,

Table 5. Avrami Fit Parameters as Reported by FitzGerald et al.[9] for the Hydration Rate of C_3S/H_2O Pastes As Measured by QENS

T (°C)	t_{max}[a]	t_0 (h)	rate parameter (h^{-m})	m	k (h^{-1})[b]
20	10.2	3.5	7.4×10^{-3}	2.15	0.1021
30	5.7	1.5	1.74×10^{-2}	2.27	0.1678
40	3.2	0.5	3.3×10^{-2}	2.59	0.2679

[a] Estimated from Figure 3 of ref 9. [b] k = (rate parameter)$^{1/m}$.

Thomas and Jennings, *Chem. Mat.*, 11:1907-14, 1999.

Cement Hydration: Jander Equation*

- In the deceleration period, the Jander equation for diffusion controlled processes has been used to model the reaction of cement during this period:

$[1-(1-\alpha)^{1/3}]^2 = k_D$

Where k_D is the rate constant for diffusion controlled processes.

* Jelenic, *Adv. Cem. Tech*. Gosh (Ed), p.397, Pergamon, 1987.
Bezjak and Jelenic, *Cem. Conc. Res.*, 10:553, 1980.

Cement Hydration: Simple Kinetic Models

- Can estimate α based upon the available water-filled porosity (ϕ_w):

$$\frac{\partial \alpha}{\partial t} = k_1 \phi_W(t)$$

where k_1 is analogous to a first-order rate constant and depends on the specific cement composition, particle size distribution, curing temperature, etc.

- This approach, based upon first order "physical" kinetics and described by Bentz*, assumes hydration rate is simply proportional to the volume fraction of this water-filled porosity
- Other models** relate the kinetics of hydration to the changing radius of an idealized cement particle or particle distribution

*D. P. Bentz, "Influence of Water-to-Cement Ratio on Hydration Kinetics: Simple Models Based on Spatial Considerations" at http://ciks.cbt.nist.gov/~garbocz/hydration_rates/index.html
**J.M. Pommersheim, J.R. Clifton, Mathematical modeling of tricalcium silicate hydration. Cem Concr Res 9 (1979) 765-770.
T. Knudsen, The dispersion model for hydration of portland cement 1. General concepts, Cem Concr Res 14 (1984) 622-630.
B. Osbaeck, V. Johansen, Particle size distribution and rate of strength development of portland cement. J Am Ceram Soc 72 (2) (1989) 197-201.

Cement Hydration: Simple Kinetic Models

Substituting $\frac{\partial \alpha}{\partial t} = k_1 \phi_W(t)$

into Powers equation for water-filled porosity

$$\phi_W(t) = \frac{\rho_{cem}(w/c) - (f_{exp} + \rho_{cem} CS)\alpha}{1 + \rho_{cem}(w/c)}$$

the result can be integrated and solved with the boundary condition that $\alpha(0)=0$ to yield:

$$\alpha(t) = Min\{1, \frac{\rho_{cem}(w/c)}{(f_{exp} + \rho_{cem} CS)}[1. - \exp(\frac{-(f_{exp} + \rho_{cem} CS)k_1 t}{1 + \rho_{cem}(w/c)})]\}$$

f_{exp} is the volumetric expansion coefficient for the "solid" cement hydration products relative to the cement reacted (often taken to be =1.15),

CS is the chemical shrinkage per gram

(the minimum function assures that $\alpha \leq 1$)

- Although derived from a different perspective, the above is similar in form to kinetics equations often derived considering nucleation and growth kinetics for cement hydration (so-called Avrami behavior)

Heat of Hydration

For the usual portland cement:

- ~ 1/2 total heat is evolved in 1-3 days
- 3/4 at 7 days
- 83-91% at 180 days

Heat of Hydration

The RATE of heat evolution is related to

- cement composition
- cement fineness
- cement content
- casting temperature

The total heat evolved is related to

- cement composition
- degree of hydration
- cement content

Heat of Hydration

TABLE 4.3 Heats of Hydration of the Cement Compounds

Reaction	ΔH (J/g) for Complete Hydration[a]			
	Pure Compounds		Clinker[b,d]	Cement[c,d]
	Calculated	Measured	Measured	Measured
$C_3S \rightarrow C\text{-}S\text{-}H + CH$	~380	520	570	490
$C_2S \rightarrow C\text{-}S\text{-}H + CH$	~170	260	260	225
$C_3A \rightarrow C_4AH_{13} + C_2AH_8$	~1160	–	–	–
$\rightarrow C_3AH_6$	900	880	840	–
→ ettringite	1670	1670	–	–
→ monosulfoaluminate	1150	1140	–	1170
$C_4AF \rightarrow C_3(A,F)H_6$	420	420	335	–
→ monosulfoaluminate	–	–	–	380
→ ettringite	730	–	–	–

[a]These values should be negative since they refer to exothermic reactions, but they are customarily written without the negative sign.
[b]One-year-old pastes of ground clinker (no added gypsum).
[c]One-year-old pastes assumed to be completely hydrated.
[d]Individual contributions determined by multiple linear regression analysis.

Heat of Hydration: Influence of Cement Composition

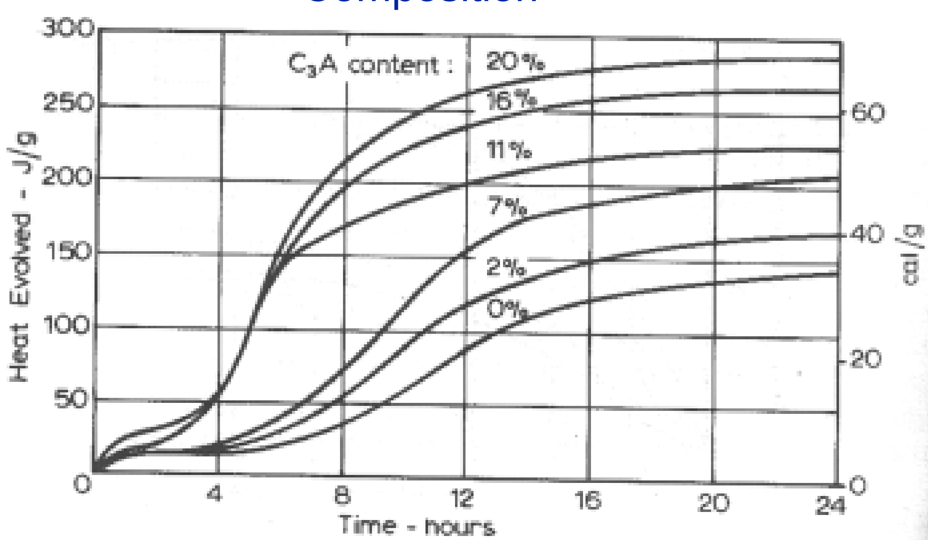
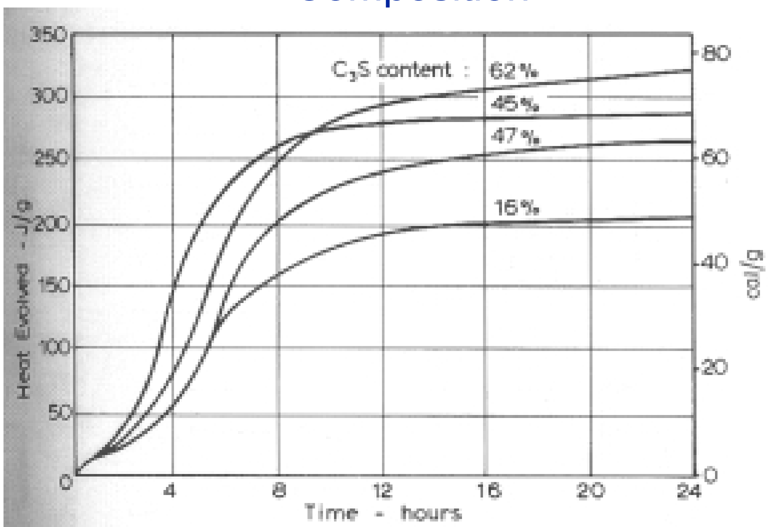


Fig. 1.15 Influence of C_3A content on heat evolution[1.32] (C_3S content approximately consta

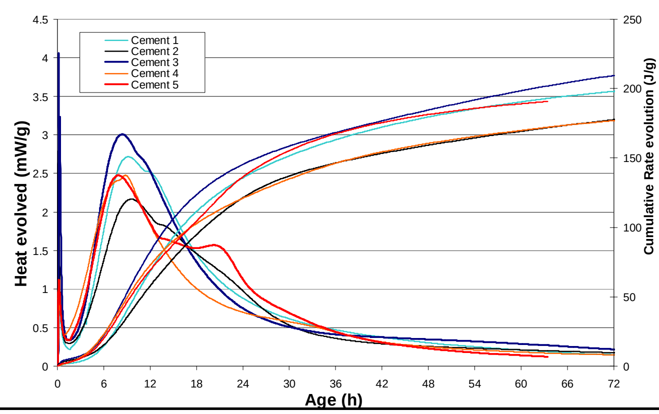
Heat of Hydration: Influence of Cement Composition



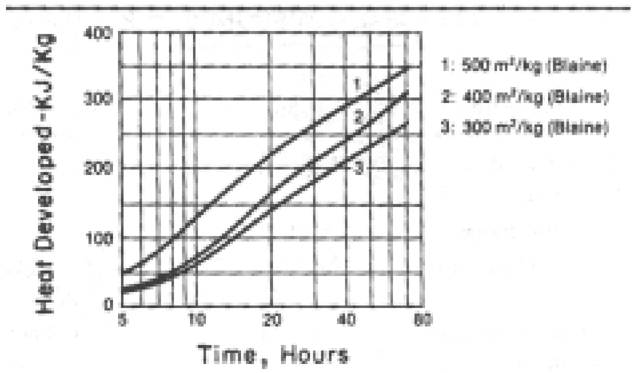
1.16 Influence of C_3S content on heat evolution[1.32] (C_3A content approximately constant)

Heat of Hydration: Influence of Cement Composition

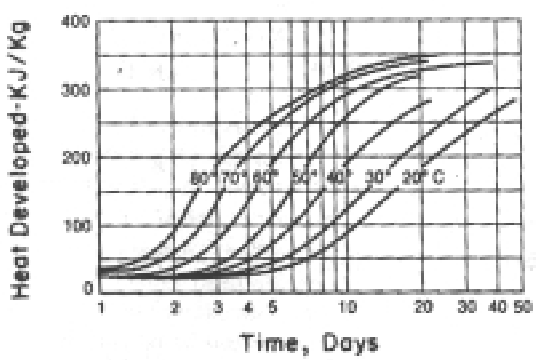
	Cement 1	Cement 2	Cement 3	Cement 4	Cement 5
C3S	59	55.9	62.9	50.4	42
C2S	16	19	7.9	20.6	26
C3A	4	7.5	10.1	7.1	9
C4AF	11	9.1	8.6	12.2	10



Heat of Hydration: Influence of Cement Fineness



Heat of Hydration: Influence of Ambient Temperature



Estimating Heat of Hydration

Verbeck and Foster estimated that the overall heat of hydration of a cement is near the sum of the heats of hydration of the individual components.

$$H = aA + bB + cC + dD$$

A,B,C,D are % by wt of C3S, C2S, C3A, C4AF

a,b,c,d are coefficients representing the contribution of 1% of the corresponding compound to the heat of hydration

H_{3days}= 240(C3S) + 50(C2S) + 880(C3A) + 290(C4AF) J/g

H_{1yr}= 490(C3S) + 225(C2S) + 1160(C3A) + 375(C4AF) J/g