

C. Zoude¹, S. Abdelouhab¹ *Belgian Ceramic Research Center, Mons, Belgium*

Introduction: Shaping complex refractory formworks

Conventional method (non-permanent wooden/metal formwork):

- ✓ Well-established and well-controlled process
- ✓ High mold robustness
- ✓ Suitable for large-scale production
- ✗ Time-consuming and costly mold manufacturing and assembly
- ✗ Limited geometric flexibility
- ✗ Difficult demolding

VS

3D printing (permanent):

- ✓ High geometric freedom
- ✓ Fast manufacturing
- ✓ No demolding required
- ✗ **Need to develop a printable ink with properties suitable for the intended application**

Goal

Develop simple methods/tests to guide the formulation and characterization of refractory inks. These inks must exhibit:

- ✓ Good printability
- ✓ A low or no cement content to reduce the carbon footprint
- ✓ Good thermomechanical properties at high temperature
- ✓ High resistance to harsh environments

1 - Testing methods

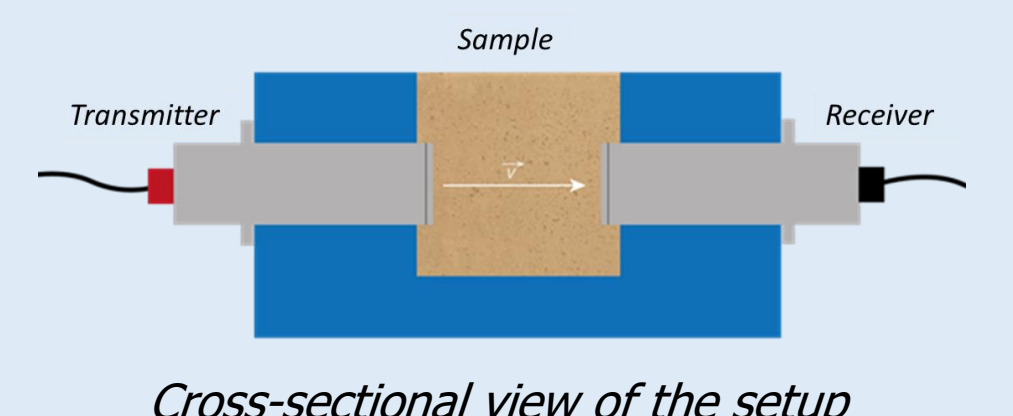
Rheological testing



Thermo Scientific HAAKE MARS III
Vane geometry with internal lamella to prevent material slippage
Four-blade rotor

Ultrasonic setting monitoring IP-8 (UltraTest)

Monitoring of P-wave propagation time through the material over time



Flow table tests for spread measurement EN 105-3

After 15 drops



Mechanical testing

Zwick/Roell Z100
Compression and flexural tests
Currently ongoing

2 - Development of refractory inks

Simplified formulation of the inks

Coarse tabular alumina aggregates 1-3 mm
Fine tabular alumina aggregates <1 mm
Binder
Additives
Mixing water

Ink 1 LCC - Low cement content

Binder: Cement 5 wt% (CAC)
Additives: Dispersant FS60 and FS10 (BASF) to improve early strength while enabling retardation and water reduction

Ink 2 NCC - No cement content

Binder: 0,25 wt% CAC and 3 wt% SiOX Zero (microsilica)

Particle size distribution optimization

Andreasen model:

$$CPFT = \left(\frac{D}{D_{max}}\right)^q$$

- CPFT: cumulative passing volume
- D: particle diameter
- D_{max}: maximum particle diameter

with q=0,26
pseudoplastic behavior, suitable for pumping

Reference: Commercial non-refractory cementitious ink for 3D printing
CAC > 20 wt%
(Albert - Construction3D)

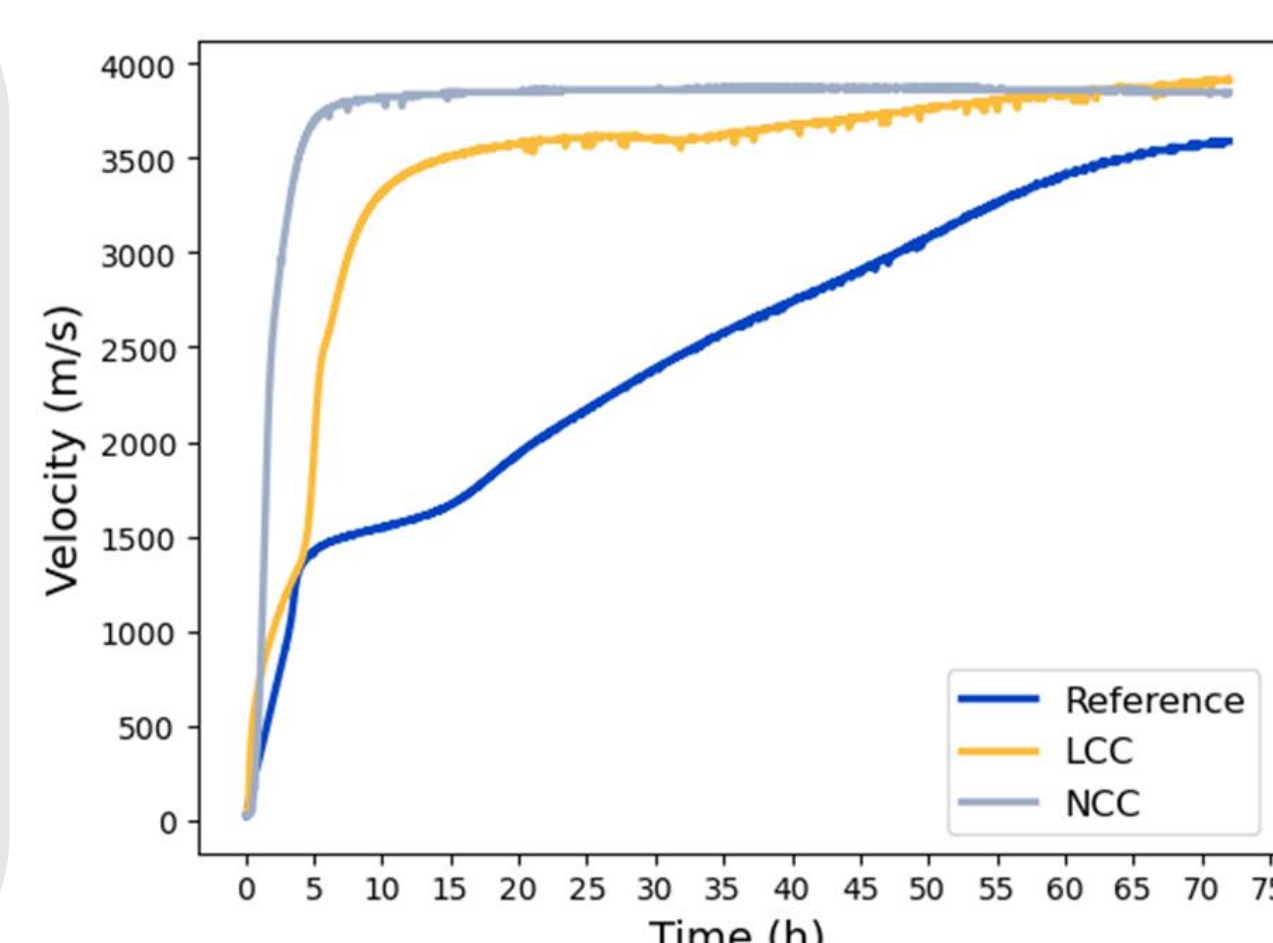
4 - Ultrasonic setting monitoring

LCC: 3 distinct stages

- First stage: Initial increase in ultrasonic velocity, without mechanical strength development. Early stiffening attributed to increased viscosity and Ca²⁺-PCE interactions, promoting early flocculation.
- Second stage: Further increase in ultrasonic velocity associated with cement hydration, network development, and mechanical strength development.
- Third stage: Progressive hardening of the material.

NCC: 2 distinct stages

- First stage: Rapid increase in ultrasonic velocity, while mechanical strength develops only after ~18 h. Progressive network structuring through Si-OH polycondensation into Si-O-Si bonds. This polymerization-driven structuring explains the absence of a secondary acoustic transition.
- Second stage: Progressive hardening of the material.



3 - Manual extrusion test and spread measurement

Reference	Lower limit	Ideal	Upper limit
Water content (%)	16 %	17,5%	18%
Manual extrusion			
Spread diameter (mm)	134,6 ± 1,1	161,2 ± 1,2	159,2 ± 0,29

LCC	Lower limit	Ideal	Upper limit
Water content (%)	7%	7,25%	7,5%
Manual extrusion			
Spread diameter (mm)	144,9 ± 0,5	152,4 ± 1,1	175,0 ± 1,8

NCC	Lower limit	Ideal	Upper limit
Water content (%)	4,75%	5%	5,25%
Manual extrusion			
Spread diameter (mm)	113,9 ± 0,8	132,3 ± 0,9	140,4 ± 2,1

5 - Rheological testing

- ✗ Rheometer torque limit reached
- ✗ Difficult and time-consuming cleaning
- ✗ Not practical for routine characterization

Alternative: Controlled penetration tests to assess the evolution of material strength over time

- Controlled-speed probe penetration tests
- Measurement of penetration force as a function of depth and time

Conclusion and Perspectives

- ✓ Validation of the Andreasen model for the development of printable refractory inks
- ✓ Validation of the use of microsilica as a cement replacement for the inks
- ✓ The manual extrusion test is a useful tool for adjusting the water content of the mixtures before large-scale printing.
- Spread tests alone are not sufficient to characterize the printability of the refractory inks
- Rheological characterization is difficult to implement

Future work

Complete the large-scale 3D printing trials and compare the mechanical properties of cast and 3D-printed specimens.
Replace commercial tabular alumina with recycled aggregates and investigate their effect on the properties and printability of the refractory inks.
Cast refractory concrete into the 3D-printed components and evaluate the mechanical properties of the combined formwork-concrete system.