Thermogravimetric Analysis of Silicate Cement Component Content

1. Background

Cement is one of the most widely used construction materials across industries including infrastructure, transportation, agriculture, and marine engineering. Among cement types, silicate cement holds a dominant role due to its versatility, durability, and long-standing industrial adoption.(1)

The primary phase of silicate cement clinker comprises silicate minerals, with alite (C_3S) and belite (C_2S) accounting for over 75% of its composition. Upon exposure to atmospheric conditions, these components react with moisture and carbon dioxide to form secondary products such as:(1,2)

- ✓ Ettringite
- ✓ C-S-H gel
- ✓ Calcium hydroxide ($Ca(OH)_2$)
- ✓ Calcium carbonate ($CaCO_3$)

Each of these phases decomposes at distinct temperatures, making **thermogravimetric analysis (TGA)** a powerful technique to identify and quantify cement hydration and carbonation products based on characteristic mass loss behavior.(3)

2. Experimental Methods

Thermogravimetric analysis was performed using the **AMI TGA 1000** under the following conditions:

- ✓ Sample mass: ~20 mg
- ✓ Sample holder: Pt crucible
- ✓ Atmosphere: Nitrogen (50 mL/min)
- ✓ Heating program: 30 °C to 1000 °C at 10 °C/min

The analysis focused on the thermal decomposition behaviors of $Ca(OH)_2$ and $CaCO_3$, allowing their relative quantities to be determined based on water and CO_2 release, respectively.

3. Results and Discussion

Figures 1 and 2 present the TGA profiles for two silicate cement samples (Sample A and Sample B). The thermograms can be interpreted in four distinct mass loss stages, and the detailed results are reported in Table 1.

Stage 1: < 200 °C

✓ Evaporation of free moisture and decomposition of low-temperature hydration products such as C-S-H gel and ettringite.

Stage 2: 400–480 °C

✓ Dehydration of calcium hydroxide ($Ca(OH)_2$).

Stage 3: 500–800 °C

✓ Decomposition of poorly crystalline $CaCO_3$, which typically forms via environmental carbonation during curing.

Stage 4: > 800 °C

✓ Breakdown of highly crystalline $CaCO_3$, typically from the original clinker phase or long-term carbonation.

Both samples exhibited comparable amounts (1.073% and 1.168%) of highly crystalline $CaCO_3$ which decomposed at temperatures above 800 °C. Sample B shows a higher percentage of $Ca(OH)_2$ decomposition (2.422% vs 0.980%), indicating a more advanced hydration stage or higher water-to-cement ratio. Sample A shows a greater amount of poorly crystalline $CaCO_3$ (12.698% vs 4.767%), suggesting more surface carbonation or environmental exposure during curing.

These results demonstrate that the **AMI TGA 1000** provides excellent resolution for differentiating hydration and carbonation products in cement and allows for reliable quantitative analysis based on thermal decomposition behavior.

Mass Loss Stage	**Sample A**			**Sample B**		
	Mass loss	**Temperature**	**Residue**	**Mass loss**	**Temperature**	**Residue**
Stage 1 (< 200 °C)	5.253%	200.0 °C	77.13%	5.562%	200.0 °C	82.93%
Stage 2 (400-480 °C)	0.980%	476.4 °C		2.422%	437.00 °C	
Stage 3 (500-800 °C)	12.698%	761.0 °C		4.767%	587.14 °C, 791.00 °C	
Stage 4 (> 800 °C)	1.073%	> 800 °C		1.168%	> 800 °C	

Table 1: Results from TGA of Silicate Cement Samples A and B

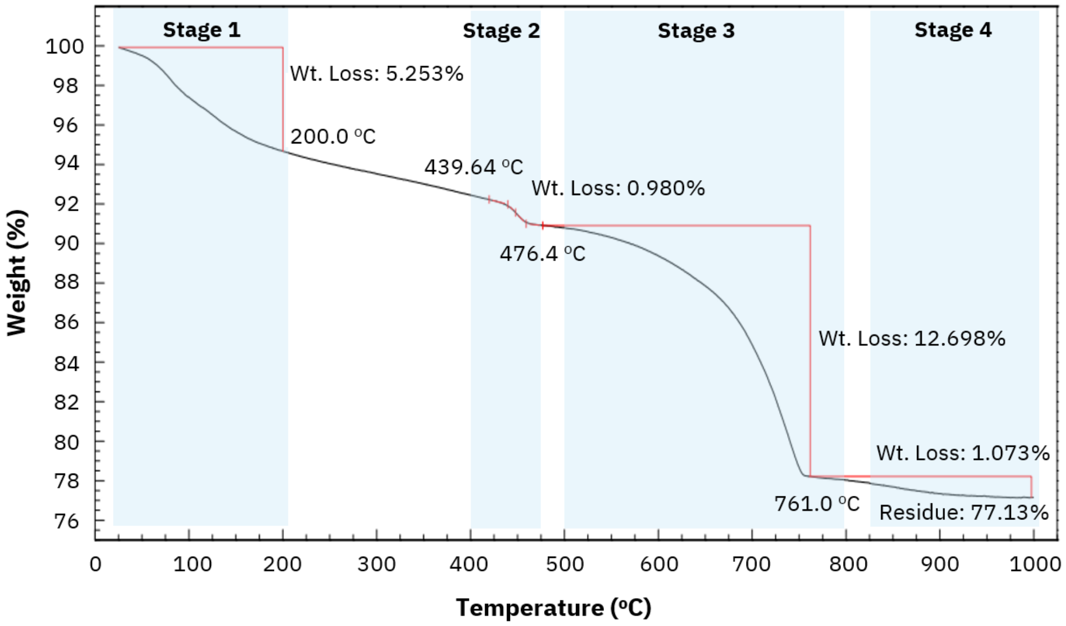

Figure 1: TGA of Silicate Cement A

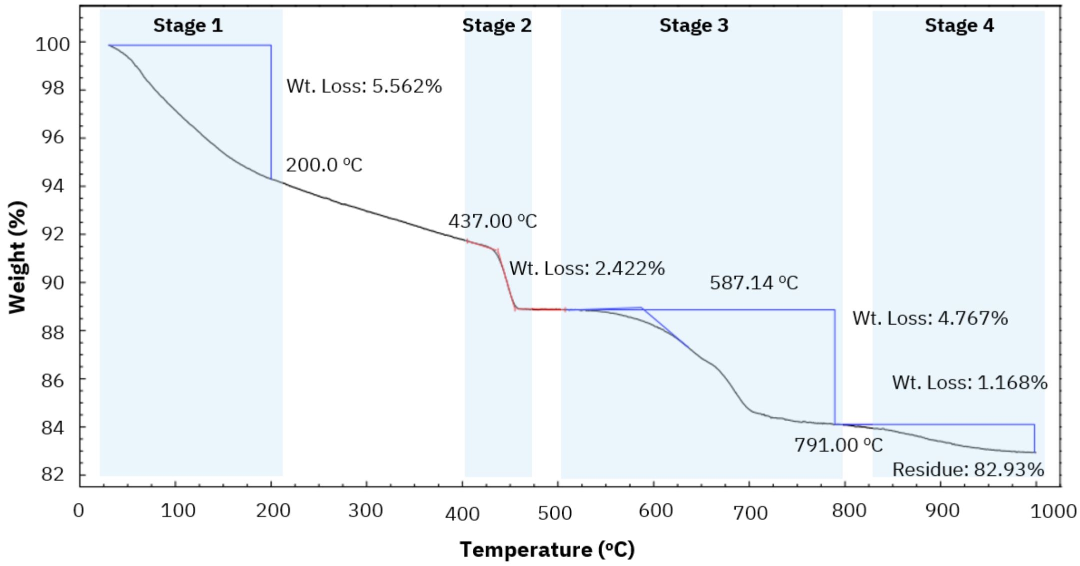

Figure 2: TGA of Silicate Cement B

4. Conclusions

Thermogravimetric analysis using the **AMI TGA 1000**, shown in Figure 3, enables clear and reliable identification of key hydration and carbonation phases in silicate cement. The system's high sensitivity and stability allow differentiation between loosely and strongly bound components across a broad temperature range — from ettringite and $Ca(OH)_2$ to amorphous and crystalline $CaCO_3$.

The relative amounts of $Ca(OH)_2$ and $CaCO_3$ reflect hydration progress and carbonation extent, both of which impact durability and performance in real-world environments.

AMI TGA 1000

- ✓ Research-grade capabilities at an accessible price point
- ✓ High sensitivity microbalance
- ✓ Resolution down to 0.1 µg
- ✓ Compact heating furnace
- ✓ Precise temperature control

Figure 3: Overview of AMI TGA 1000

5. References

(1) Lavagna, L.; Nisticò, R. An insight into the chemistry of cement – A review. *Appl. Sci.* **2023**, *13*, 203.

(2) Choudhary, H. K.; A. V., A.; Kumar, R.; Panzi, M. E.; Matteppanavar, S.; Sherikar, B. N.; Sahoo, B. Observation of phase transformations in cement during hydration. *Constr. Build. Mater.* **2015**, *101*, 122-129.

(3) Alarcon-Ruiz, L.; Platret, G.; Massieu, E.; Ehrlacher, A. The use of thermal analysis in assessing the effect of temperature on a cement paste. *Cem. Concr. Res.* **2005**, *35*, 609-613.