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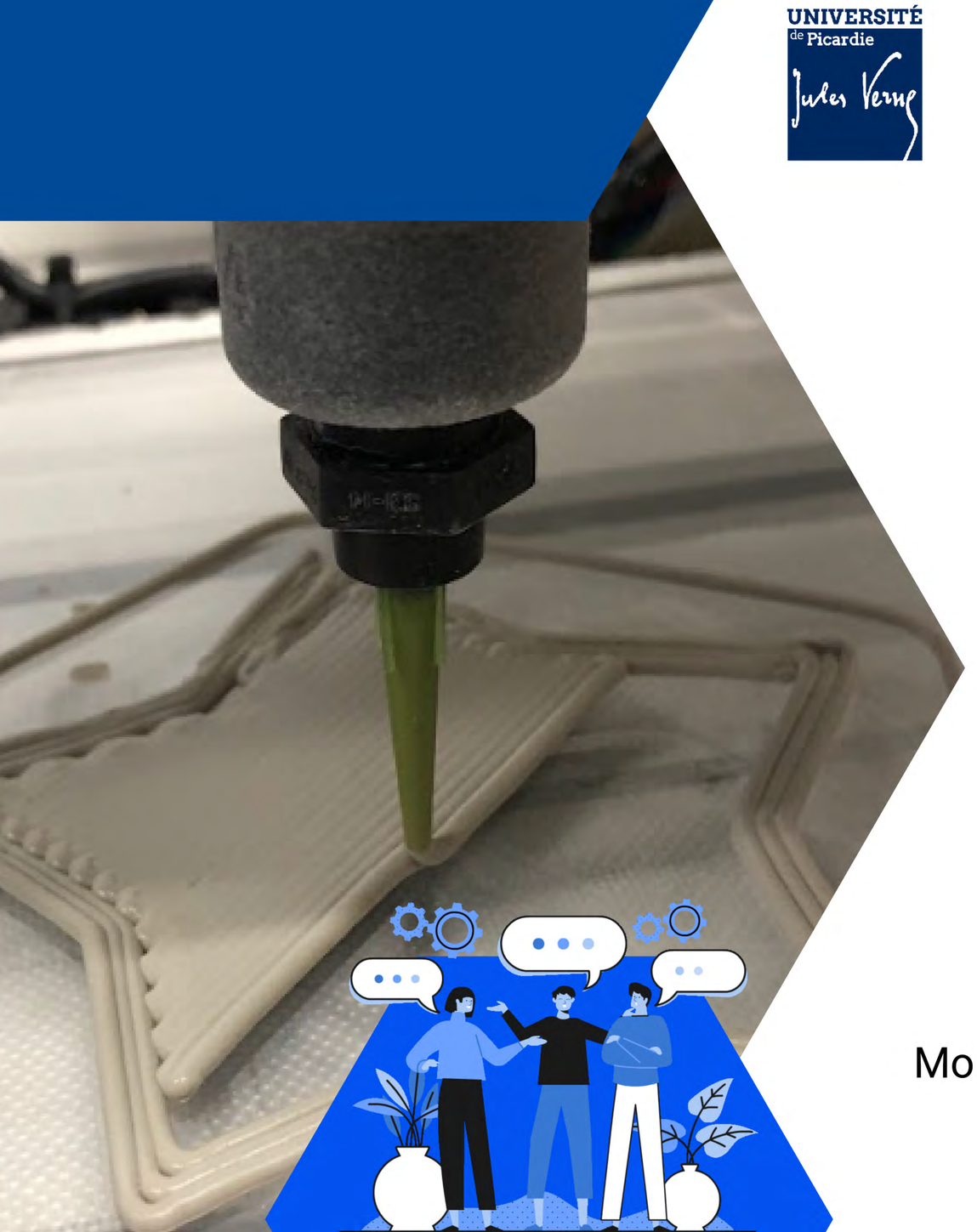
GEOPOLYMER INSTITUTE



GEOPOLYMERCAMP

11 July 2023- Saint Quentin

ADDITIVE MANUFACTURING OF CERAMIC TYPE GEOPOLYMER FOR COMPLEX AND TINY PARTS

Abrar Gasmi [1]

Mohamed Guessasma [1], Christine Pélegris [1], Ralph Davidovits [1 2]

[1] Laboratoire des Technologies Innovantes - UR UPJV 3899

[2] Institut Géopolymère

OVERVIEW

- CONTEXT & CHALLENGES
- ROBOCASTING PROCESS DEVELOPMENT
- RHEOLOGICAL CHARACTERIZATION
- PRINTABLE GEOPOLYMER FORMULA
- OUTLOOK

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CONTEXT & CHALLENGES

PROBLEMATIC ?

Manufacturing process ?

Eco-responsible production?

Robocasting?

Ecological material?

Geopolymer?

CHALLENGES ?

EXTRUDABILITY

1

LAYER SLUMP

2

DIMENSIONAL STABILITY

3

SHRINKAGE

4

GOALS ?

PHASE 1

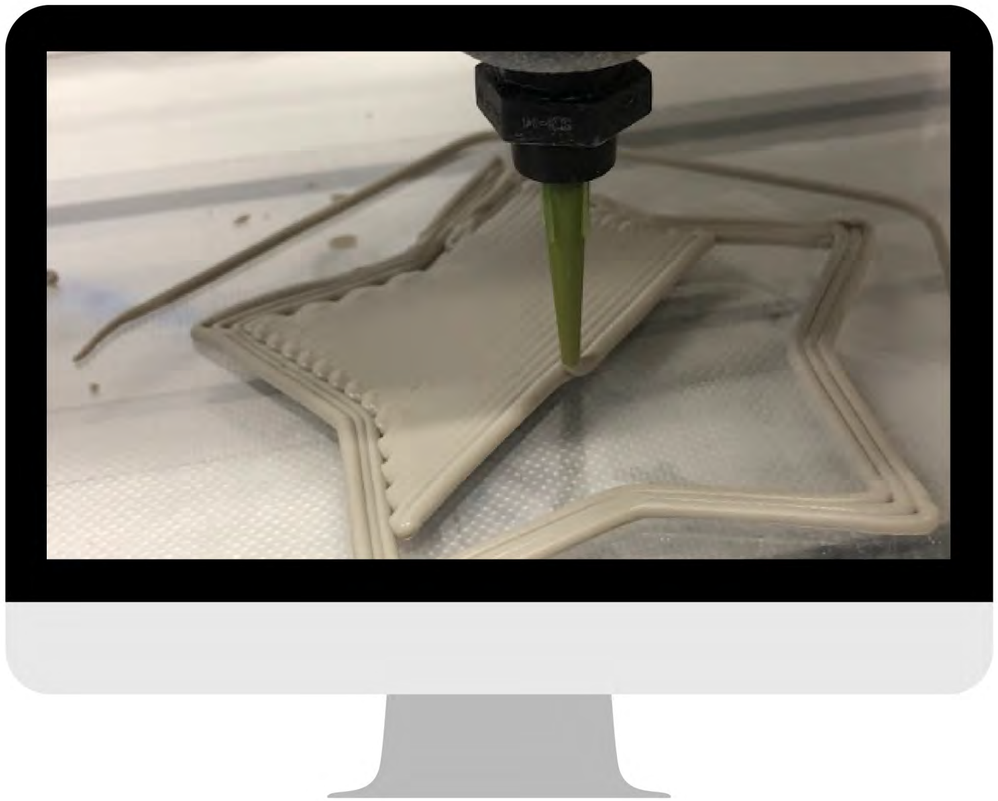
Extruding geopolymer formulation with specific mechanical and physical properties ?

PHASE 2

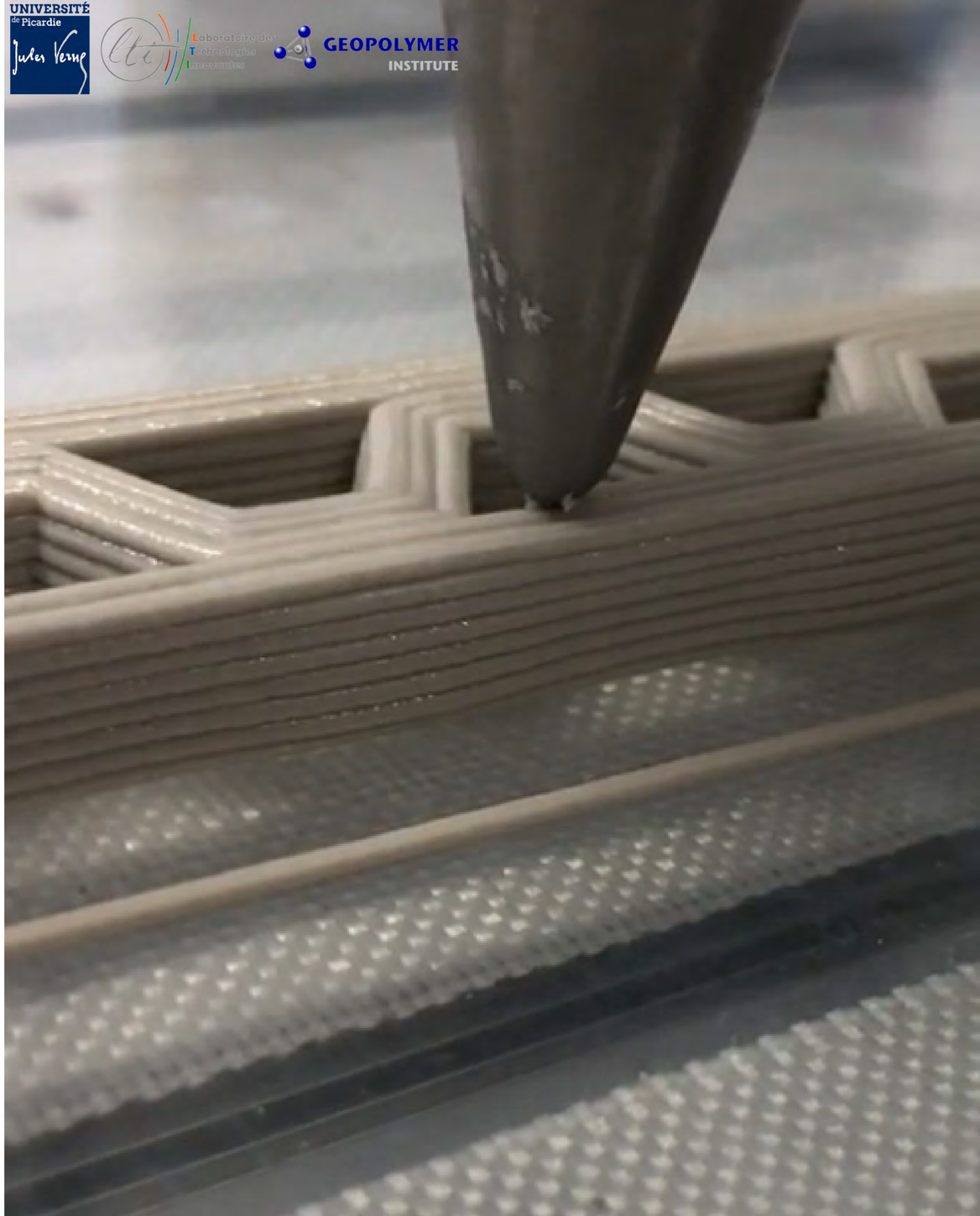
Additive manufacturing process depending on the rheology ?

PHASE 3

Printing small and complex geopolymer prototypes ?



ROBOCASTING PROCESS DEVELOPMENT



PRINTING ANOMALIES ?

PASTE PROPERTIES

- Particle Size,
- Surface Tension,
- Viscosity,
- Storage Modulus,
- Thixotropy,
- Yield Stress…

PRINTING PARAMETERS

- Nozzle
- Diameter,
- Layer Hight,
- Barrel Pressure,
- Printing Speed,
- CAD,
- Slicer…

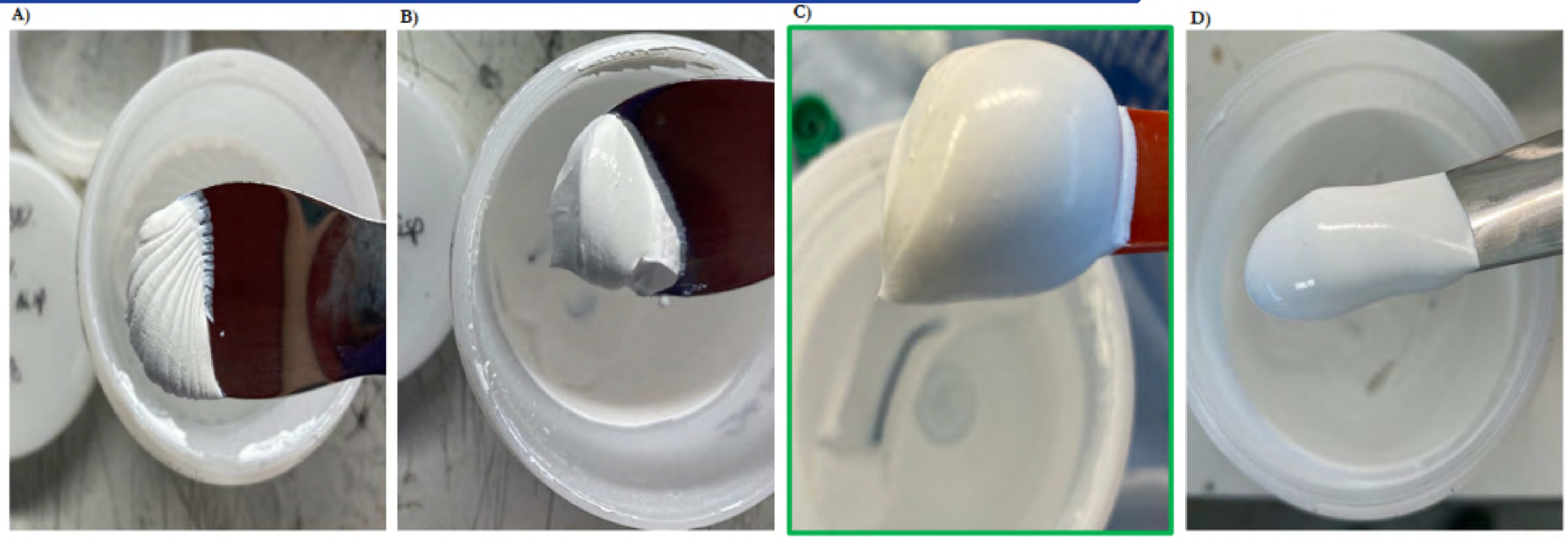
PRINTING ENVIRONMENT

-Temperature,
-Humidity,
-Air or oil bath,
- Bed Temperature…

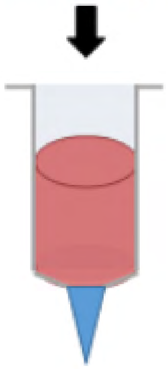
POST-PROCESSING

-Setting Time,
- Drying Temperature,
- Humidity,
- Debinding Schedule,
- Sintering Schedule…

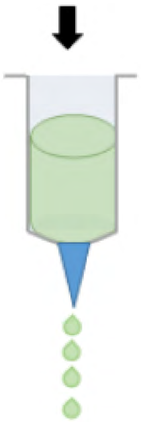
PRINTING ANOMALIES ?



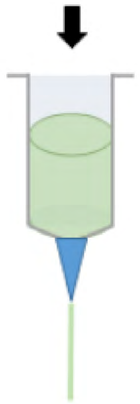
A) Paste too viscous and inhomogeneous. [1]



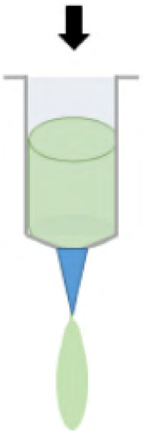
B) Paste too viscous



C) Printable paste



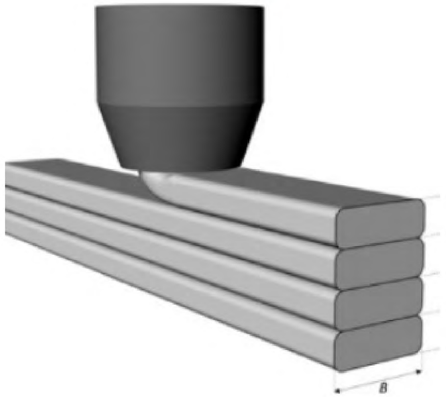
D) Paste too liquid



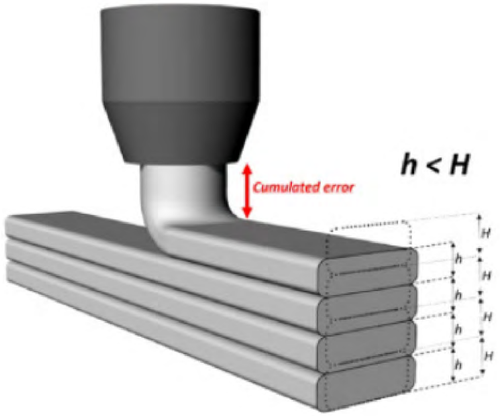
[1] :Maillard, Mathilde. Imprimabilité de pâtes céramiques par robocasting: Applications aux matériaux denses et multimatériaux. Diss. Université de Lyon, 2022.

PRINTING ANOMALIES ?

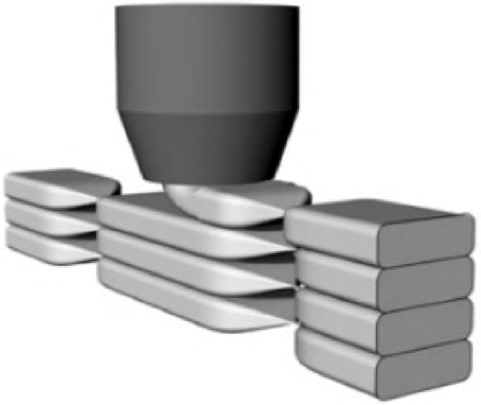
Out of control pressing of the layers



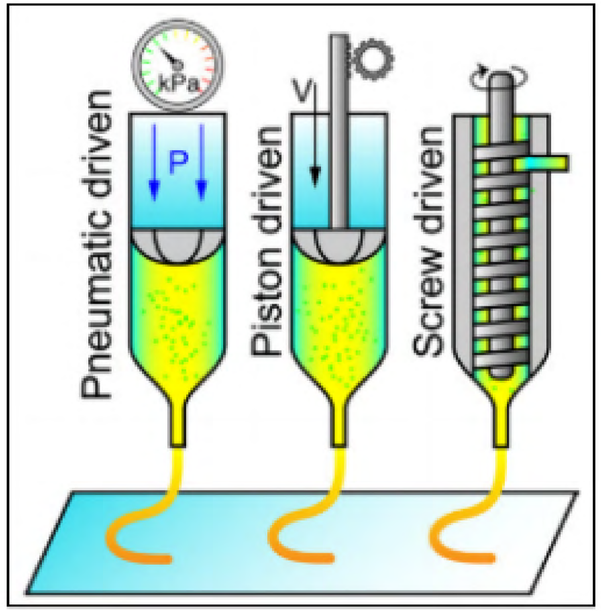
Layer flow out leading to coiling phenomenon

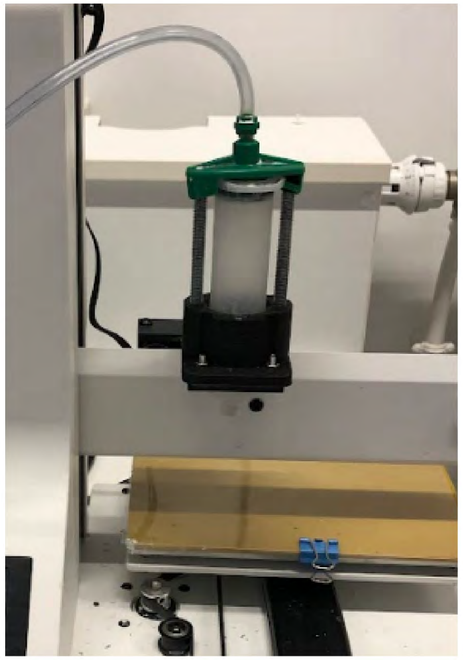


Longitudinal tearing of the layers



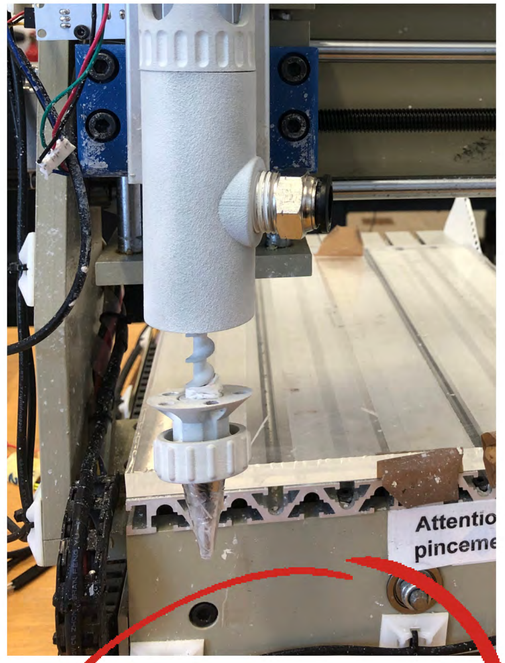
PRINTING ANOMALIES ?





Pneumatic Driven

Piston Driven

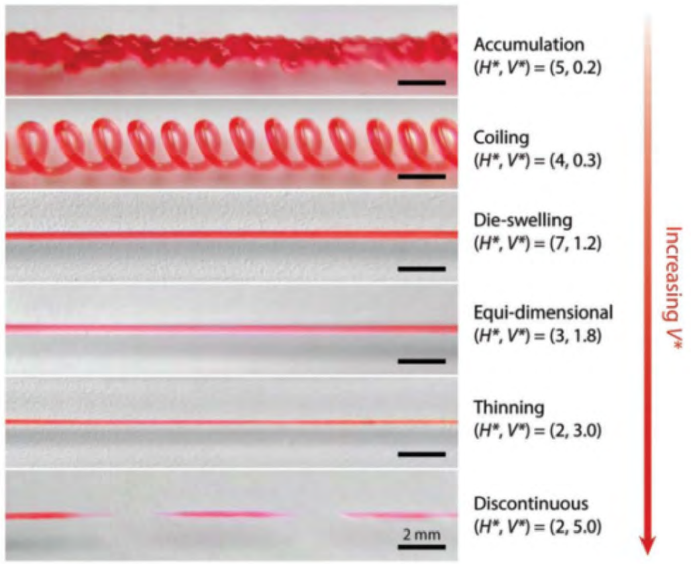


Screw Driven

Time-varying rheological properties

PRINTING ANOMALIES ?

Variation of printing parameters

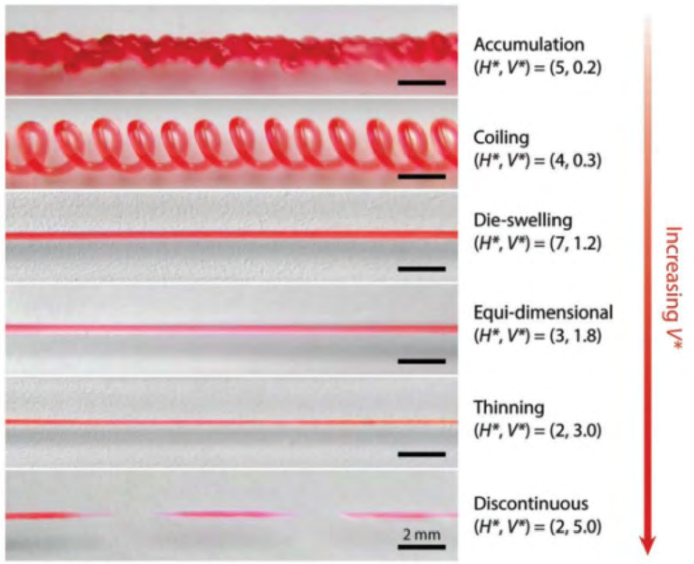


Variation of printing parameters: syringe speed V and the height between the nozzle and the platen H [2]

[2] :Maillard, Mathilde. Imprimabilité de pâtes céramiques par robocasting: Applications aux matériaux denses et multimatériaux. Diss. Université de Lyon, 2022.

PRINTING ANOMALIES ?

Variation of printing parameters



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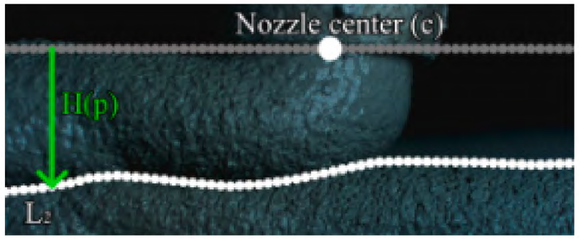
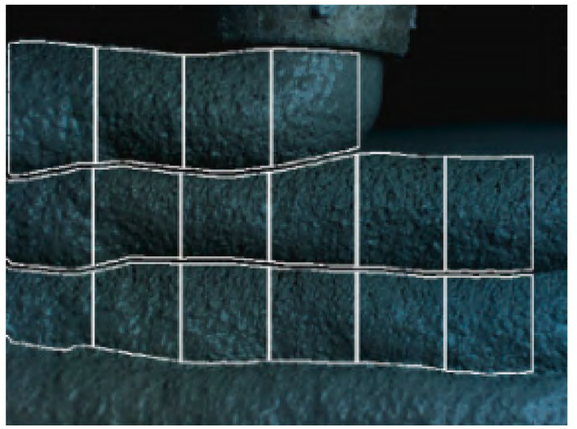
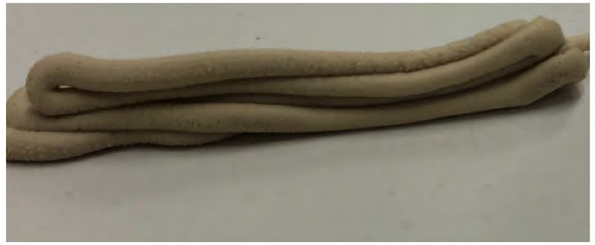
Interlayer characterization anomaly





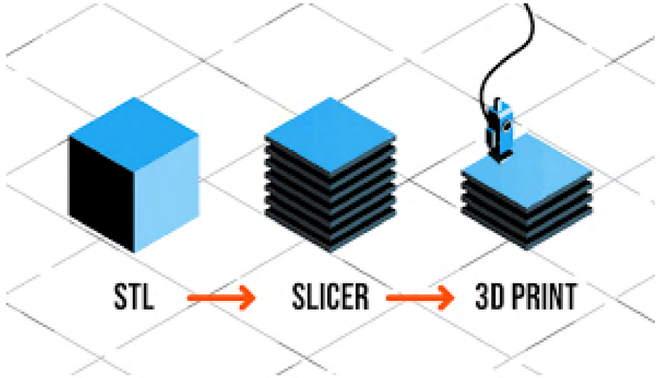


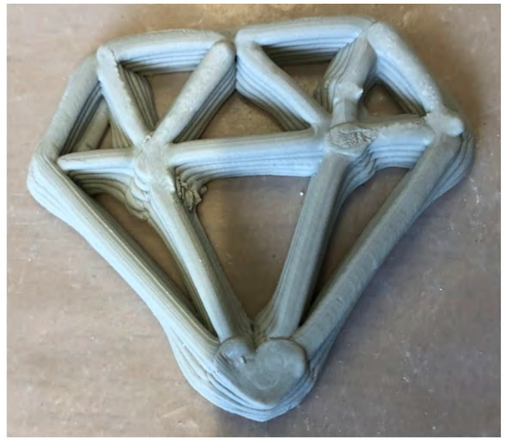
Relative nozzle height measurement [3]

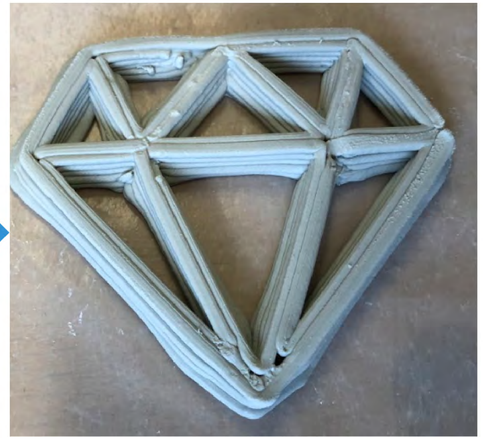
[3] :Ranjbar, Navid, et al. "Rheological characterization of 3D printable geopolymers." Cement and Concrete Research 147 (2021): 106498.

PRINTING ANOMALIES ?

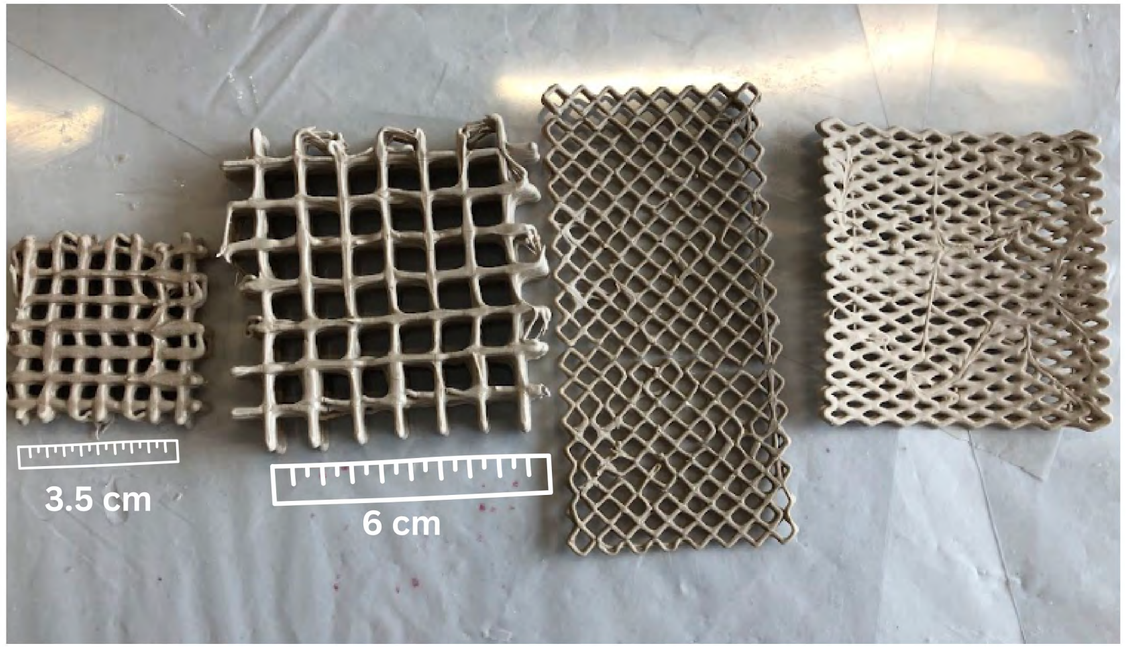
Path Optimization



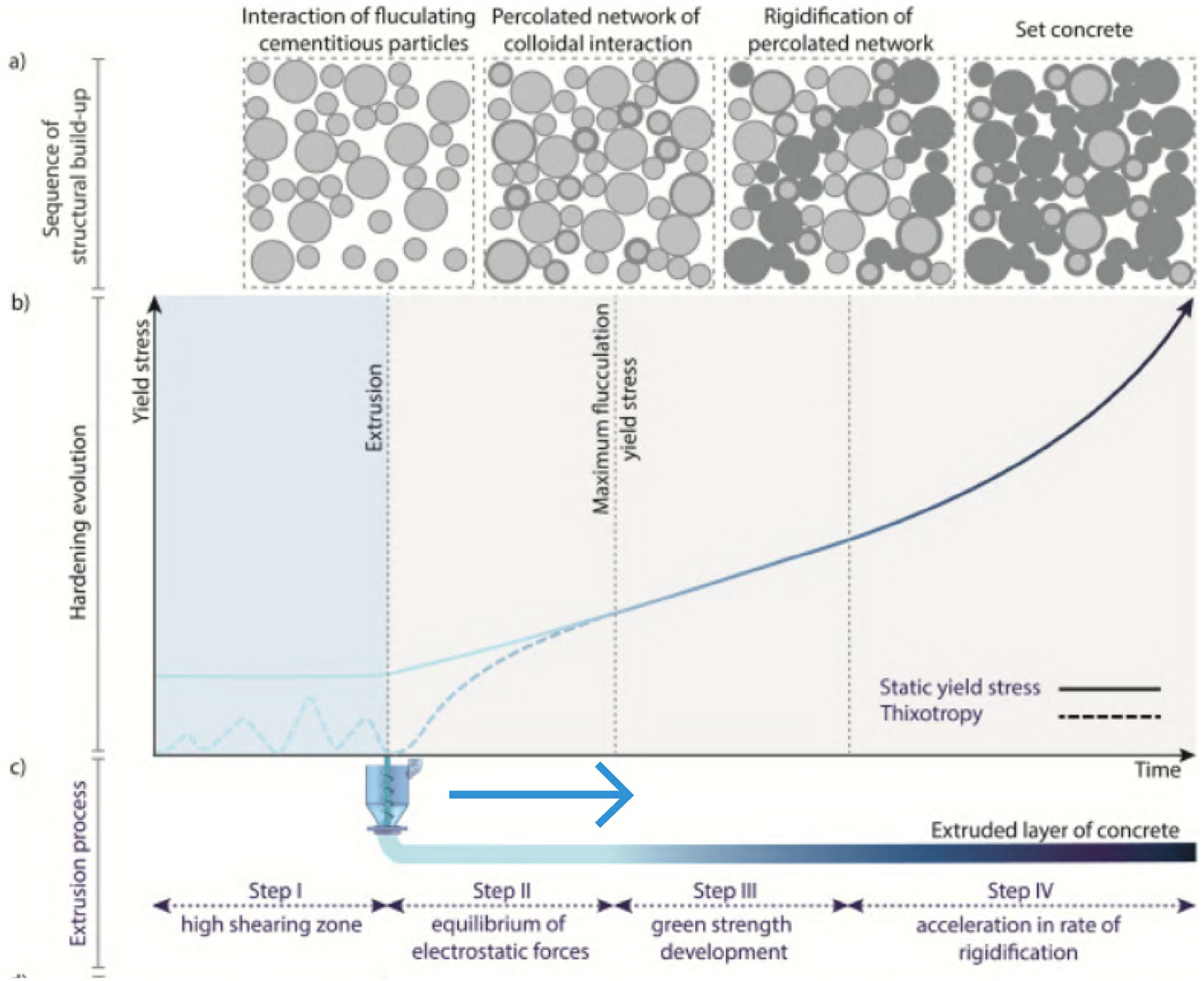




Parts Geometry



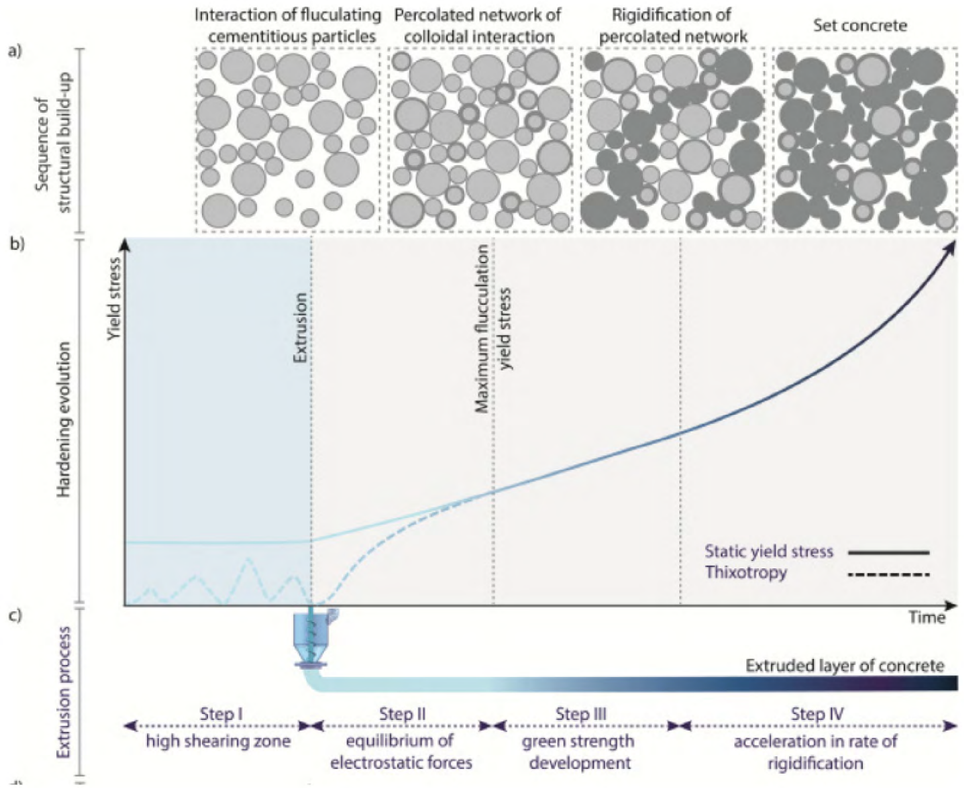
POST-PROCESSING ANOMALIES ?



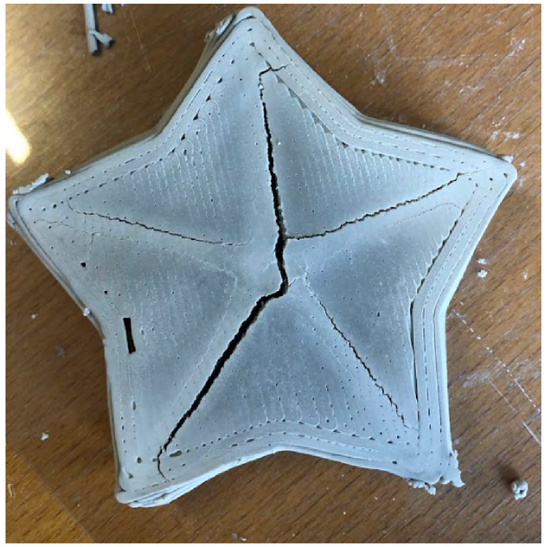
Multi-aspect schematic of the early hardening evolution of extruded concrete from mixing initiation up to setting [4]

[4] :Ranjbar, Navid, et al. "Rheological characterization of 3D printable geopolymers." Cement and Concrete Research 147 (2021)

POST-PROCESSING ANOMALIES ?

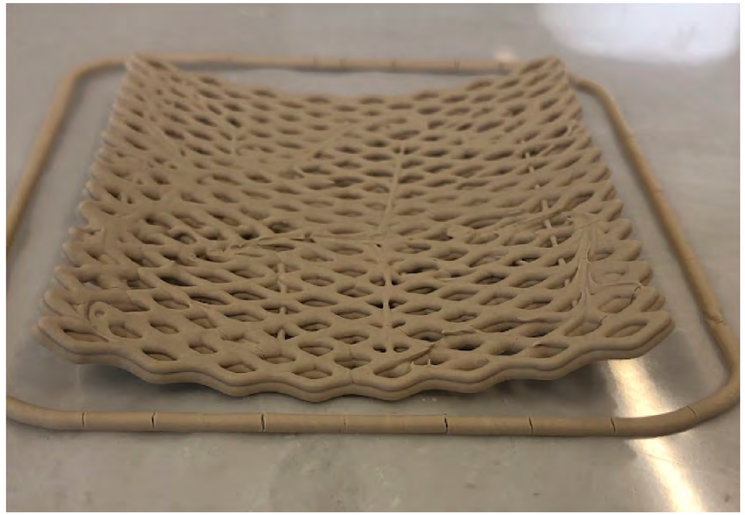


Multi-aspect schematic of the early hardening evolution of extruded concrete from mixing initiation up to setting



Crack Propagation

Dimensional Instability



Warping

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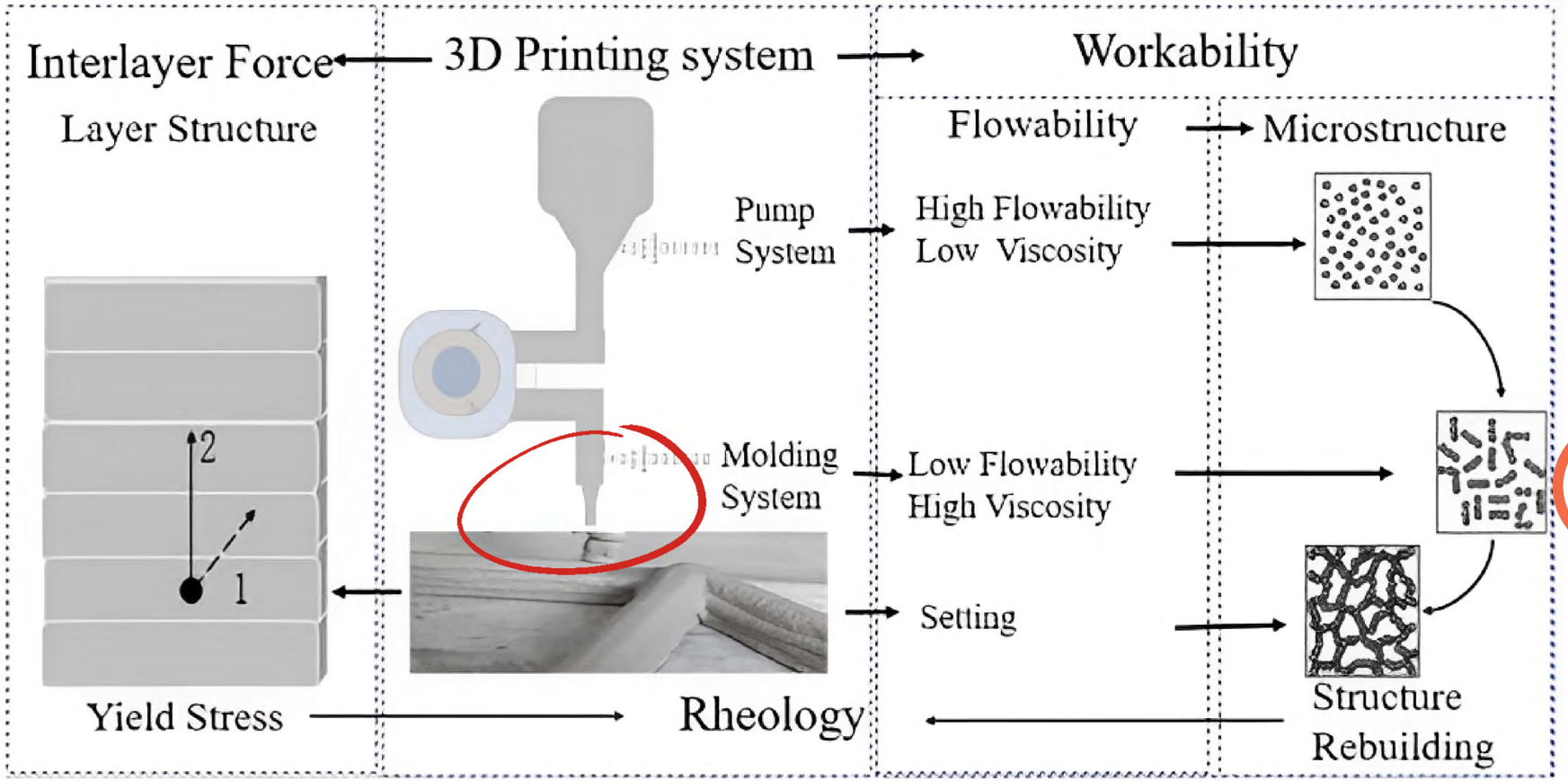
RHEOLOGICAL CHARACTERIZATION

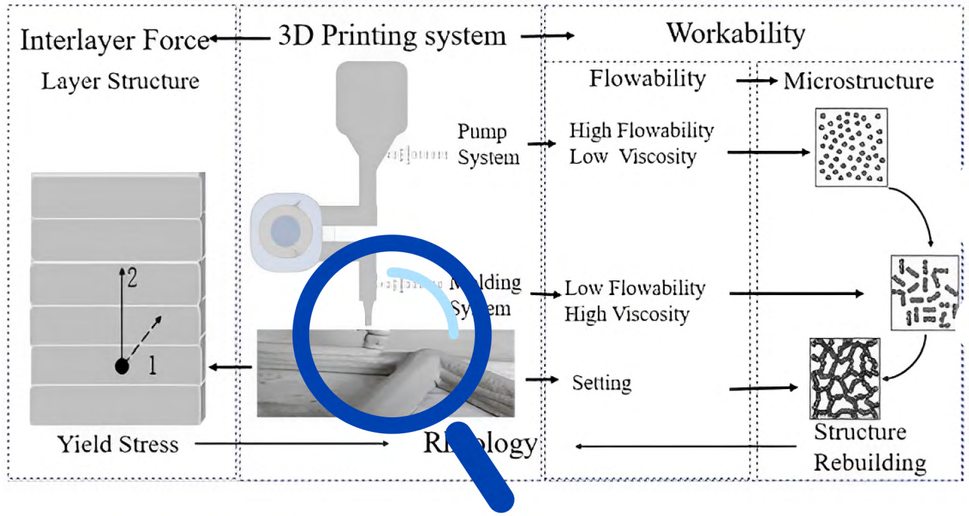
IMPORTANCE OF RHEOLOGY ?



Workability of 3D printing geopolymer [5]

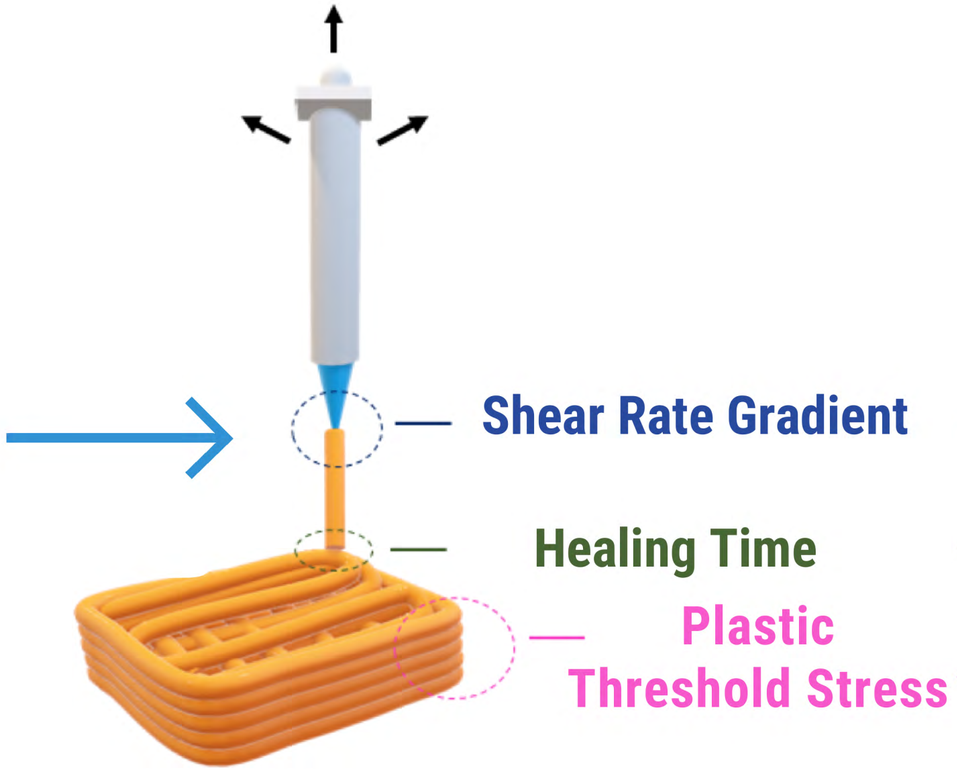
[5]: D-W Zhang et al. "The study of the structure rebuilding and yield stress of 3D printing geopolymer pastes." Construction and Building Materials 184 (2018)

RHEOLOGICAL REQUIREMENTS



Workability of 3D printing geopolymer

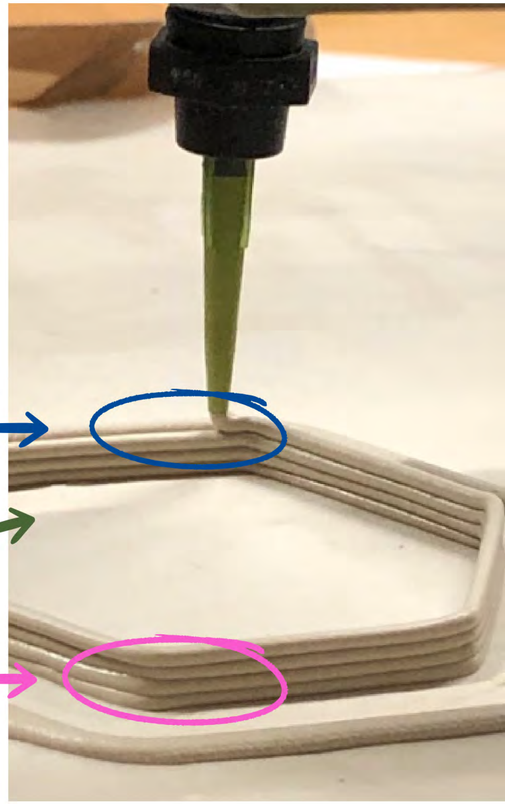
EXTRUSION & DEPOSITION PHASE



Shear Rate Gradient

Healing Time

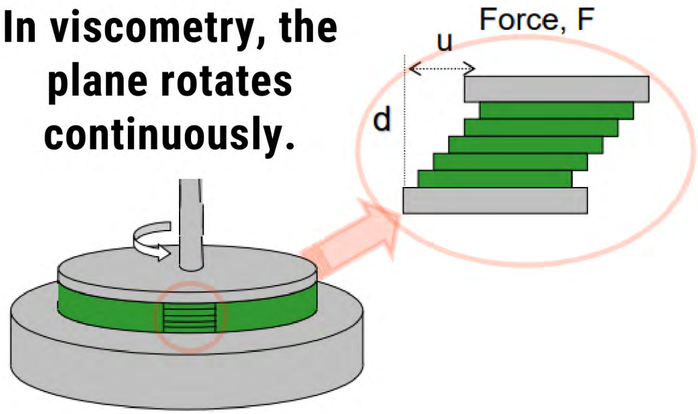
Plastic Threshold Stress



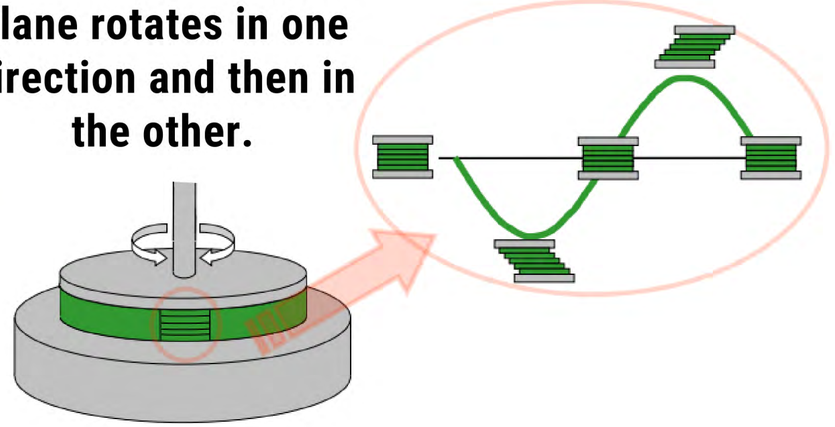
RHEOLOGICAL CHARACTERIZATION

Kinexus Rotational Rheometer Lab+

In viscometry, the plane rotates continuously.

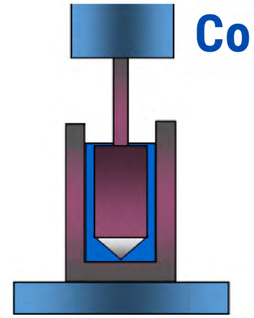


In oscillation, the plane rotates in one direction and then in the other.



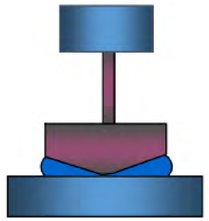
Concentric cylinders

Viscosity Very low to medium



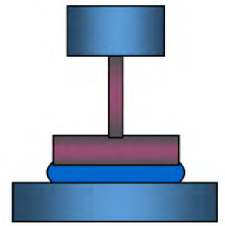
Cone and Plane

Viscosity Low to Medium

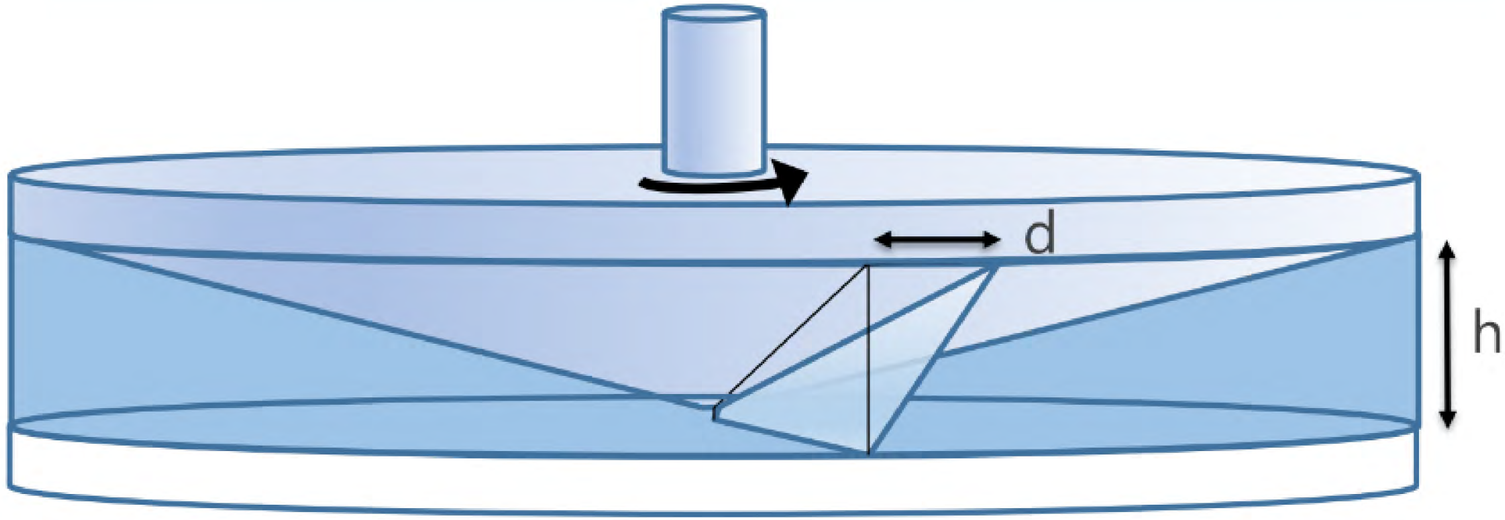


Parallel planes

Low Viscosity to Soft Solids



RHEOLOGICAL CHARACTERIZATION



Shear stress (τ)
=
Applied force (F) / Surface

Unit : Pa

Shear Deformation (γ)
=
Displacement (d) / Gap (h)

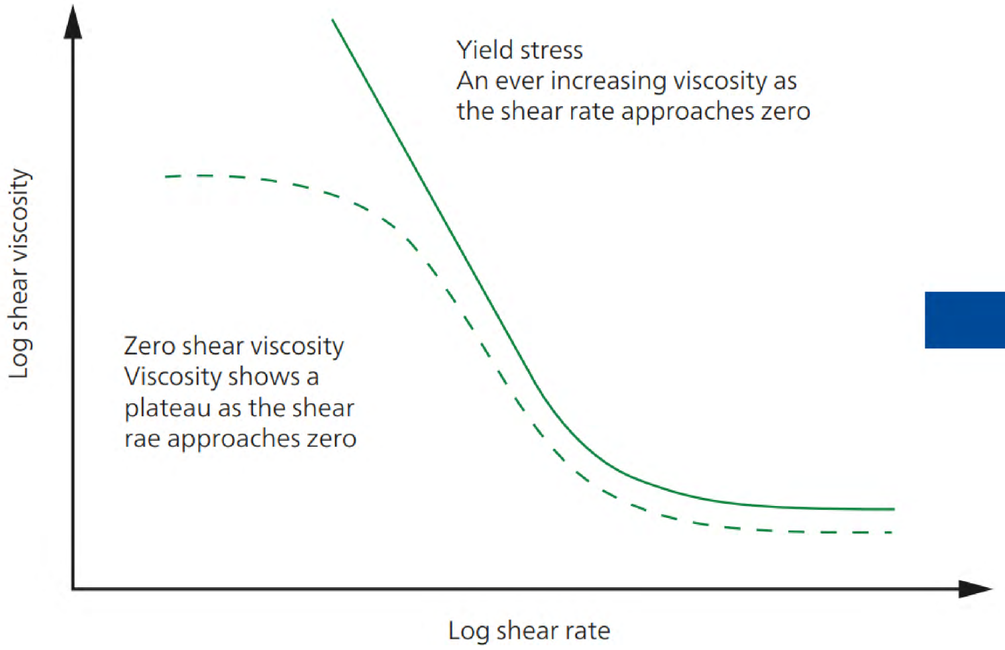
Unit : None

Shear rate (γ̇)
=
Deformation (d) / Gap (h) x Time (s)

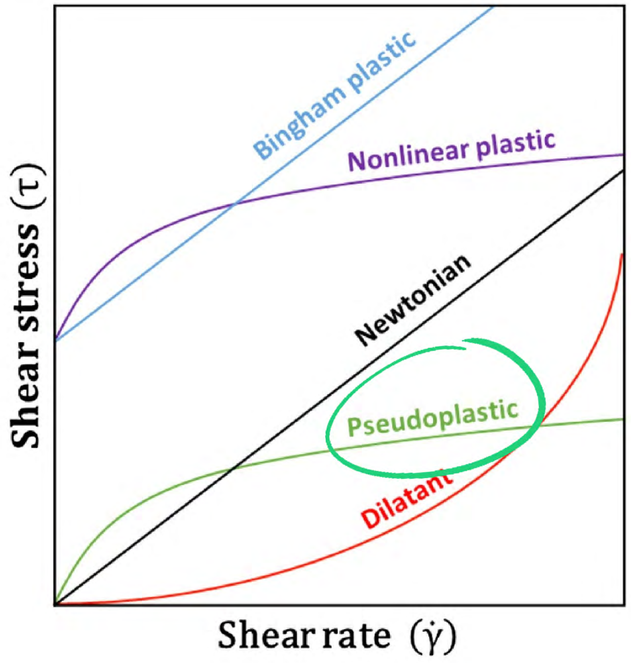
Unit : 1/s

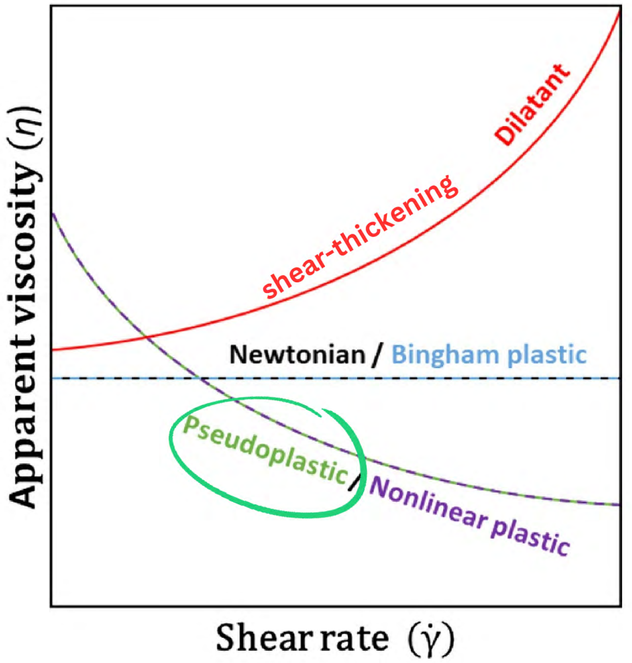
RHEOLOGY TESTS

VISCOMETRY SHEAR RATE



Typical flow curves for shear thinning fluids with a zero shear viscosity and an apparent yield stress [6]





Pseudoplastic : Shear-thinning

Dilatant : Shear- thickening

Rheological Behavior

[6]: A Basic Introduction to Rheology, Netzch

RHEOLOGY TESTS

THREE STEP SHEAR RATE -THIXOTROPY TEST

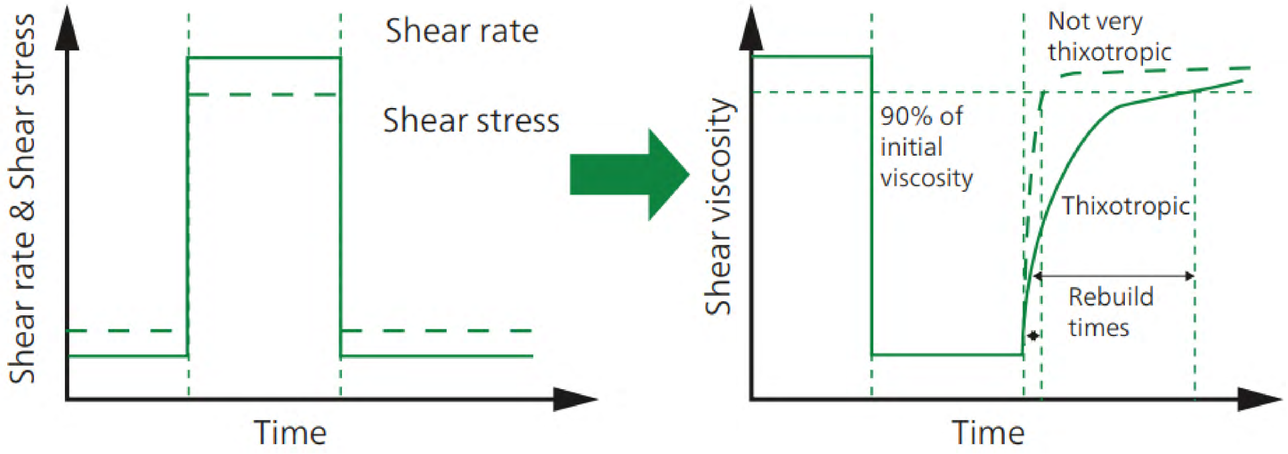
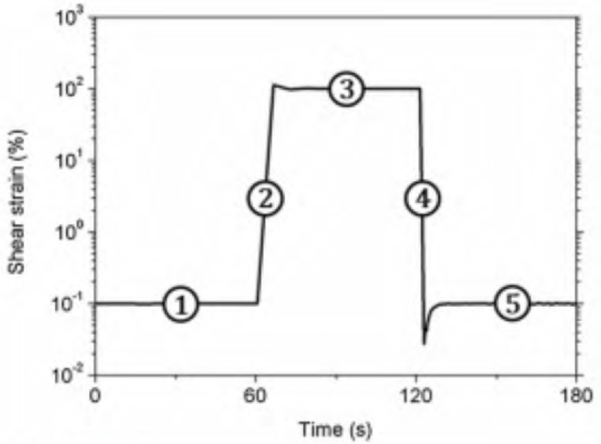
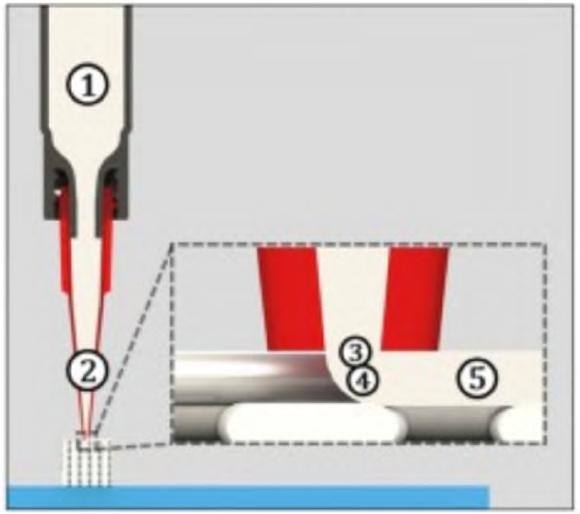


Illustration showing a step shear rate test for evaluating thixotropy and expected response for non-thixotropic and thixotropic fluids





DIW printing process [7]

[7] :del-Mazo-Barbara, et al. "Rheological characterisation of ceramic inks for 3D direct ink writing: A review."
Journal of the European Ceramic Society 41.16 (2021)

RHEOLOGY TESTS

OSCILLATION AMPLITUDE & FREQUENCY

Oscillation Amplitude - Determine Linear Viscoelastic Region (LVER)

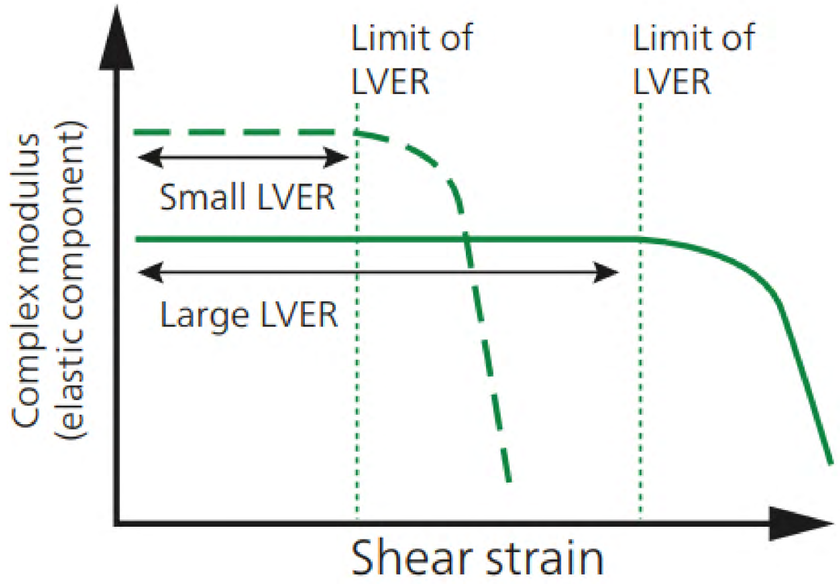
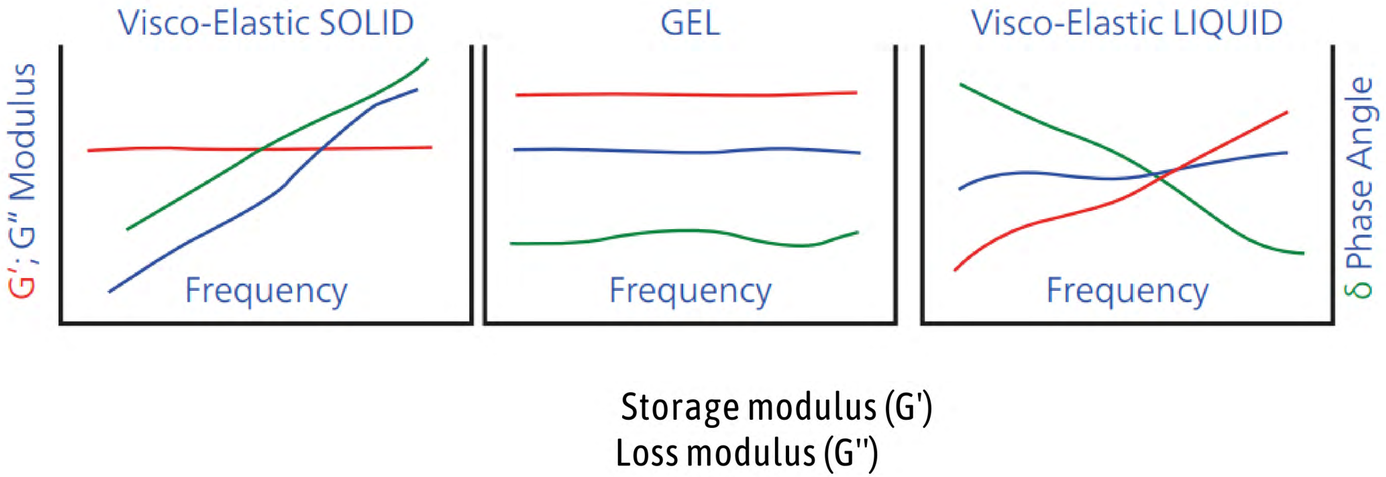


Illustration showing the LVER for different materials as a function of applied strain

Oscillation Frequency - Measure Viscoelastic Response



Typical frequency response for a visco-elastic solid, visco-elastic liquid and a gel in oscillatory testing

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PRINTABLE GEOPOLYMER FORMULA

GEOPOLYMER FORMULA COMPONENTS

1 GEOPOLYMER BINDER

Metakaolin M88 - TEMPOZZ XATICO
Potassium Silicate - Geosil WOELLNER

2 GEOPOLYMER FILLERS

Micronized Feldspar - IMERYS
Wollastonite - XATICO

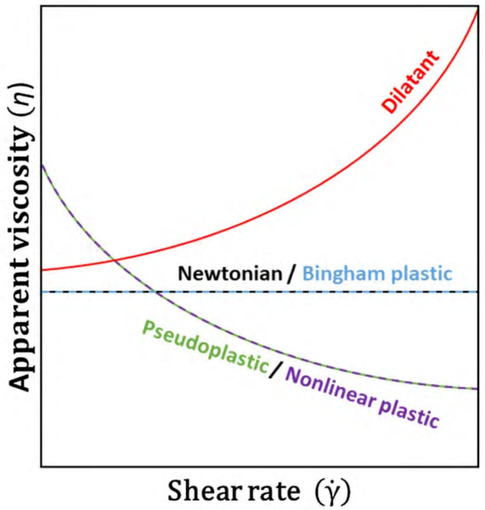
3 ADMIXTURE OR GELLING AGENT

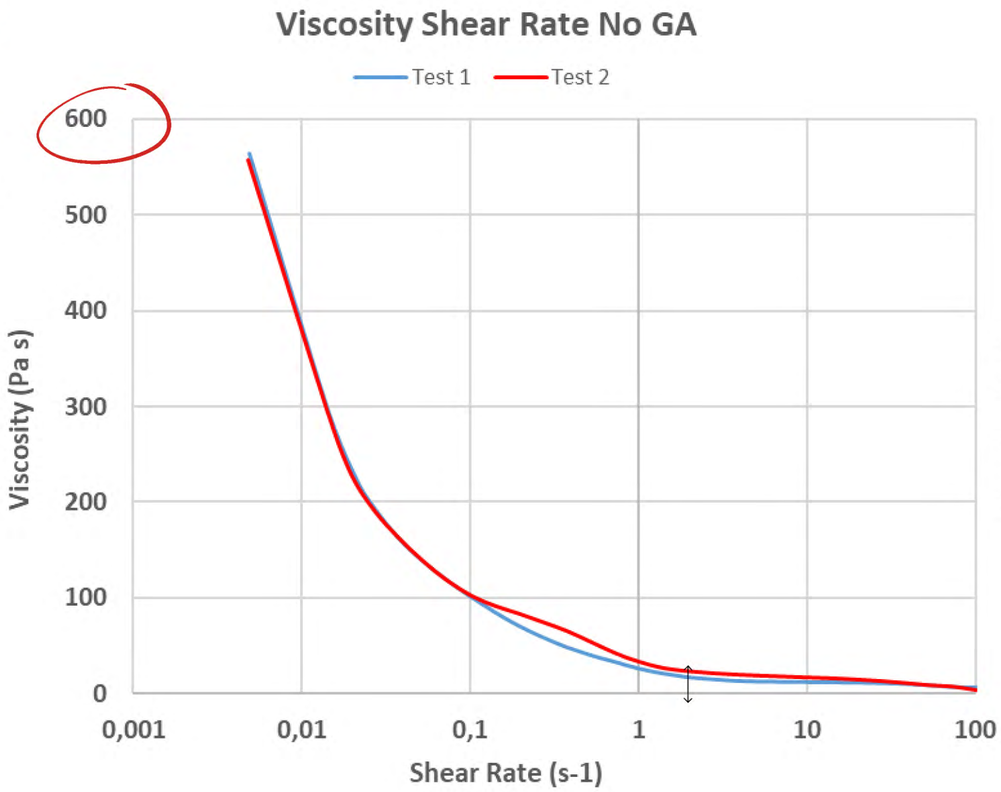
Xanthan Gum - Food Grade

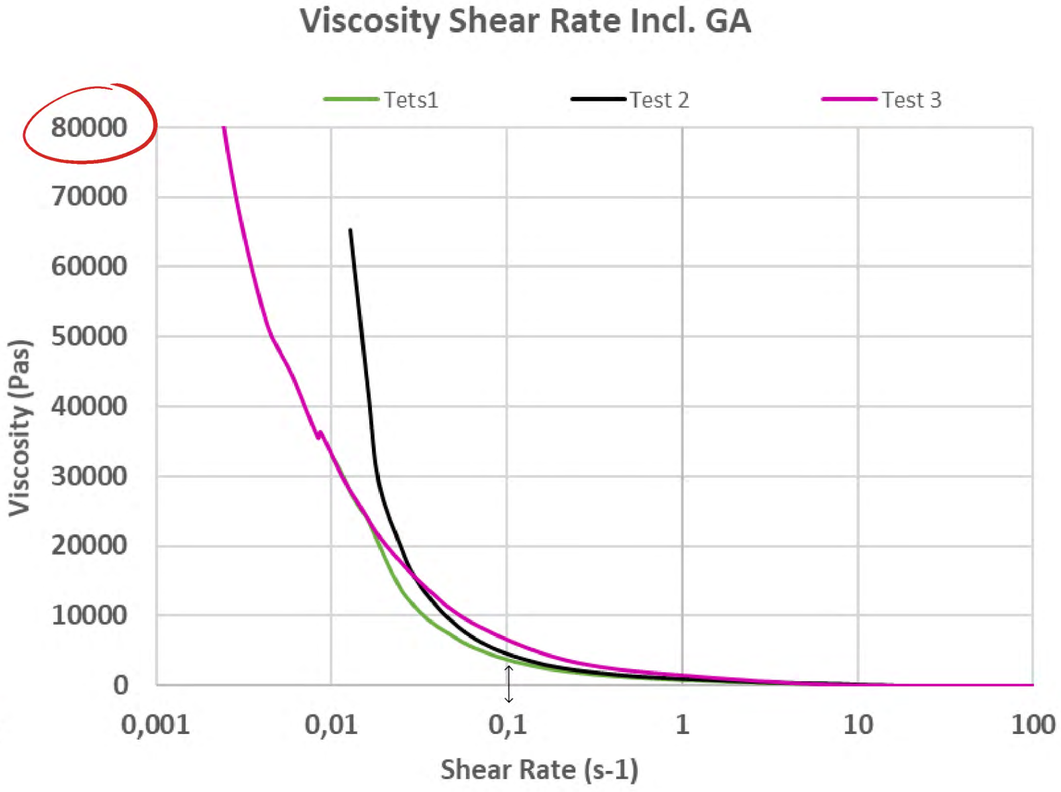
RHEOLOGICAL PROPERTIES

Viscometry Shear Rate

Incl. and No Gelling Agent



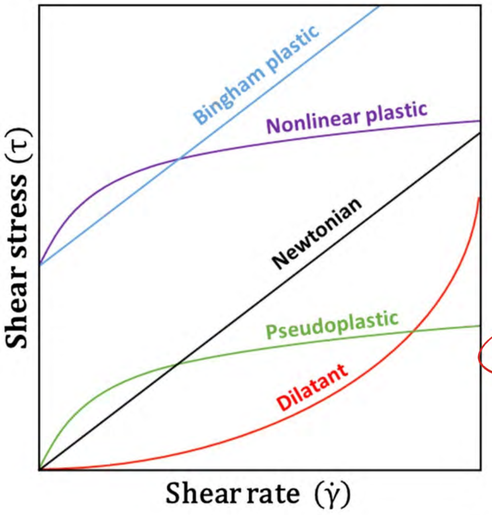


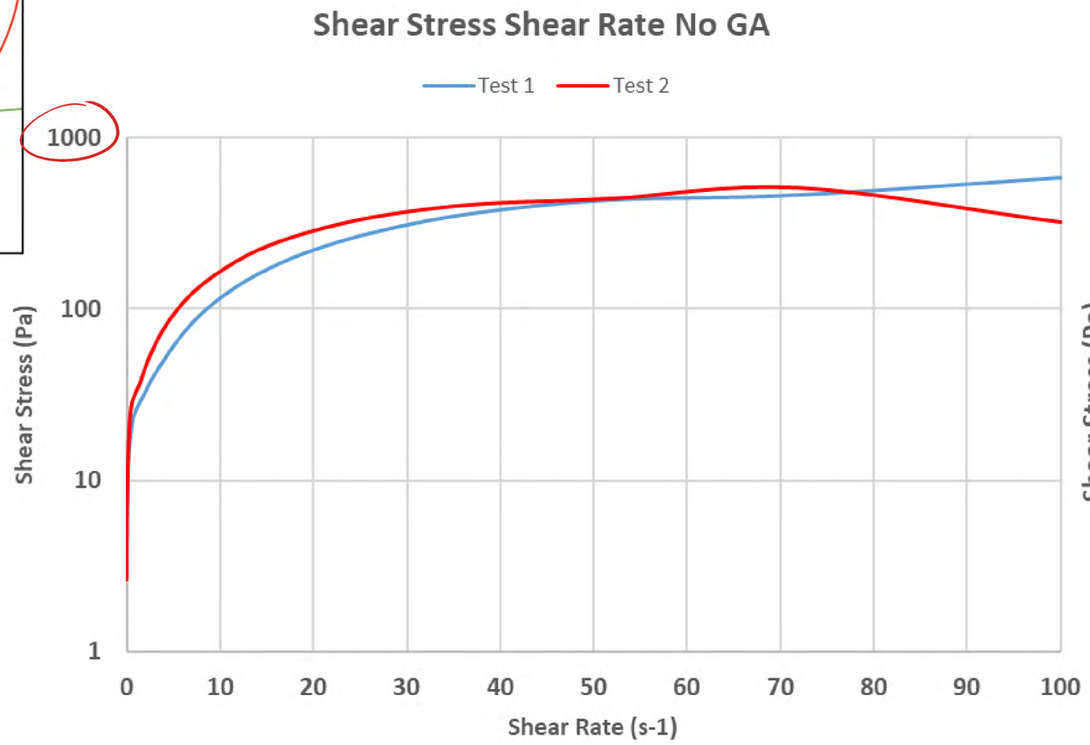


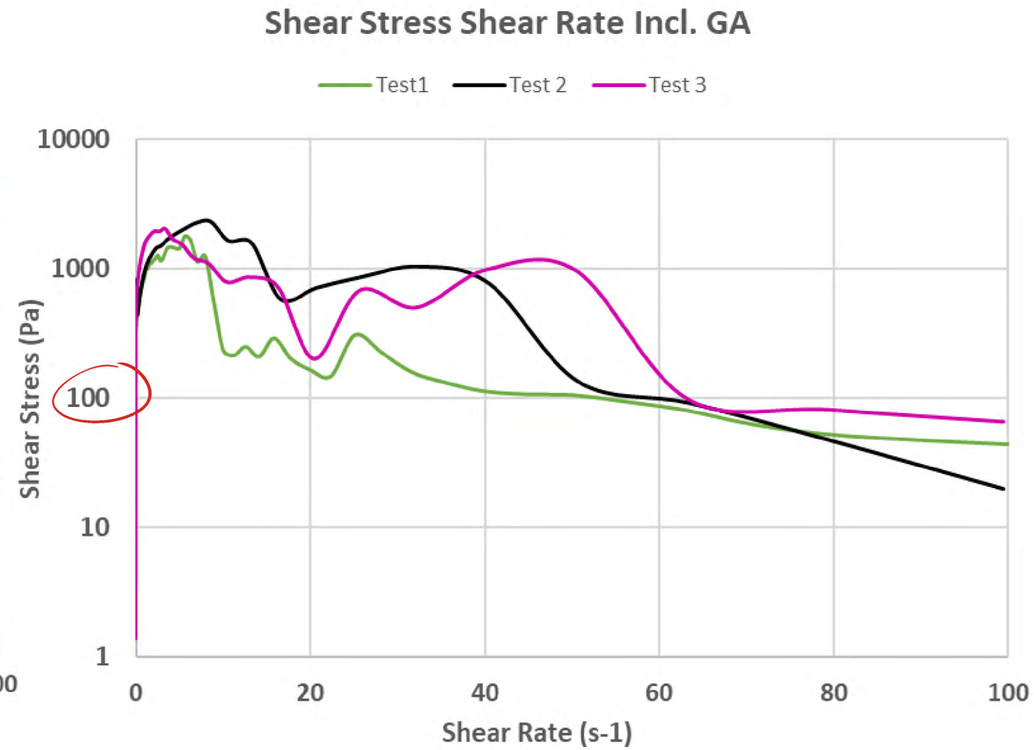
RHEOLOGICAL PROPERTIES

Viscometry Shear Rate

Incl. and No Gelling Agent



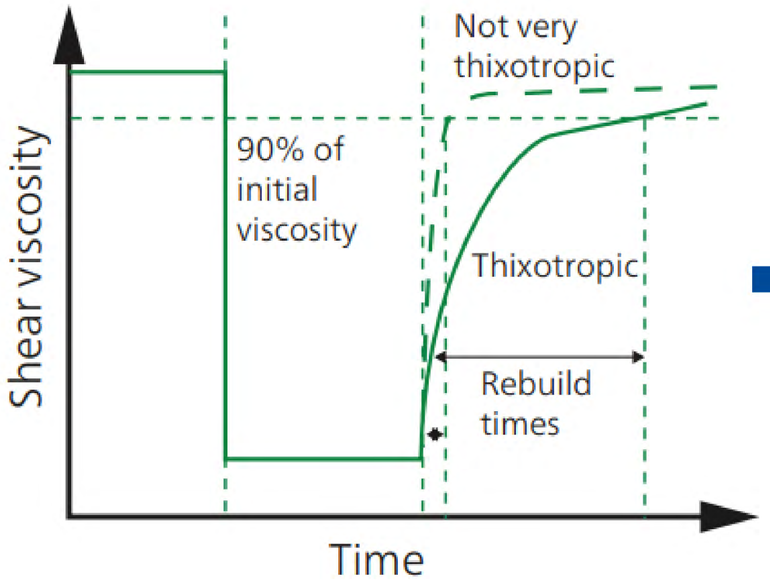


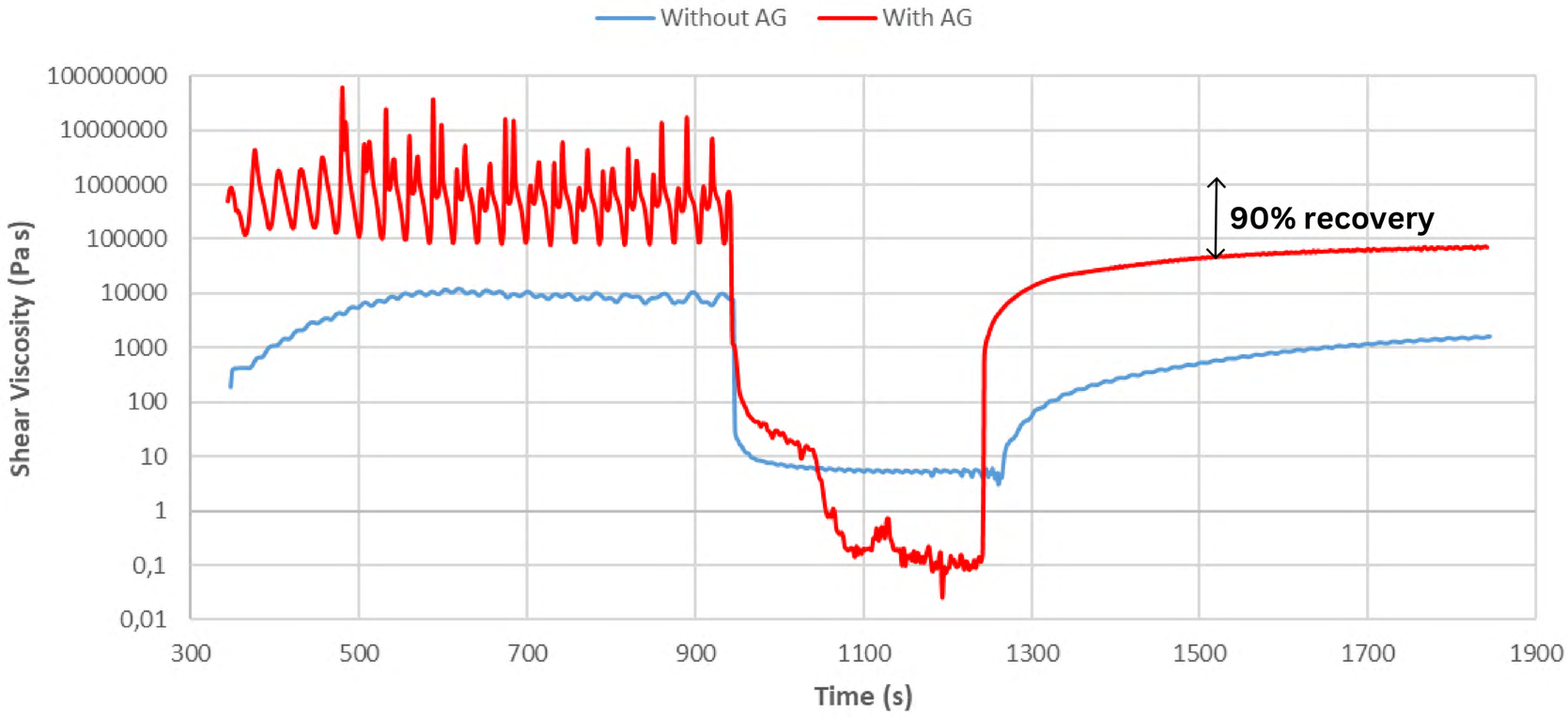


RHEOLOGICAL PROPERTIES

Three Step Shear Rate -Thixotropy Test

Incl. and No Gelling Agent

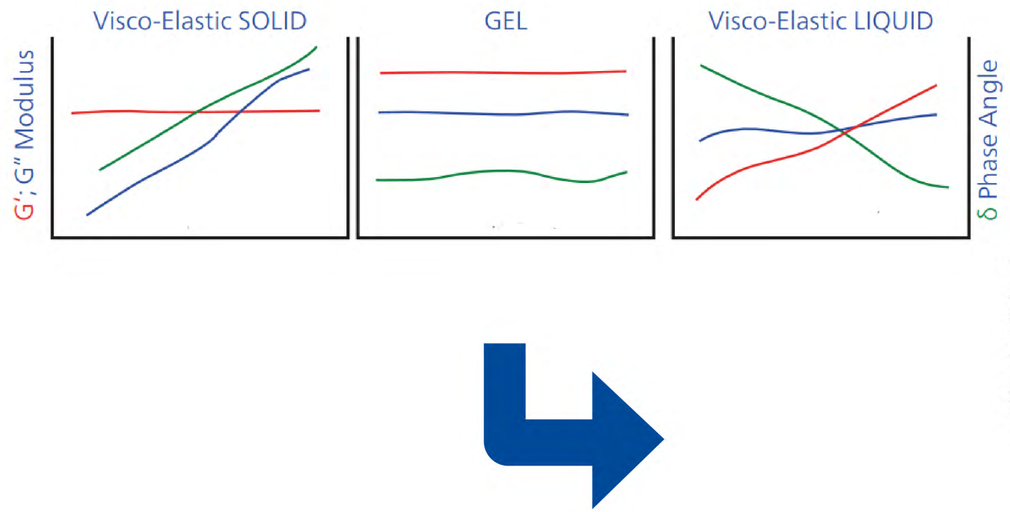


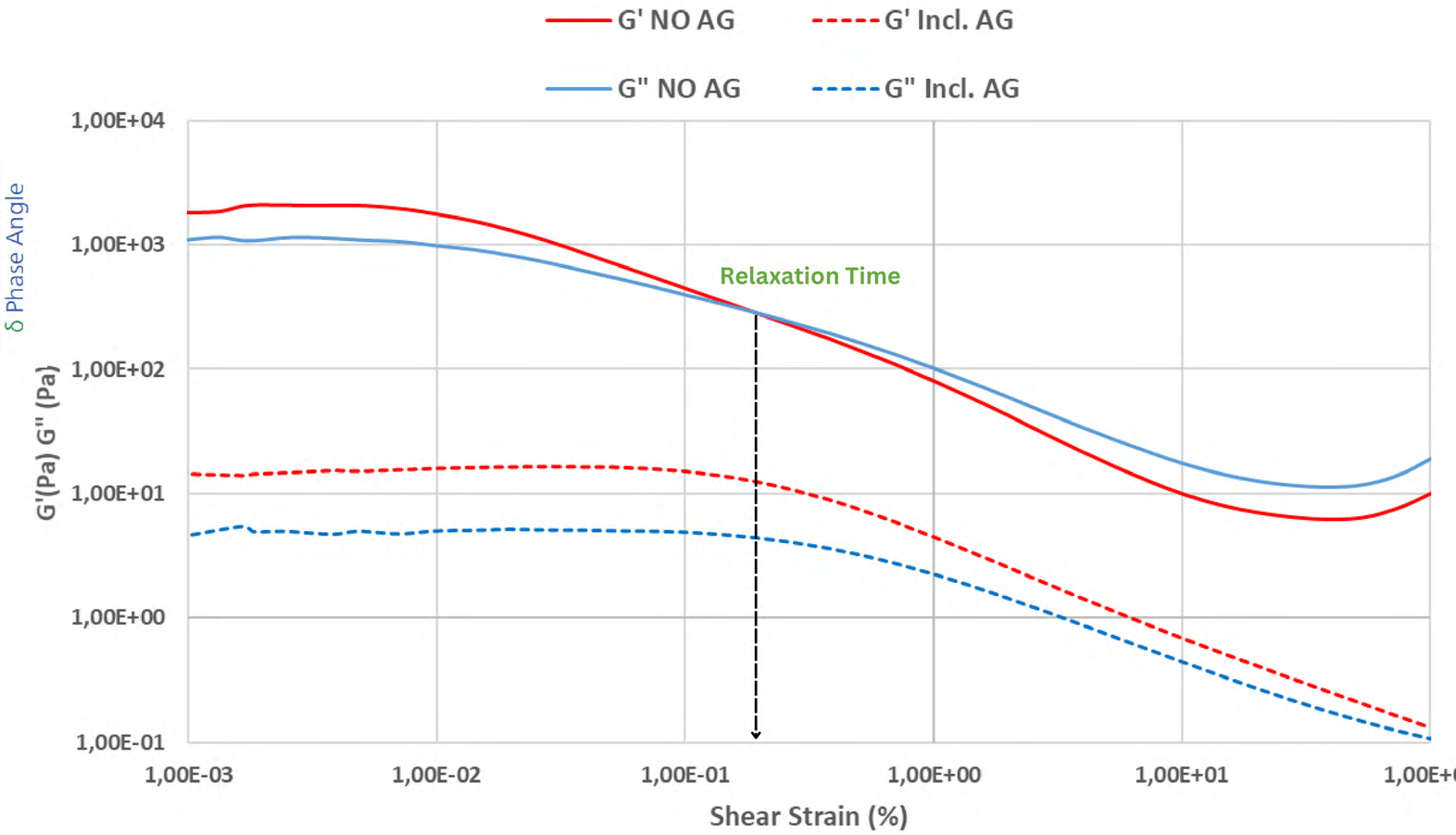


RHEOLOGICAL PROPERTIES

Oscillation Amplitude

Incl. and No Gelling Agent

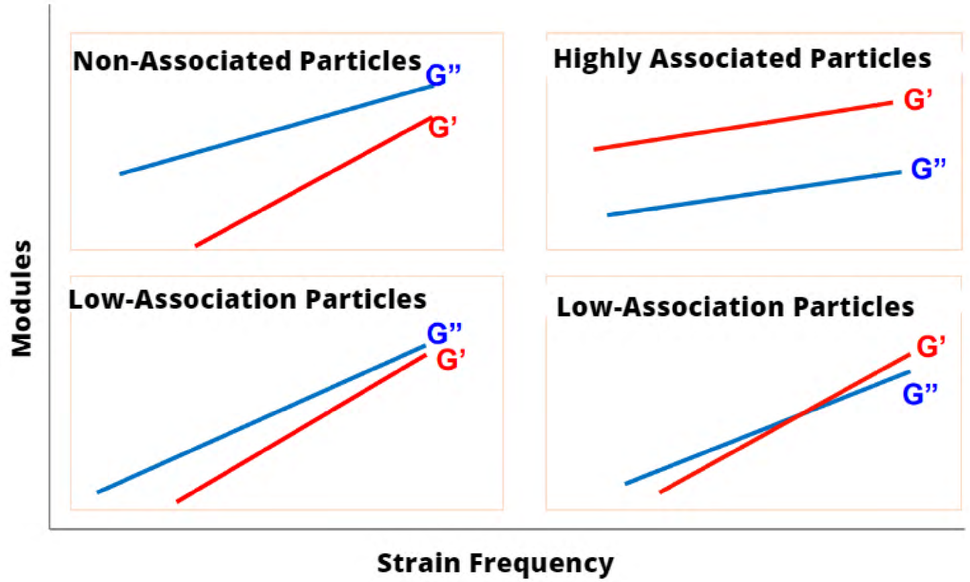


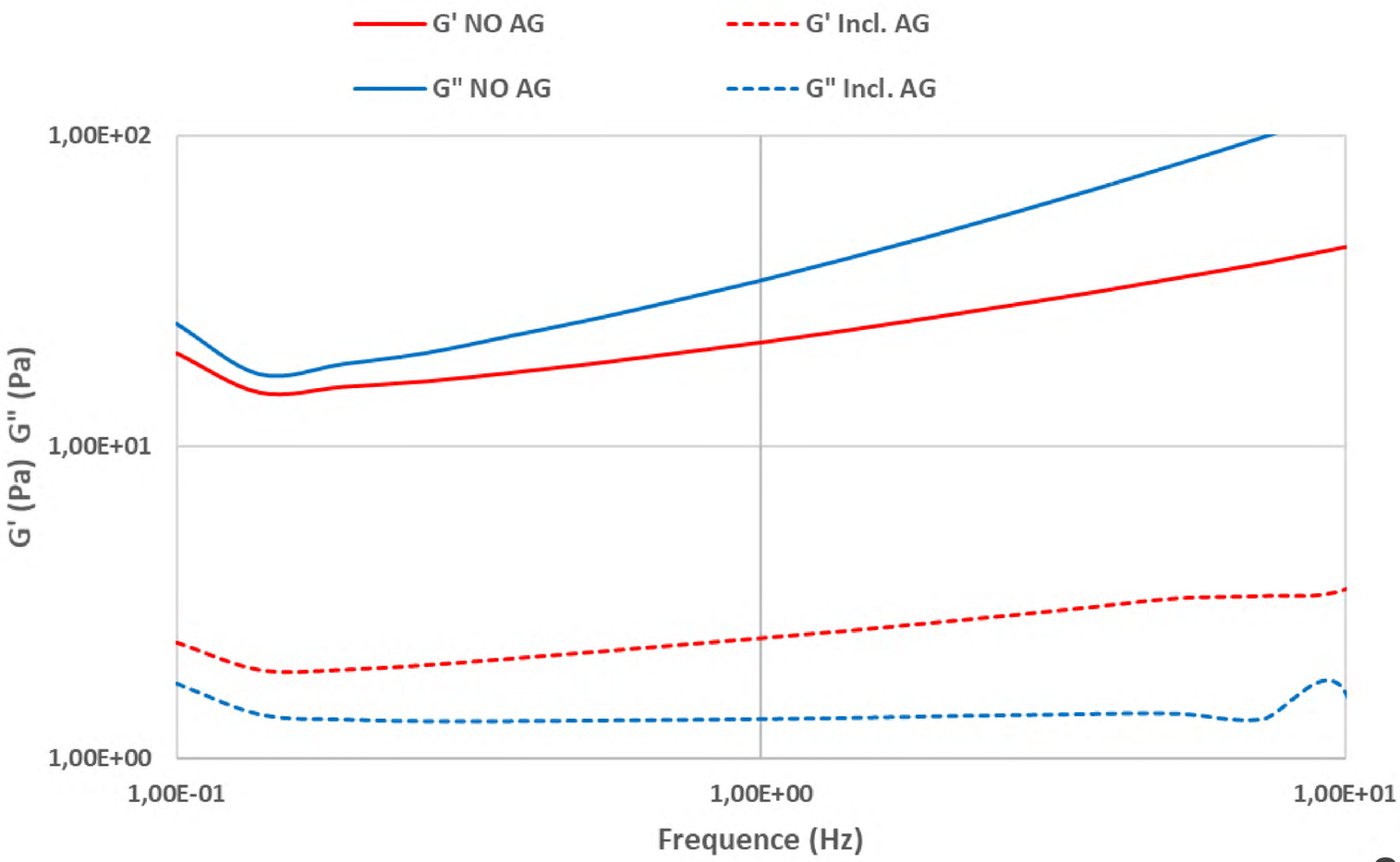


RHEOLOGICAL PROPERTIES

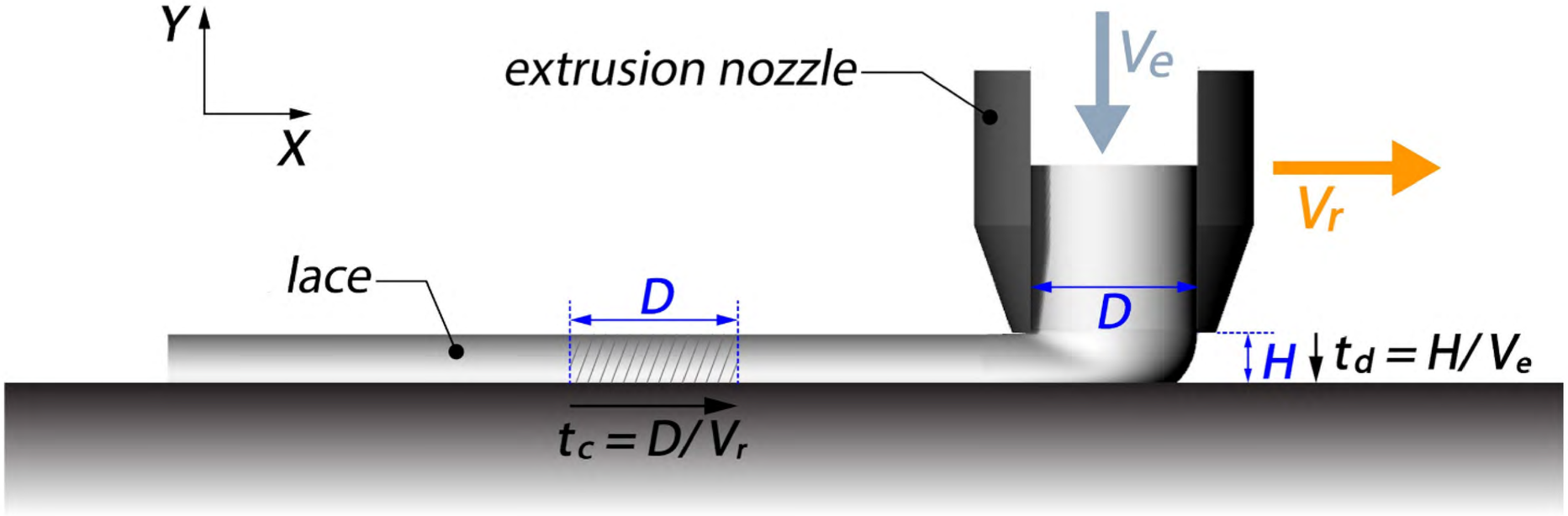
Frequence Amplitude

Incl. and No Gelling Agent





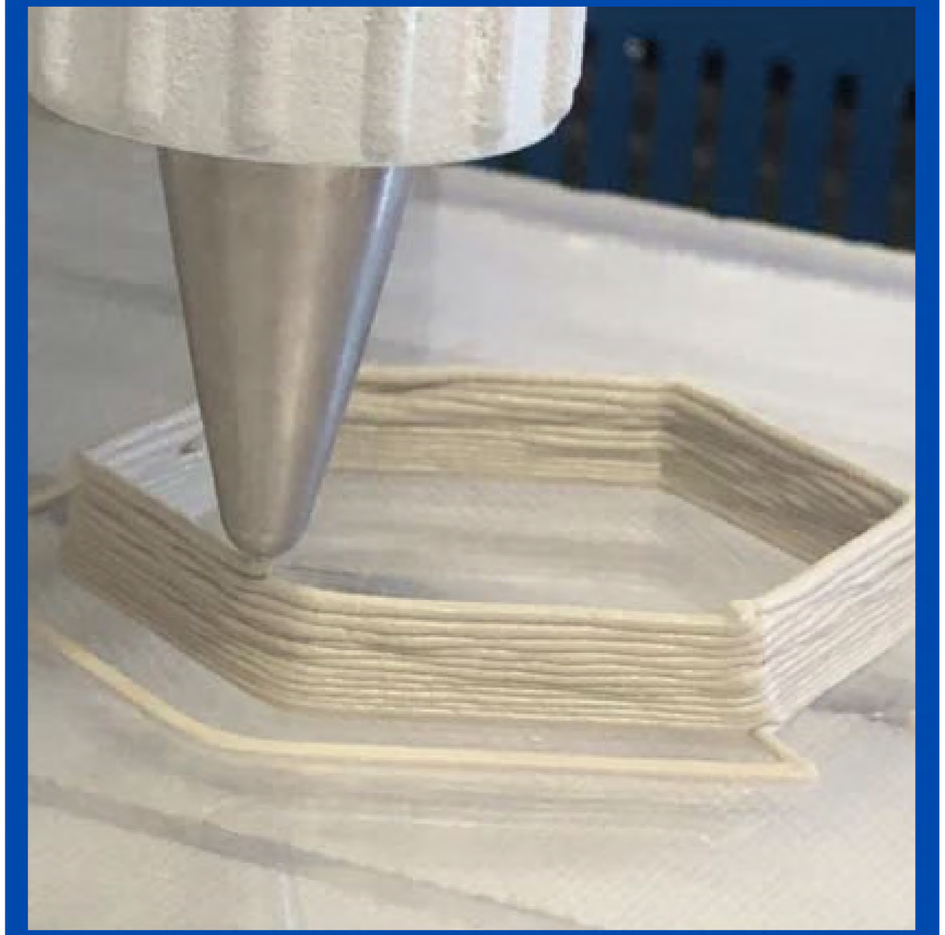
PRINT SETTINGS



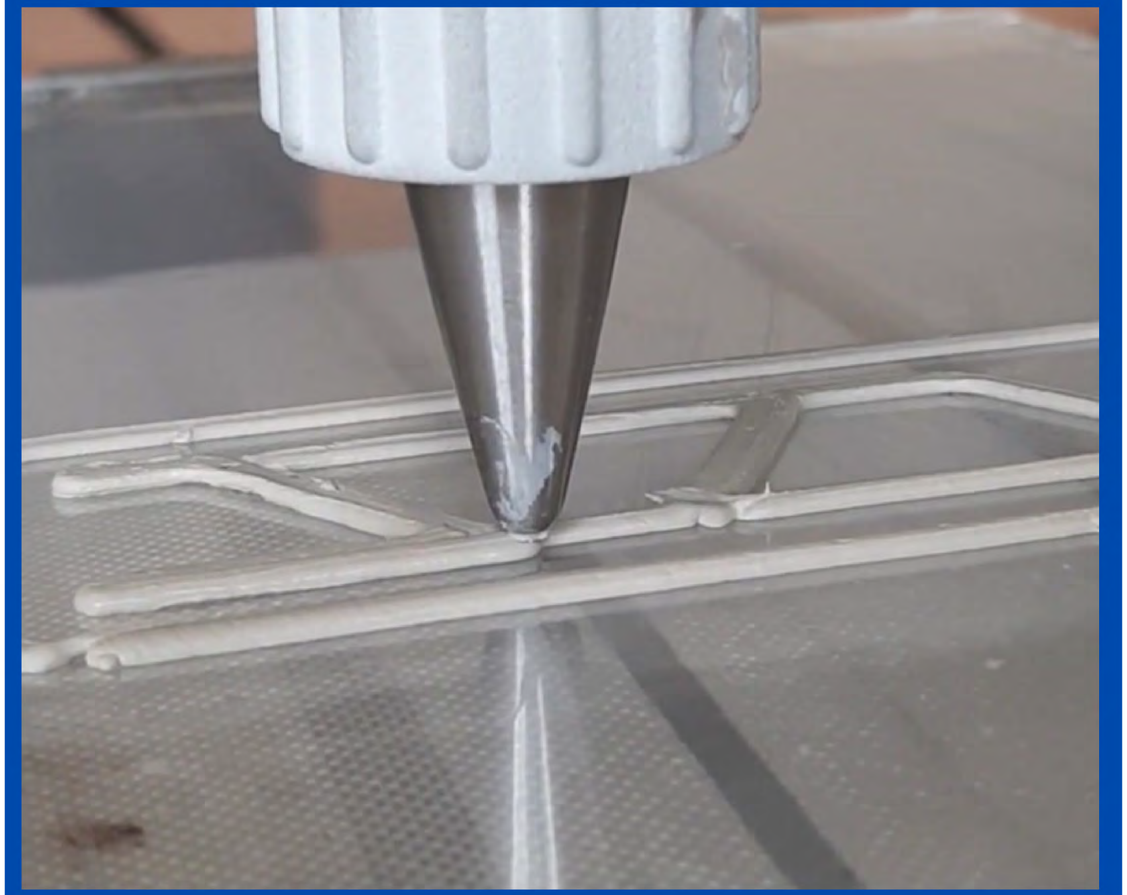
Ve: Extrusion speed
Vr: Robot Speed
H: Layer Hight
D: Noozle Diameter

PRINT SETTINGS

Without Gelling Agent



Over pressing of the sublayers
Nozzle =1mm

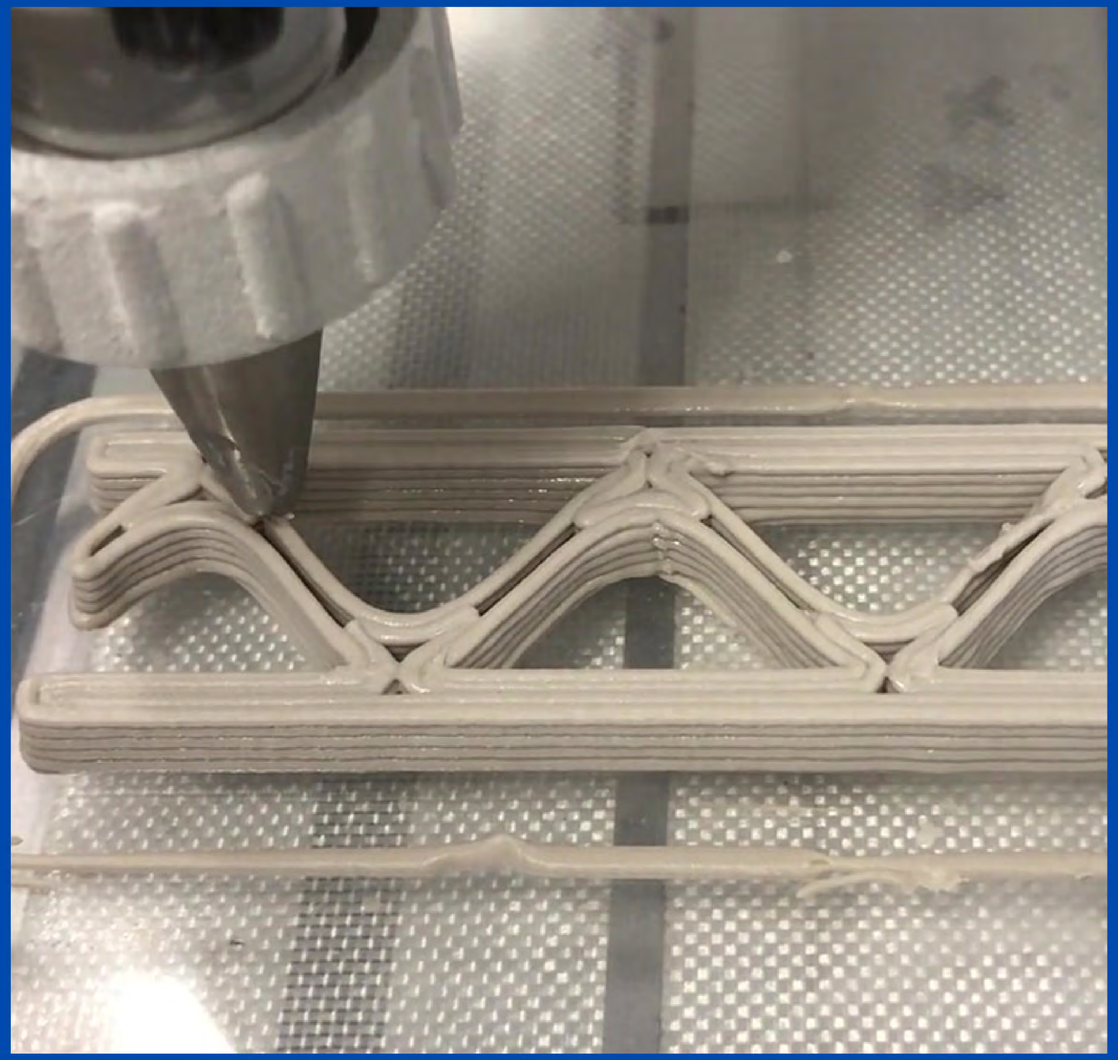


Uncontrolled layer flow

PRINT SETTINGS

With Gelling Agent

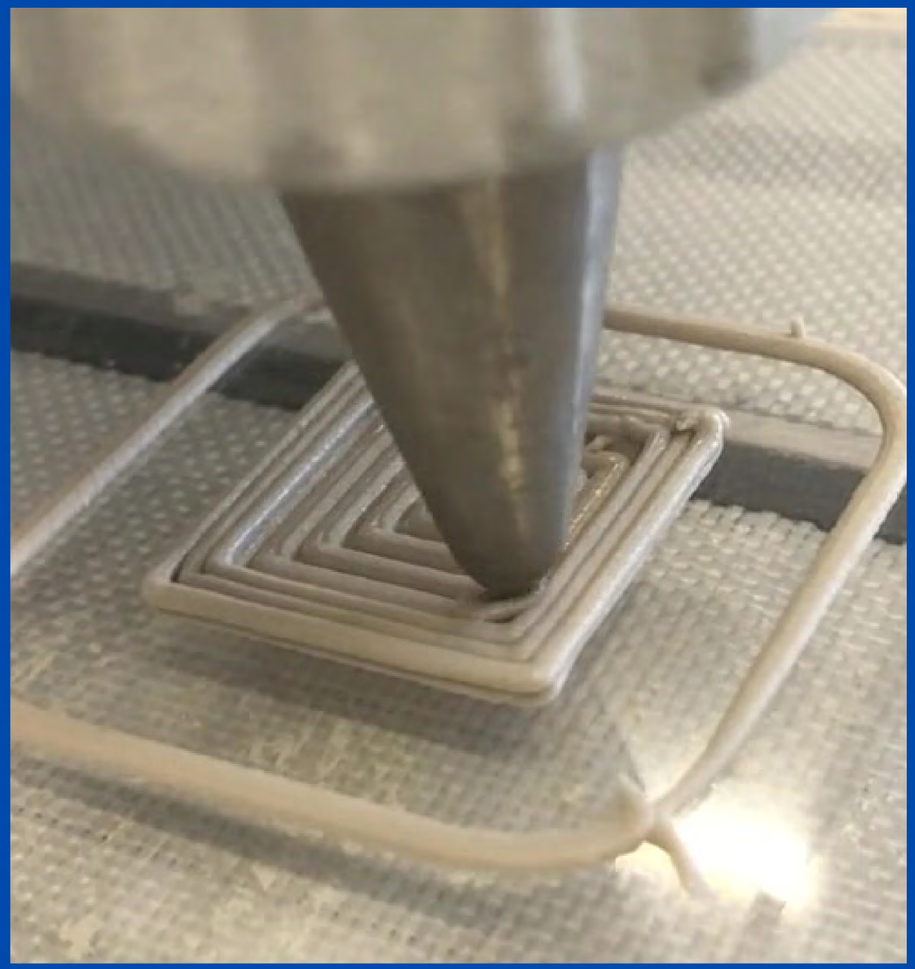
Controlled layer flow
Nozzle Diameter = 1mm



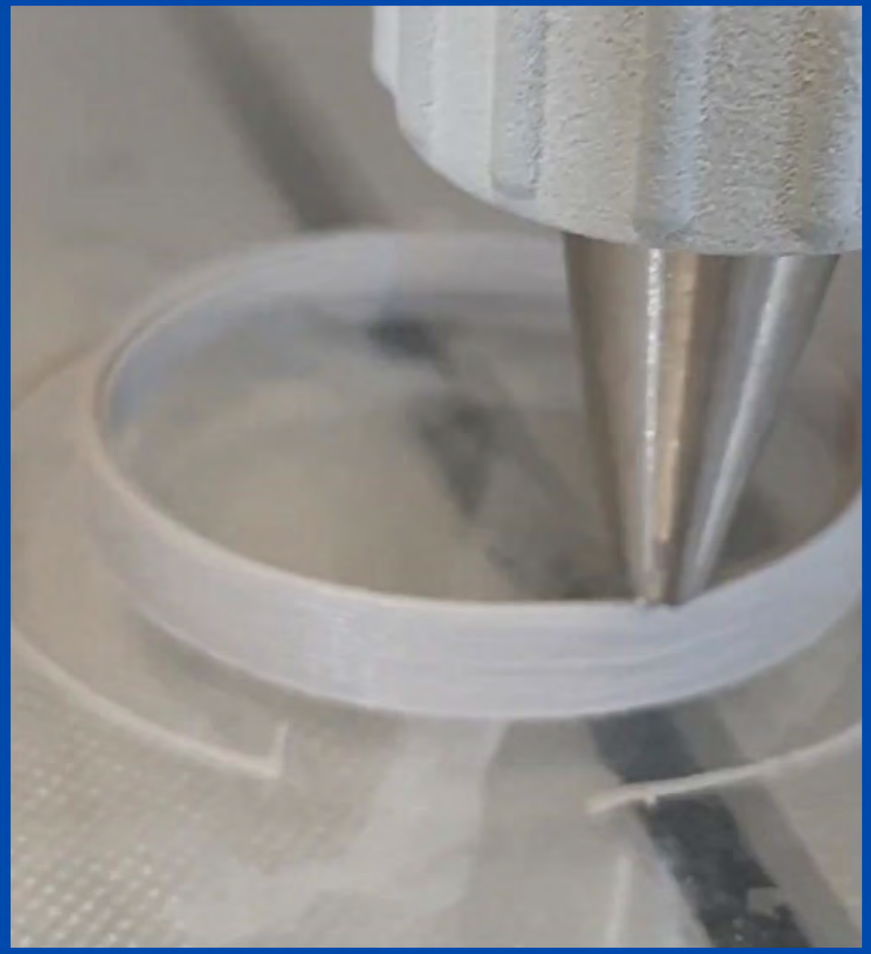
Well-balanced pressing of the sublayers

PRINT SETTINGS

With Gelling Agent

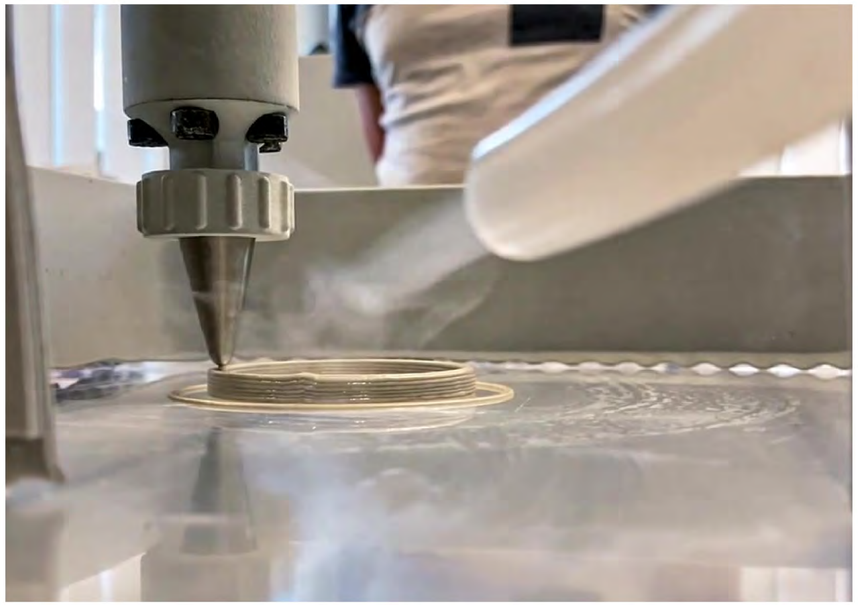


Infill



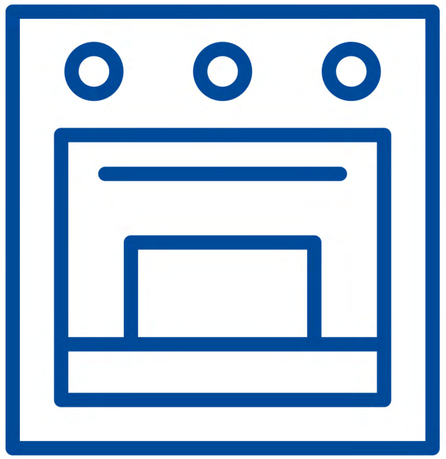
Nozzle Diameter =0.7mm

POST PROCESSING SETTINGS



Printing in humid atmosphere

24 H



24 H oven curing

Incorrect curing process
-VS-
Good post-processed curing

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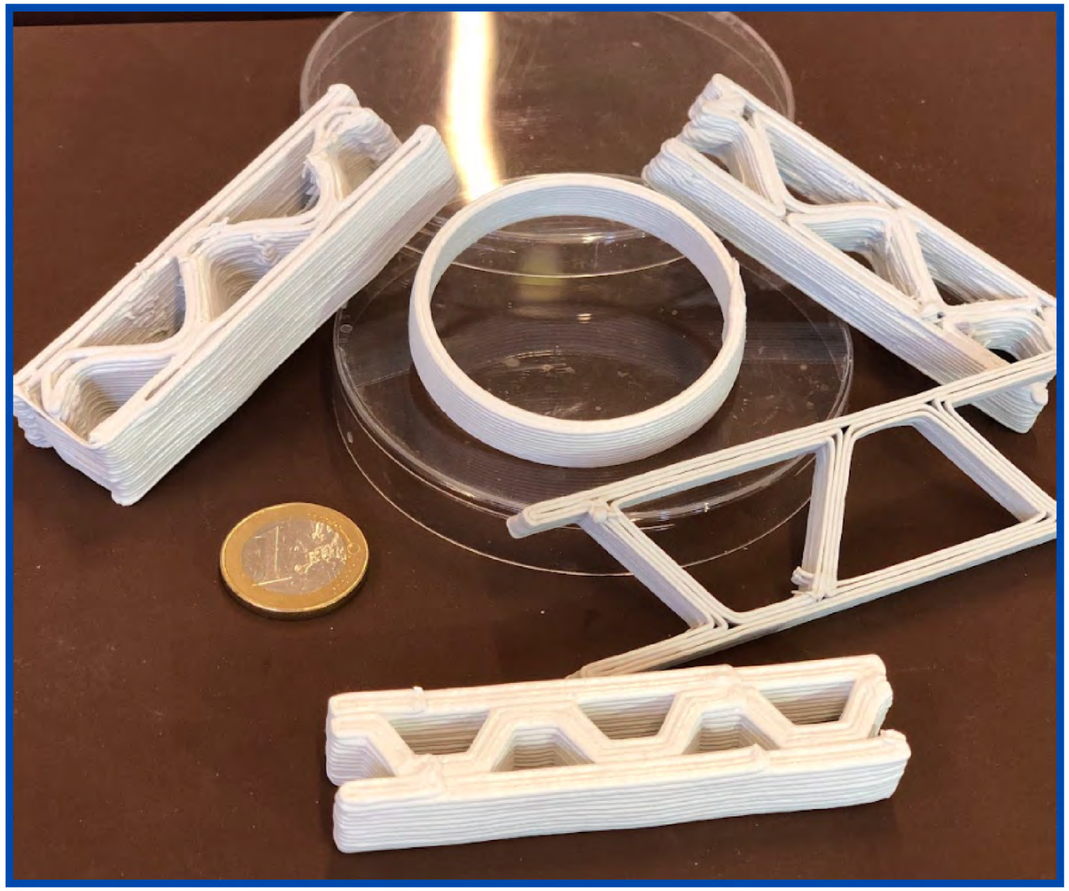
OUTLOOK

CONCLUSION



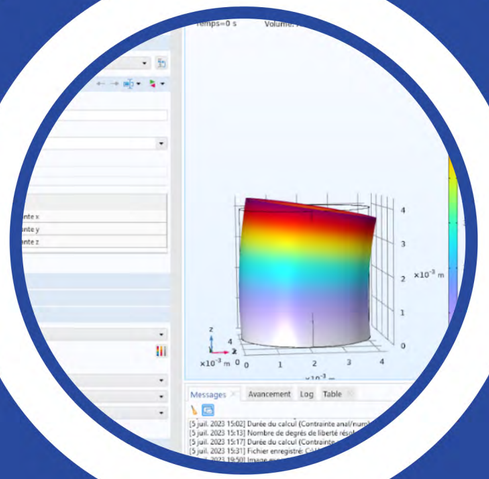
UNDERSTANDING THE INFLUENCE OF RHEOLOGY ON GEOPOLYMER BEHAVIOR THROUGHOUT THE EXTRUSION PROCESS

OPTIMIZING PRINTING PARAMETERS ACCORDING TO GEOMETRY AND RHEOLOGY OF GEOPOLYMER

ENSURING DIMENSIONAL STABILITY OF THE PART UNDER CONTROLLED POST-PROCESSING

OUTLOOK

DEVELOPMENT OF A PRINTING MACHINE WITH SENSORS FOR GEOPOLYMERS



DEVELOPMENT OF A PREDICTIVE MODEL VIA NUMERICAL SIMULATION OF THE ROBOCASTING PROCESS

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Thank you !

Questions ?

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Co-supervisor : **Mr Ralph DAVIDOVITS**