Supporting Production Consistency for Cement

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NIST ENGINEERING LABORATORY

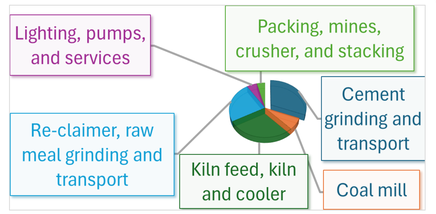
Introduction

- The NIST Standard Reference Material (SRM) program provides well-characterized materials ranging from peanut butter to cement precursors. The SRMs are used to calibrate instruments and to verify the accuracy of measurements.
- The measurements are reflected in values that are traceable back to the International System of Units (SI).
- SRMs are assigned certified values with associated uncertainties and are checked for long-term stability while being available for purchase.
- SRMs are used across a wide variety of industries as precise, accurate measurements and are essential for many aspects of commerce.

"Truth in a Bottle"
NIST SRMs

Cement Production

- Cement production has evolved over millennia; modern-day cement (Portland cement) has been consistent since its creation in the mid 19th Century, with some modifications to components and machinery.
- Producing cement requires calcining natural components, primarily limestone and clays, that result in clinker with a typical size of 25 mm.
- The clinker is ground to reduce the particle size to 3 micrometers to 30 micrometers, enhancing hydration when used to produce concrete.
- Several steps, such as sintering and grinding, are energy-intensive and will continue to be optimized for lower energy. Control of cement fineness is essential.

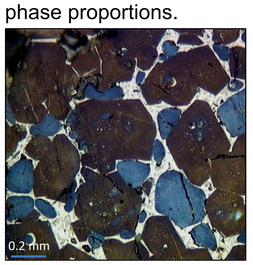


Percentage of electrical consumption for stages of the cement production process[1]

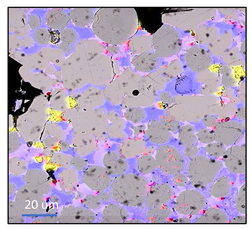
Cement Clinker – SRM 2687a, 2686b, 2688

- NIST provides SRMs for Portland cement clinker with grain size, particle distribution, and composition representing the range of North American clinker production.
- Clinker is composed of multiple distinct phases but is dominated by alite, belite, aluminate, and ferrite. Accessory phases, such as periclase, can enhance reactivity, performance, and/or reduce the energy required for grinding.
- Microscopy, chemical analysis, and x-ray diffraction enable characterization of clinker to establish ideal phase proportions.

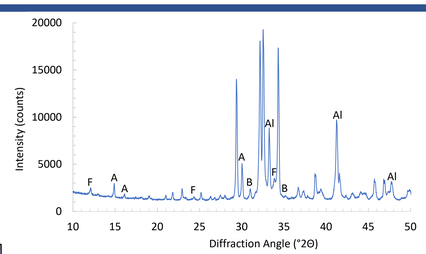
Stereomicroscope image of 2687a



Light microscope image of 2687a



SEM backscattered electron image Mg =red, Al=blue, and S=yellow highlighting interstitial phases



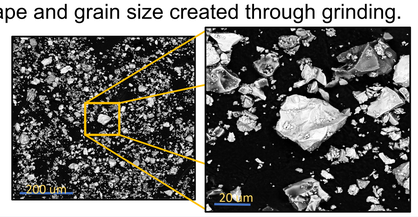
X-ray powder diffraction pattern with characteristic peaks; A=Alite, B=Belite, Al=Aluminate, F=Ferrite

Phase	Mean	St. Dev	1	2	3
Alite	57.2	0.2	60.8	55.3	55.6
Belite	28.0	0.2	24.4	29.9	29.7
Aluminate	6.7	0.2	7.2	6.54	6.46
Ferrite	6.5	0.2	6.2	6.73	6.71
Periclase	0.4	0.0	0.40	0.44	0.43
Arcanite	0.5	0.0	0.53	0.49	0.49
Aphthitalite	0.4	0.0	0.29	0.40	0.38
Free Lime	0.2	0.0	0.20	0.18	0.21

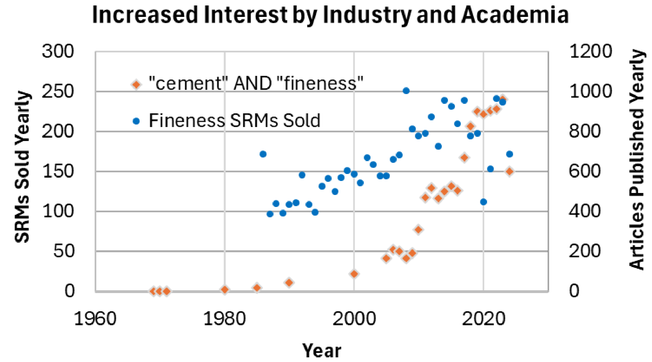
Rietveld refinement of whole clinker XRD patterns

Cement Fineness – SRM 114r

- The use of SRM 114r continues to be applicable for new cement requirements and supplemental cementitious material, e.g. fly ash, slag, silica fume, and other natural pozzolans as the industry continues to grow.
- The Blaine Permeability test, using SRM 114r as a comparator, captures the particle size distribution and shape effect with air passing through a powder compact.
- Electron microscopy images illustrate the variable shape and grain size created through grinding.



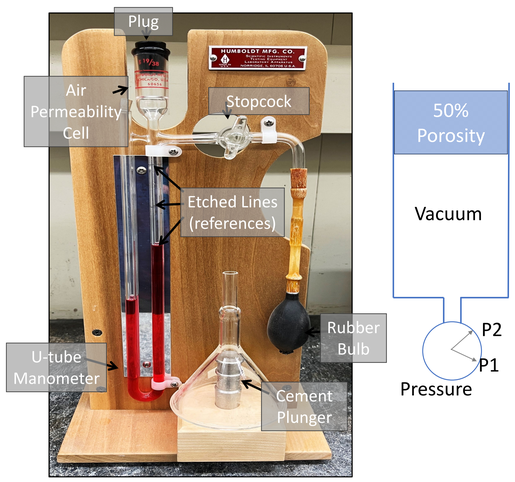
Backscattered electron microscopy images (SRM 114r)



- The yearly sales number for the cement fineness SRM 114r track with the increasing number of Journal articles that contain the keywords of "cement" and "fineness."

Blaine Air-Permeability Apparatus

- Blaine Air-Permeability Apparatus determines the fineness of cements, in terms of specific surface area.
- This test was initially developed in 1943 and has survived the test of time.[2]



Blaine Air-Permeability Apparatus with integral parts identified

Δt_s => P1 to P2

$$S = \frac{S_s\sqrt{t}}{\sqrt{t_s}}$$

S = surface area, m^2/kg
t = time, s

- Simplified analysis by comparing to a reference material.

Note:
- Subscript *s* is reference sample
- Alternative equations with more variables, ASTM C204-24

Test Methods

- Multiple methods can be used to evaluate cement fineness.
- Several are standardized and used within industry to verify the accuracy of their measurements.
- Datasets from the tests vary, relaying different properties of cement with their results.
- Evident by the table, no method can completely characterize cement fineness, thus considerations are made to adequately assess cement.
- Considerations when choosing a suitable test include cost, required materials, complexity, and data produced.

Test Method	Apparatus	Data	Pros	Cons
ASTM C204[3]	Blaine Air-Permeability Apparatus	Blaine fineness (m^2/kg)	Low cost, Simple methodology	Manually performed, Does not evaluate particle size
ASTM C430[4]	45 micrometer sieve	Mass retained (%)	Low cost	Does not evaluate surface area, particle size
ASTM C1891[5]	45 micrometer sieve, air jet	Mass retained (%)	Low cost	Does not evaluate surface area, particle size
AASHTO T 353[6]	Laser diffractometer	Particle size (nm-mm)	Measurement range, rapid analysis, High reproducibility, Analyze both wet and dry samples	Does not evaluate surface area, particle shape, agglomeration of particles
Nitrogen Brunauer-Emmett-Teller (BET)	Surface adsorption / pore size analyzer	m^2/g	High precision, characterization of fineness and microstructure	Complex, time-intensive, variability

Common test methods that evaluate cement fineness; does not represent all standards that are currently used

Summary

- Concrete is the second most used substance in the world after water and requires cement to achieve its load-bearing properties.
- Manufacturers produce 14 billion cubic meters of concrete annually, with a market size of $440 billion. The concrete industry is developing technologies to be more competitive through novel materials, advanced manufacturing methods, and improved measurement practices.
- Accurate measurement techniques are needed to produce quality concrete by measuring the particle size, fineness, and surface area of cement and its companion components.
- Over the decades, alternative measurement techniques have been developed but can encounter challenges.
- Manufacturers and builders worldwide need to be sure that their concrete will perform as expected and that the finished product will have the desired properties. This ensures faster construction and safer, more reliable structures.
- Innovations in test methods and cement production are needed to address future cement formulations and to reduce energy requirements.

Needs / Innovation

- Innovations are needed in both the production of cement to reduce energy requirements and in standardized measurement practices for more predictable strength development of this ubiquitous global commodity and infrastructure material.
- With innovations in cement design, including new binders and supplemental cementitious materials, fineness continues to be an important engineering parameter for optimizing performance.
- New validation test methods will be beneficial to address these increased demands on fineness. These methods complement the simple and rapid air fineness test using the Blaine Air-Permeability apparatus for manufacturing quality control.
- For example, dynamic image analysis (DIA) is a particle characterization method for determining size distributions and shape parameters.

References

[1] Niranjan, S. et al., "Review on energy conservation and emission reduction approaches for cement industry," Environmental Development, 44, 2022, 100767.

[2] Blaine, R. L. "A simplified air permeability fineness apparatus." ASTM Bull 123 (1943): 51-55.

[3] ASTM C204-24, Standard Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus, ASTM International, West Conshohocken, PA, 2024, www.astm.org.

[4] ASTM C430-18, Standard Test Method for Fineness of Hydraulic Cement by the 45-µm (No. 325) Sieve, ASTM International, West Conshohocken, PA, 2018, www.astm.org.

[5] ASTM C1891-21, Standard Specification for High-Strength Concrete Mixes, ASTM International, West Conshohocken, PA, 2021, www.astm.org.

[6] AASHTO T 353 : 2014(R2018), Method of Test for Particle Size Analysis of Hydraulic Cement and Related Materials by Light Scattering, Washington, DC, 2018.