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shaping7

Applied R&D Projects in Additive Manufacturing

Hélio Jorge
CTCV-Technology Centre for Ceramics and Glass. Coimbra. Portugal

10-09-2019. 7th Shaping Conference. Aveiro. Portugal

SOCIEDADE PORTUGUESA DE CERÂMICA E VIDRO

ECERS

FAB@CTCV
Projeto de Demonstração de Tecnologias de Fabrico Avançado para a Indústria

Cofinanciado por: CENTRO 2020 PORTUGAL 2020 UNIÃO EUROPEIA Fundo Europeu de Desenvolvimento Regional

Index

- CTCV introduction
- Overview of the AM technologies for ceramics in the market
- An R&D collaborative project of robocasting for decorative ware
- A study on the processing of alumina by robocasting

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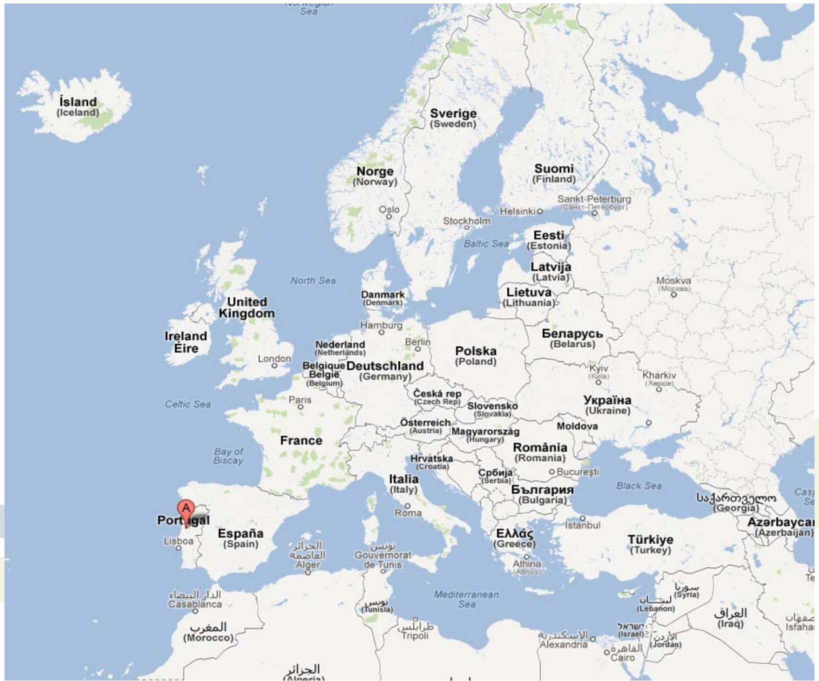
CTCV Introduction

- Established in 1987 by ceramic and glass industrial associations and companies
- Non-profit organisation
- One of the Centres of Technology Interface (CIT) of Portugal
- Located in Coimbra, Centro Region, Portugal
- Business Activities
  - Innovation and Development (R&D)
  - Technical Consultancy
  - Testing
  - Training



CTCV Introduction

CITT Centro Interface
CTCV

- 60 collaborators | 550 costumers (2018)
- Accredited organisation for supply of services under Portuguese financial instruments - Innovation/R&D, Entrepreneurship, Incubation, Industry 4.0 e and Circular Economy
- Participation in more than 20 R&D projects (H2020, Portugal 2020 and others)
- Member of Portuguese clusters: PRODUTECH, Sustainable Habitat Cluster and MOBINOV

PRODUTECH

centroHabitat
Plataforma para a Construção Sustentável

MOBINOV :: Cluster Automóvel
PORTUGAL

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Mission of the R&D Department

- Industrial research, development and innovation (R&Di)

  TRL starting from Level 3
- Innovation, Intellectual Property and Technology Transfer
- Participation in R&D and innovation funded projects

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Infrastructure

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R&D Department

Activities

- Applied research and development
- Pre-industrial studies
- Prototyping
- Small production

Technical Fields

- Materials
- Ceramics
- Manufacturing technologies
- Digital technologies for product development

Technical fields

MATERIALS

- ceramic material
  - traditional ceramics
  - technical and advanced ceramics
- integration with other material families
  - polymers
  - nanomaterials

MANUFACTURING TECHNOLOGIES

- ceramic technologies
  - pressing
  - extrusion
  - sintering / firing
- powder technologies
  - powder injection moulding
    - ceramic injection moulding
    - metal injection moulding
  - additive manufacturing
    - robocasting
    - fused filament fabrication

- **PRODUCT DEVELOPMENT**
  - reverse engineering
  - virtual modelling, photo reality
  - rapid prototyping

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AM methods for ceramics available in market

Classification of the methods for additive manufacturing :

- Photopolimerisation
- Material injection

→ Liquids

- Material extrusion
- Lamination

→ Solids

- Binder jetting
- Powder melting
- Direct deposition

→ Powders

Photopolymerisation / Stereolithography/ SLA

Materials (Suspension)

- Alumina, Zirconia, Silicon nitride
- Tricalcium Phosphate TCP and hydroxyapatite

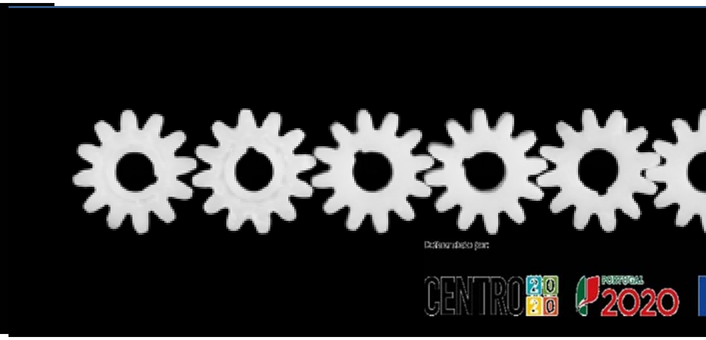
- density: >99%

Potential applications

- Biomedicine / bone replacement
- Dental restoration
- Casting cores
- Technical ceramics apps

Available technologies

- Lithoz (Austria)
- 3D Ceram (France)
- Admatec (Holand)



Binder jetting / binder inkjet

Materials (Powder)

- Gypsum / Binder
- Alumina - Silica
- Silica

- density: ~50%

Available technologies

- 3DSystems (USA)
- Voxeljet (Germany)
- Tethon3D (USA)

Potential applications

- Casting tools
- Refractory
- Glazed decorative ware

Casting tool
Silica

Decorative piece
glazed alumina-silica

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Material extrusion

Materials (Paste)

- Plastic clay
- ...

Available technologies

- WASP (Italy)
- Lutum (Holand)
- 3D Potter (USA)
- Stone Flower (Germany)

Potential applications

- Decorative ware
- Sanitary ware
- Ceramic craft
- ...

Decorative pieces (green state)
Porcelain (CTCV)

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RoboCer.3D Project

Porcelanas da Costa Verde (Porcelain table ware manufacturer)

University of Aveiro

CTCV-Technology Centre for Ceramics and Glass

Additive manufacturing of porcelain ware

Used technology: Robocasting (or LDM – liquid dispensing modelling)

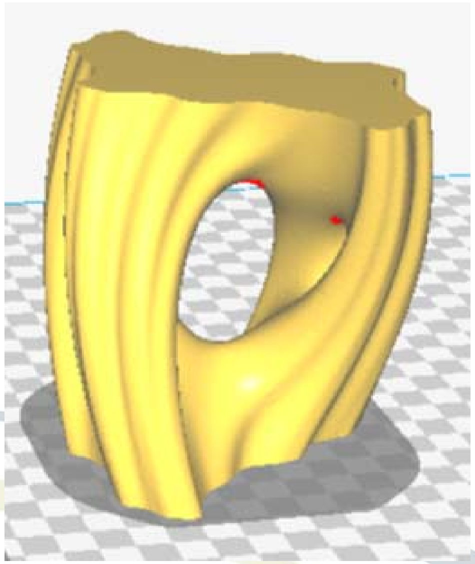
Technology developments:

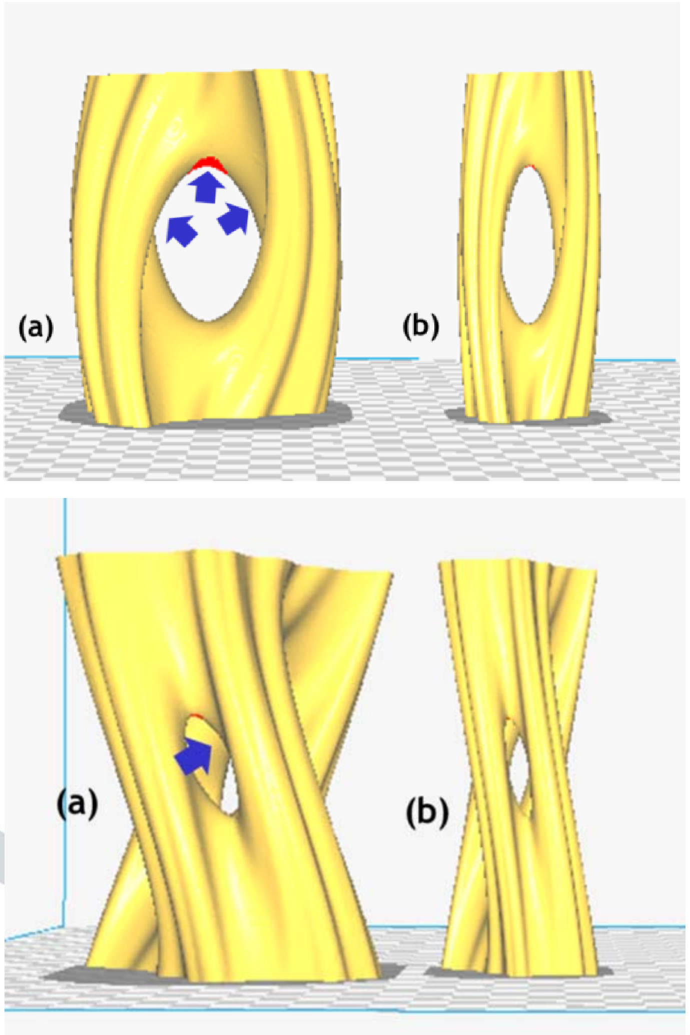
- Ceramic body composition manipulation
- Printer programing using a slicing software
- Setting up of the printing process
- Glazing and firing testing
- Printing validation tests:
    - Conventional geometry parts
    - Design-for-3DP new parts

Printing of a decorative piece of 10x10x40cm

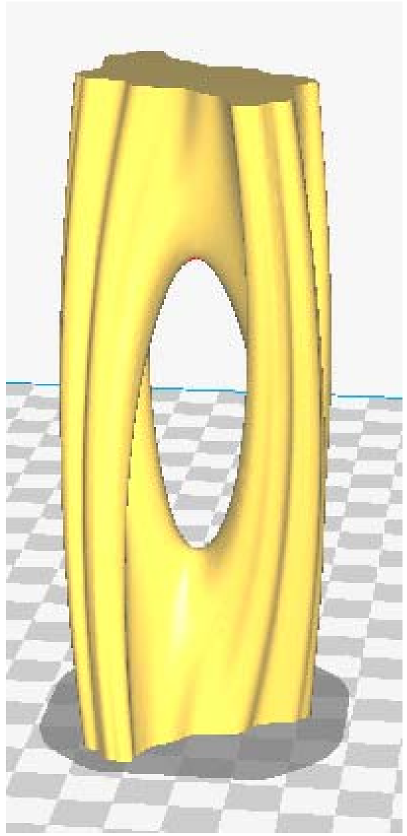
1st step: Evaluation of the print ability of the part

Original model



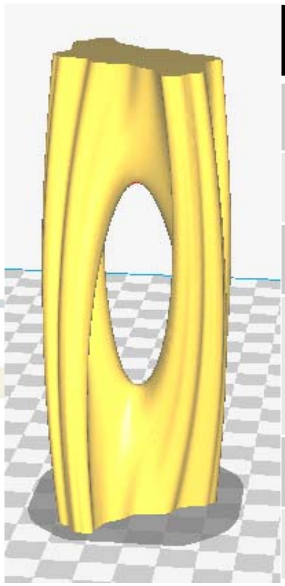


Reviewed model

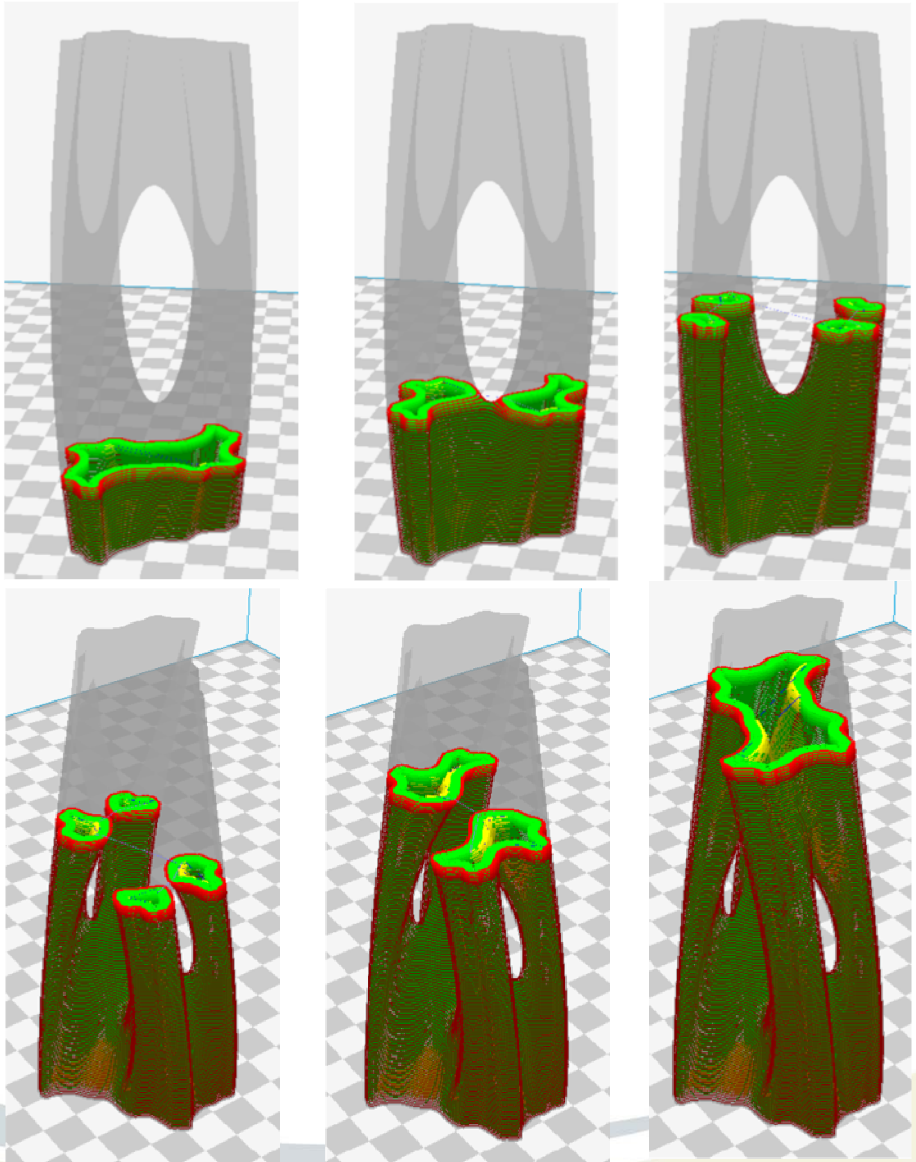


Printing of a decorative piece of 10x10x40cm

2nd step: Slicing / Print programming

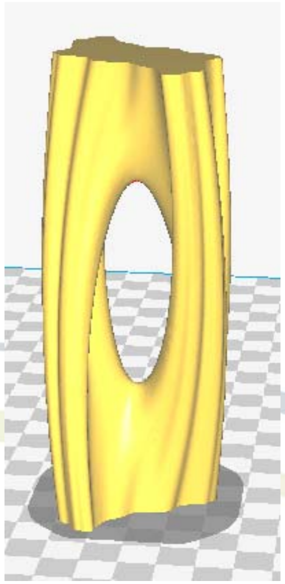


Cura Parameter	
Nozzle size	0,8 mm
Layer thickness	0,7 mm
Printing speed	50 mm/s
Wall thickness	3,2 mm [4 contornos]
Bottom/Top thickness	4 mm
Fill density	0%



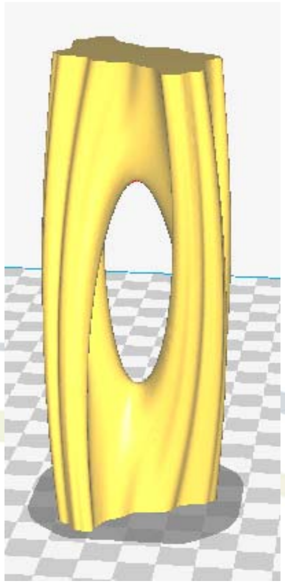
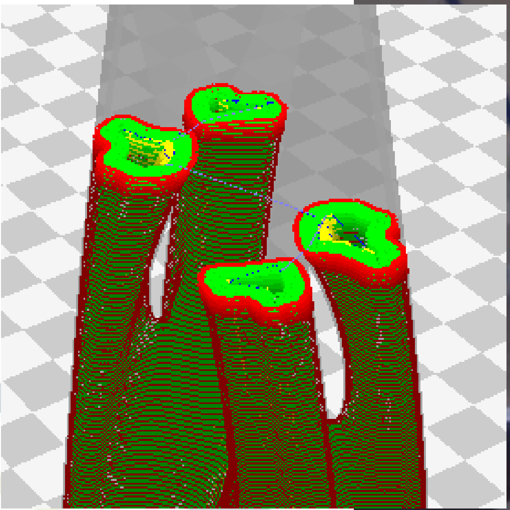
Printing of a decorative piece of 10x10x40cm

3th step: Printing



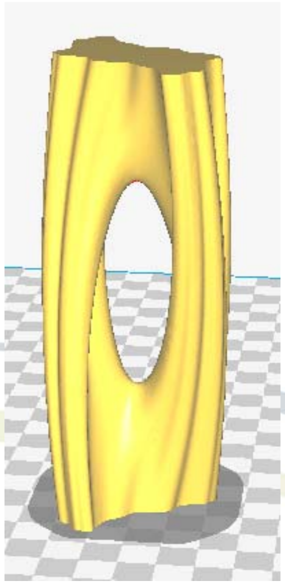
Printing of a decorative piece of 10x10x40cm

3th step: Printing

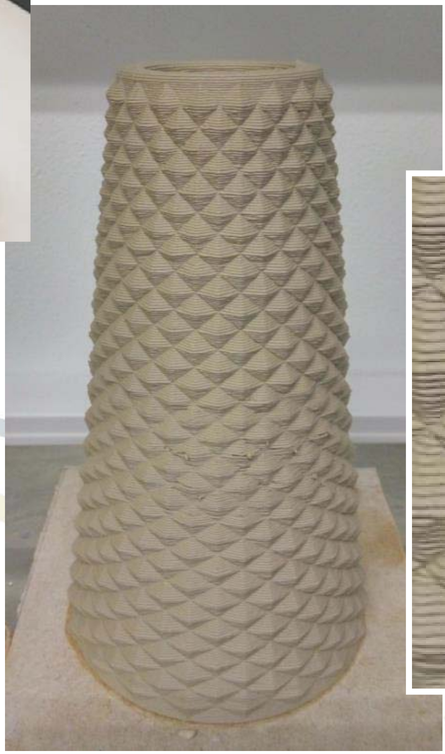

Printing of a decorative piece of 10x10x40cm

Glazed part




Examples of parts produced by Robocasting

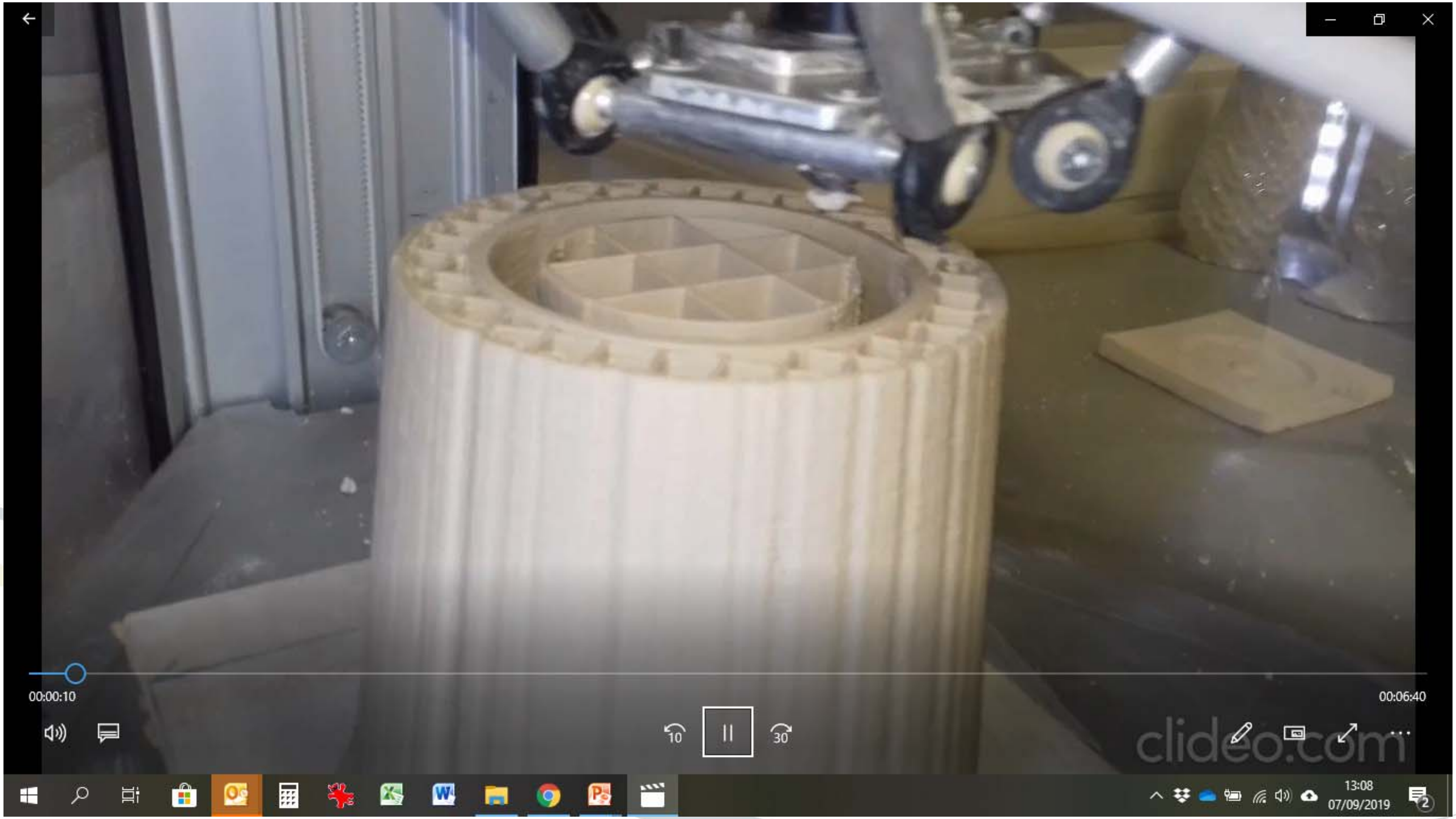
Green parts



Glazed parts

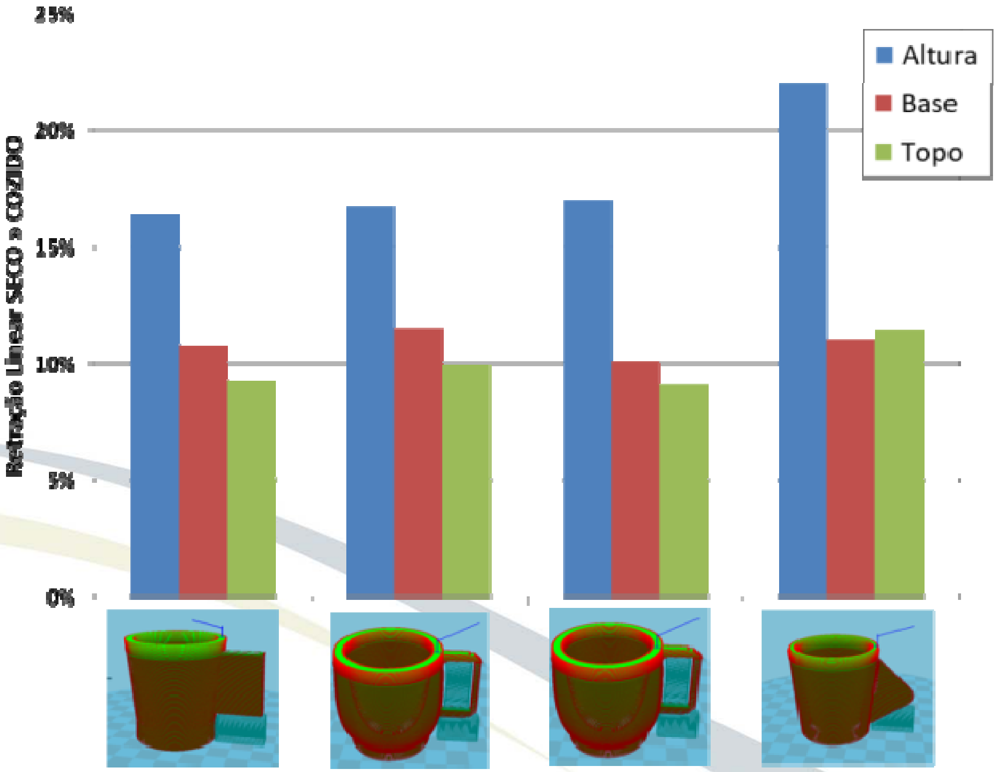
Fired parts

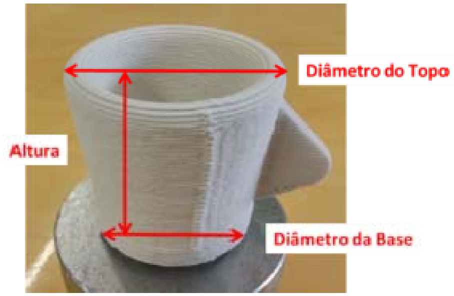
Printing



Technological parameters

Sintering shrinkage: effect of building orientation (ZZ vs XY)





- Shrinkage in ZZ higher than in XY
    - ZZ: 16-22%
    - XY: 9-11%

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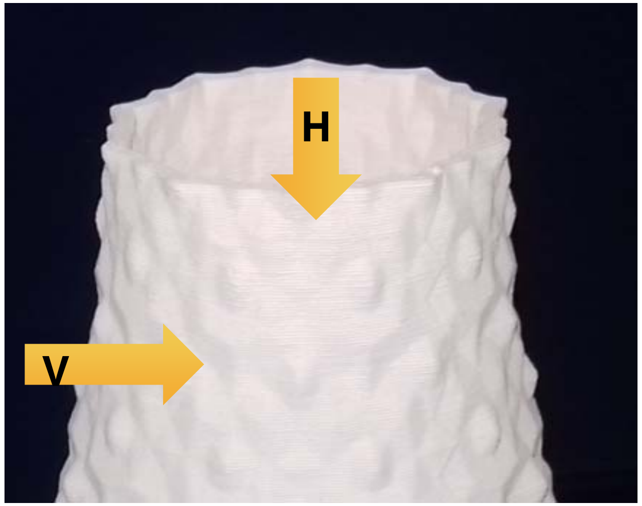
Projeto: RoboCer3D
Parceiros:: costaverde
universidade de aveiro
CTCV materiais
Cofinanciado por: COMPETE 2020 PORTUGAL 2020 UNIÃO EUROPEIA Fundo Europeu de Desenvolvimento Regional
Cofinanciado por: CENTRO 2020 PORTUGAL 2020 UNIÃO EUROPEIA

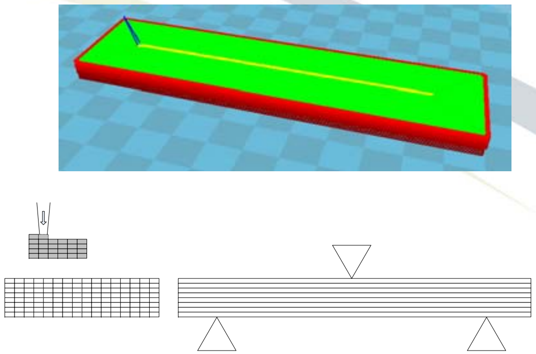
Technological parameters

Densification and mechanical strength: comparasion with extrusion process and the influence of building direction

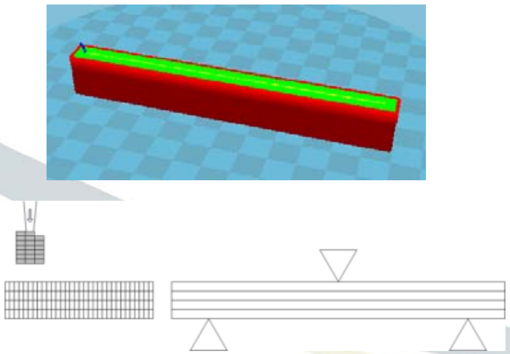
	Robocasting		Extrusion
	Printed in H position	Printed in V position	
Water absorption (wt%)	0,14 ± 0,07	0,25 ± 0,05	0,16 ± 0,04
Apparent density (g/cm^3)	2,38 ± 0,01	2,36 ± 0,01	2,38 ± 0,01
Flexure strength (MPa)	58 ± 13	69 ± 10	71 ± 5



H position



V position



- Low variation in WA and density
- Difference of 15% in FS between the material printed in 2 positions
- Resistência do material impresso na posição V similar à referência

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Processing of alumina suspensions by robocasting

MSc Thesis of Filipa Lopes

Supervised by Prof. Luísa Durães* and Hélio Jorge**

* CIEPQPF. Department of Chemical Engineering. University of Coimbra. Portugal

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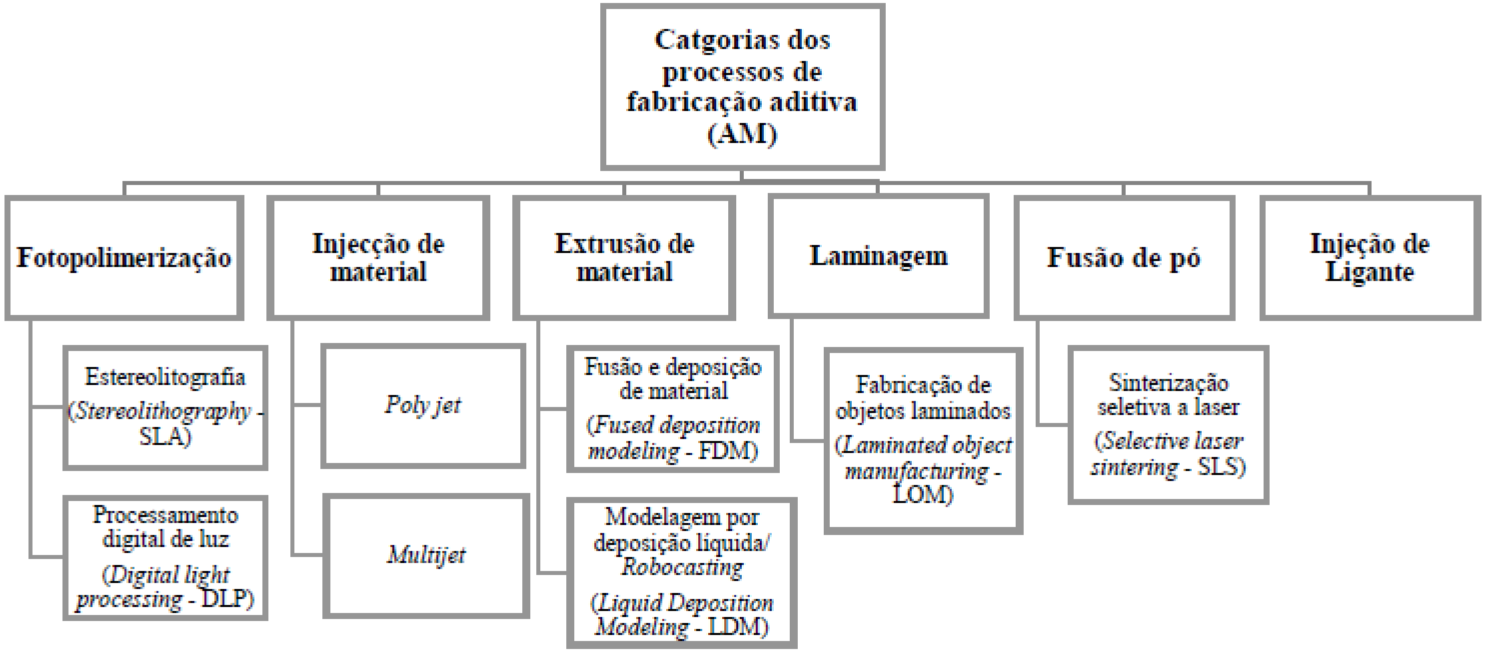
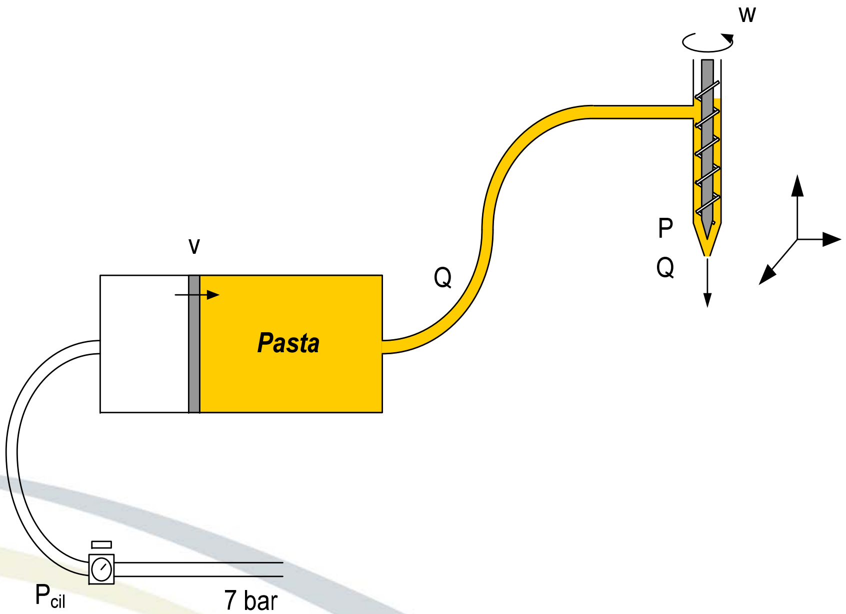
Robocasting system used



- Photopolimerization based methods
  - high final shape precision
  - small size parts
  - high densification materials
  - but available at a high investment cost

- Robocasting / LDM
  - medium shape precision or pre-forms
  - higher size parts
  - Expected to accessed with low/medium investment cost

Robocasting system used



Phase 1 – Assessment of the print ability of ceramic formulations

MATERIALS

- Alumina 99.8%, D50 = 0.4 μm [Almatis CT 3000 SG]
- 2 formulation systems:
  - Sacarose, Polyvinyl alcohol (PVA), Oleic acid (OA) & water - “S” System
  - com. plasticizer (Zusoplast C92), com. lubricant (Zusoplast 126/3), sacarose & water – “Z” System

PROCEDURE

- Ceramic formulations made by hand kneading
  - Demineralized water
  - Solid additives in solution
  - Liquid additives
  - Solids content was set a maximum value defined by manual perception

Formulations

Formulation	Sacarose /%(w/w)	PVA /%(w/w)	Oleic acid /%(w/w)	Solids loading / % (vol/vol)
S_PVA_OA_1	6.8	0.3	3.3	50.9
S_PVA_OA_2	7.4	0.1	1.2	43.2
S_PVA_OA_3	7.8	0.4	1.3	46.3
S_PVA_OA_4	9.7	0.2	3.7	48.8
S_PVA_OA_5	9.7	0.4	1.6	45.7
S_PVA_OA_6	9.8	0.6	2.2	45.8
S_PVA_OA_7	9.9	0.2	2.0	48.2
S_PVA_OA_8	9.9	0.4	2.0	48.1
S_PVA_OA_9	9.9	0.4	2.2	47.6
S_PVA_OA_9+	9.9	0.4	2.2	53.0
S_PVA_OA_10	11.5	0.2	3.9	49.9

Formulation	*Zusoplast C92* /%(w/w)	*Zusoplast 126/3* /%(w/w)	Sacarose /%(w/w)	Citric acid /%(w/w)	Solids loading / % (vol/vol)
P	0.4	-	-	-	39.1
P_L_1	0.4	0.1	-	-	47.1
P_L_2	0.4	0.3	-	-	49.5
P_L_3	1.5	0.3	-	-	49.2
P_L_4	1.9	0.3	-	-	42.4
P_L_S_1	0.1	0.1	7.6	-	48.0
P_L_S_2	0.4	0.1	7.6	-	48.4
P_L_S_3	0.4	0.1	9.8	-	45.0
P_L_S_4	0.4	0.3	0.4	-	49.9
P_L_S_5	0.4	0.3	7.5	-	46.7
P_I[illegible]	0.1	0.7	4.9	0.1	45.6
P_L[illegible]	0.1	0.7	5.1	0.3	46.3

Assessment of formulations

Formulation behaviour assessment:

- Plasticity
- Tacking
- Stiffness
- Robocasting trails

Cofinanciado por:

Assessment of formulations

Formulation behaviour assessment:

- **Plasticity**
- Tacking
- Stiffness
- Robocasting trails

Plasticity category table

Classification	Description	Example
●○○○ Non compacted	Not possible to have an aggregated solid mass	
●●●○○○ Breaking	Aggregated solid mass, but not possible to mould a roll without breaking	
●●●●●○ Cracking	Roll moulded, but it cracks when bending.	
●●●●●● Bending	Homogenous roll that bends without cracking	

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Assessment of formulations

Formulation behaviour assessment:

- Plasticity
- **Tacking**
- Stiffness
- Robocasting trails

Tacking category table

Category	Description	Image
●○○○○ Slightly tacky	It sticks slightly but it can be handle and easy to mould.	
●●●●○○ Tacky	Even sticking to the table and gloves, it is able to be moulded	
●●●●●● Highly tacky	The material sticks to the table and gloves, and cannot be shaped	

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I1	Não percebo o que queres dizer na última parte desta frase luisa; 09/09/2019

Assessment of formulations

Formulation behaviour assessment:

- Plasticity
- Tacking
- **Stiffness**
- Robocasting trails

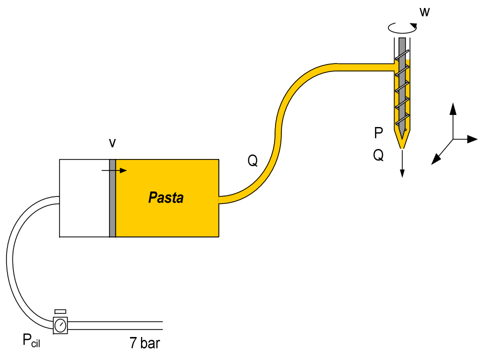
Stiffness category table

Category	Description
●○○○○○	Low stiffness
●●○○○○	Low-Medium stiff
●●●●○○	Medium-high stiff
●●●●●●	Stiff

Assessment of formulations

Formulation behaviour assessment:

- Plasticity
- Tacking
- Stiffness
- **Robocasting trials**



Printing performance evaluation topics:

- Constant flow through the extrusion nozzle
- First layer sticking of the extrudate to the building platform
- Layer by layer sticking
- Side extrudate sticking
- Structural integrity of the building form

Good part => good formulation behaviour

Assessment results

Formulation	Sacarose /%(w/w)	PVA /%(w/w)	Oleic acid /%(w/w)	Solids loading / % (vol/vol)	Plasticity	Tacking	Stiffness	Printing tested?
S_PVA_OA_1	6.8	0.3	3.3	50.9	●●○○○○	○○○○○○	○○○○○○	
S_PVA_OA_2	7.4	0.1	1.2	43.2	○○○○○○	○○○○○○	○○○○○○	
S_PVA_OA_3	7.8	0.4	1.3	46.3	●●●○○○	○○○○○○	●●●●○○	
S_PVA_OA_4	9.7	0.2	3.7	48.8	●●●○○○	○○○○○○	○○○○○○	Yes
S_PVA_OA_5	9.7	0.4	1.6	45.7	●○○○○○	○○○○○○	○○○○○○	
S_PVA_OA_6	9.8	0.6	2.2	45.8	●●●●○○	○○○○○○	●●●●○○	
S_PVA_OA_7	9.9	0.2	2.0	48.2	●●●●○○	○○○○○○	●○○○○○	Yes
S_PVA_OA_8	9.9	0.4	2.0	48.1	●●●●●○	●○○○○○	○○○○○○	Yes
S_PVA_OA_9	9.9	0.4	2.2	47.6	●●●●○○	○○○○○○	●○○○○○	
S_PVA_OA_9+	9.9	0.4	2.2	53.0	●●●●●○	●○○○○○	●●○○○○	
S_PVA_OA_10	11.5	0.2	3.9	49.9	●○○○○○	○○○○○○	○○○○○○	

Formulation	*Zusoplast C92* /%(w/w)	*Zusoplast 126/3* /%(w/w)	Sacarose /%(w/w)	Citric acid /%(w/w)	Solids loading / % (vol/vol)	Plasticity	Tacking	Stiffness	Printing tested?
P	0.4	-	-	-	39.1	●●●●●●	●●●●●●	○○○○○○	Yes
P_L_1	0.4	0.1	-	-	47.1	●●●●●●	○○○○○○	●●●●●●	
P_L_2	0.4	0.3	-	-	49.5	●●●●●●	●●●○○○	●●○○○○	
P_L_3	1.5	0.3	-	-	49.2	●●●●○○	●○○○○○	●●●●●●	
P_L_4	1.9	0.3	-	-	42.4	●●●●●●	●●●●●●	○○○○○○	
P_L_S_1	0.1	0.1	7.6	-	48.0	●●●●●●	●●●○○○	●●○○○○	
P_L_S_2	0.4	0.1	7.6	-	48.4	●●●●●●	○○○○○○	●●●●●○	
P_L_S_3	0.4	0.1	9.8	-	45.0	●●●●●○	●●●●●●	●●●○○○	
P_L_S_4	0.4	0.3	0.4	-	49.9	●●●●○○	○○○○○○	●●●●○○	
P_L_S_5	0.4	0.3	7.5	-	46.7	●●●●●●	●●●●○○	●●●○○○	
P_[illegible]	0.1	0.7	4.9	0.1	45.6	●●●●○○	●●●○○○	[illegible]	[illegible]
P_L_S_AC_2	0.1	0.7	5.1	0.3	46.3	●●●●●●	●●●○○○	●●○○○○	Yes

Phase 2 – Robocasting and characterization

Plastic body compounding → Printing → Debinding and Sintering

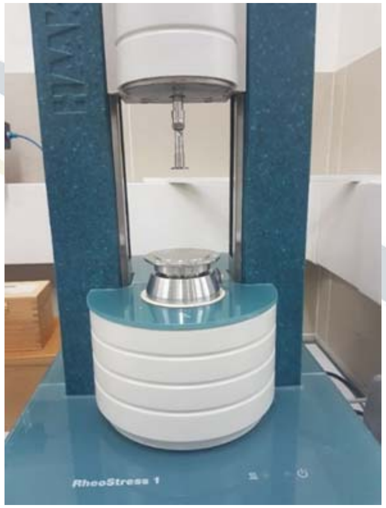
Formulation	Alumina size, D50 /µm	Sacarose /%(w/w)	PVA /%(w/w)	Oleic acid /%(w/w)	Solids loading / % (vol/vol)
S_PVA_OA_9	0.4	9.9	0.4	2.2	47.6
S_PVA_OA_9+	4	9.9	0.4	2.2	53.0

Formulation	Alumina size, D50 /µm	*Zusoplast C92* /%(w/w)	*Zusoplast 126/3* /%(w/w)	Sacarose /%(w/w)	Citric acid /%(w/w)	Solids loading / % (vol/vol)
P_L_S_5	0.4	0.4	0.3	7.5	-	46.7
P_L_S_AC_1	0.4	0.1	0.7	4.9	0.1	45.6

Phase 2 – Robocasting and characterization

Plastic body compounding → Printing → Debinding and Sintering

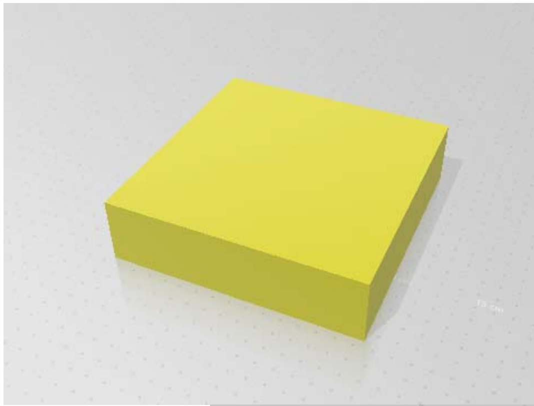
- Rheology — Rotational shear rheometry with a Haake Rheotress 1
- Hardness — Indentation force measurement with a Geotester Penetrometer

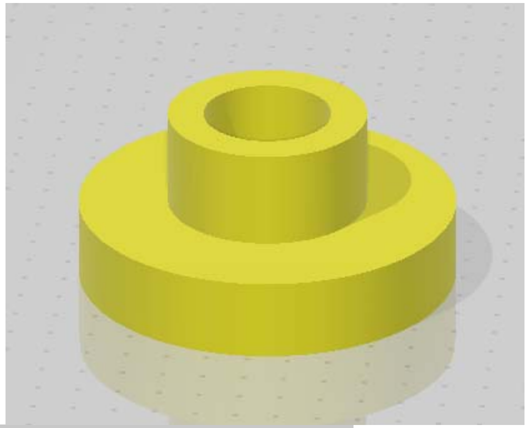


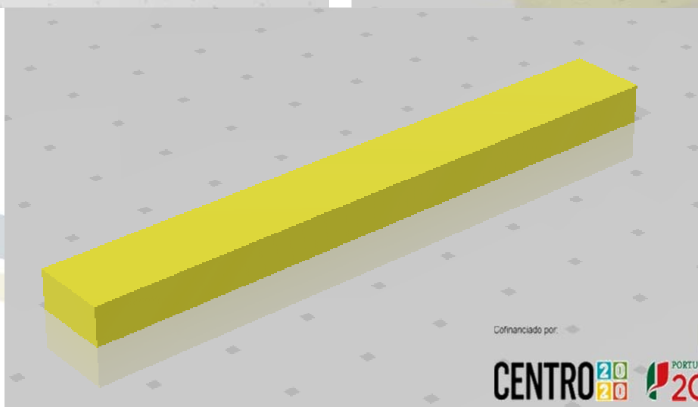
Phase 2 – Robocasting and characterization

Plastic body compounding → Printing → Debinding and Sintering

Cura Parameters	
Version	V2.3.1
Nozzle diameter / mm	1
Layer thickness /mm	0,7
First layer thickness /mm	0,5
Wall printing speed /mm.s^{-1}	30
Fill printing speed /mm.s^{-1}	30
Bottom and top printing speed /mm.s^{-1}	30
Travel speed /mm.s^{-1}	60
Flow /%	100
Wall thcikness /mm	3
Wall line counts/ -	3
Bottom and top thickness /mm	3
Fill density /%	100







Phase 2 – Robocasting and characterization

Plastic body compounding → Printing → Debinding and Sintering

- Porosity — Arquimedes principle
- Flexure strength — 3-point flexure testing

Rheology of the formulations

Rheometer procedure issues faced calling for patience work:

- High normal force before reach the gap programmed (stiff formulations)
- Slip-stick behaviour suspected (high filled suspensions)
- Sample spreading

Test conditions: Parallel plates geometry R=10mm; Gap h=0.5mm

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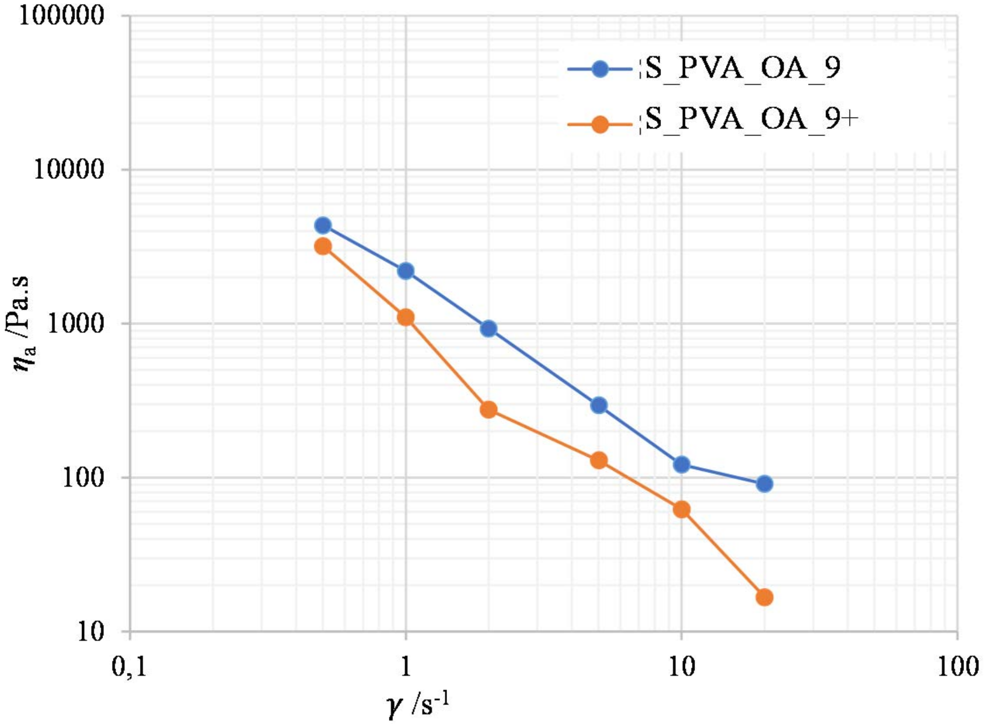
Rheology of the formulations

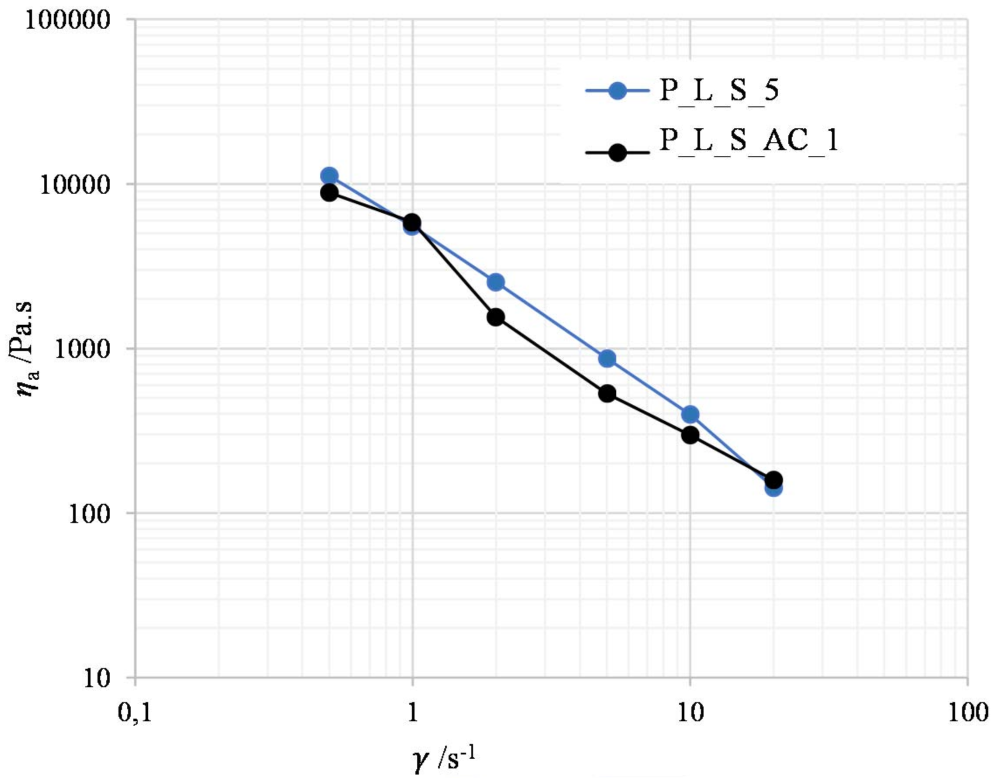


Formulation	Powder D50 /µm	Sacarose /%(w/w)	PVA /%(w/w)	Oleic acid /%(w/w)	Solids loading / % (vol/vol)
S_PVA_OA_9	0.4	9.9	0.4	2.2	47.6
S_PVA_OA_9+	4	9.9	0.4	2.2	53.0

- Pseudoplastic behaviour of both formulations
- Formulation with higher particle size shows lower viscosity, despite having a higher solids loading

Test conditions: Parallel plates geometry R=10mm; Gap h=0.5mm

Rheology of the formulations



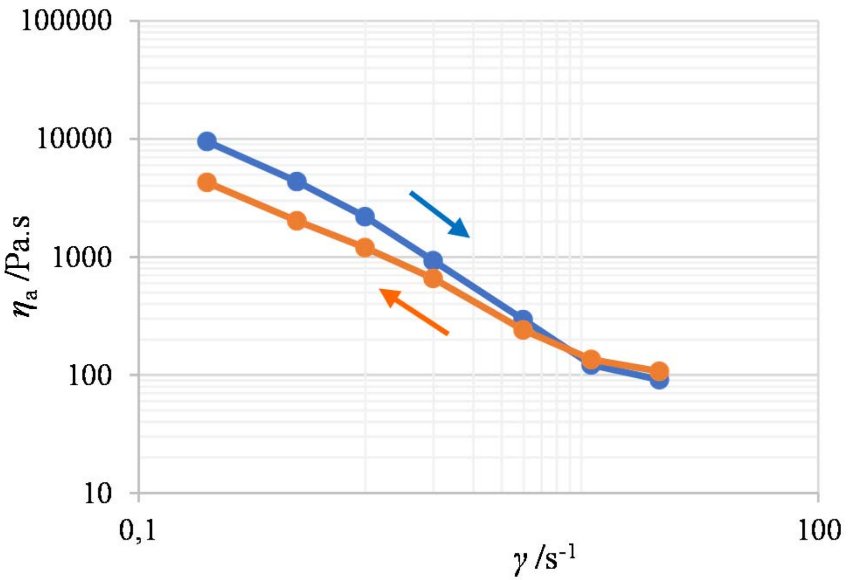
Formulation	Powder D50 /µm	*C92* /%(w/w)	*126/3* /%(w/w)	Sacarose /%(w/w)	Citric acid /%(w/w)	Solids loading / % (vol/vol)
P_L_S_5	0.4	0.4	0.3	7.5	-	46.7
P_L_S_AC_1	0.4	0.1	0.7	4.9	0.1	45.6

- Pseudoplastic behaviour of both formulations
- Slightly difference between viscosity of the two formulations

Test conditions: Parallel plates geometry R=10mm; Gap h=0.5mm

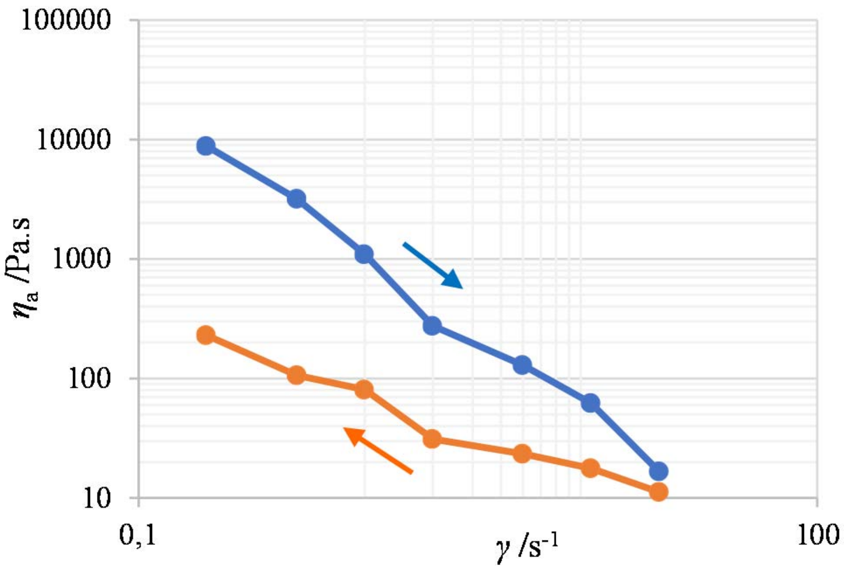
Rheology of the formulations

S_PVA_OA_9



- Thixotropic behaviour

S_PVA_OA_9+

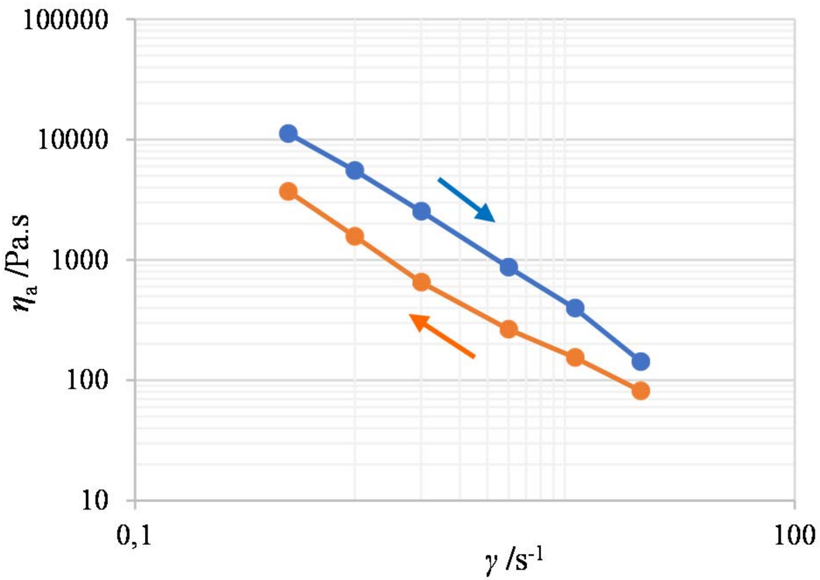


- Thixotropic behaviour

Formulation	Powder D50 /µm	Sacarose /%(w/w)	PVA /%(w/w)	Oleic acid /%(w/w)	Solids loading / % (vol/vol)
S_PVA_OA_9	0.4	9.9	0.4	2.2	47.6
S_PVA_OA_9+	4	9.9	0.4	2.2	53.0

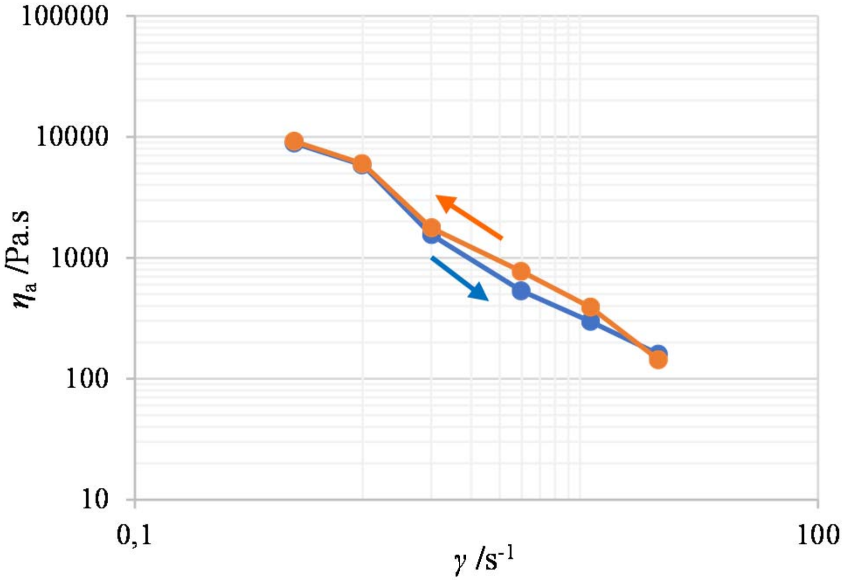
Rheology of the formulations

P_L_S_5



- Thixotropic behaviour

P_L_S_AC_1



- Non-thixotropic behaviour

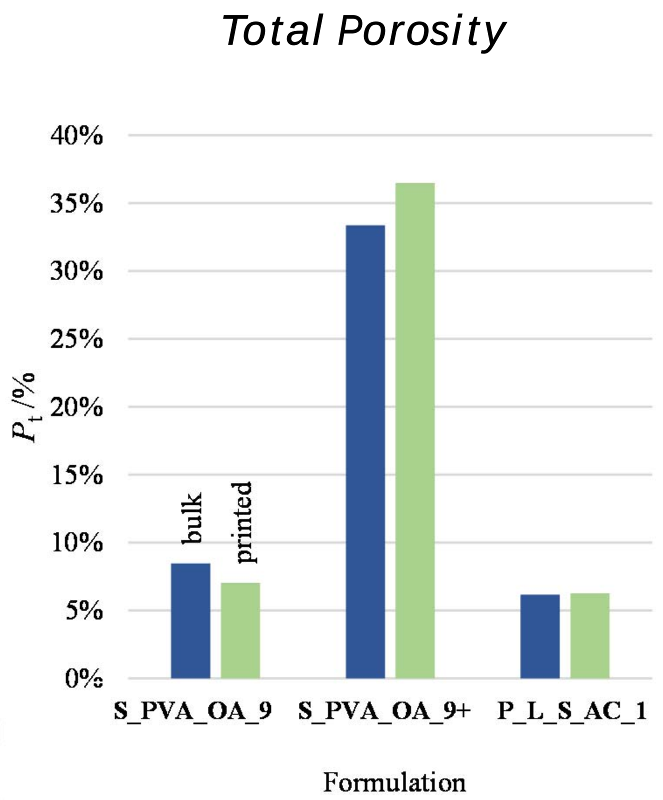
Formulation	Powder D50 /µm	*C92* /%(w/w)	*126/3* /%(w/w)	Sacarose /%(w/w)	Citric acid /%(w/w)	Solids loading / % (vol/vol)
P_L_S_5	0.4	0.4	0.3	7.5	-	46.7
P_L_S_AC_1	0.4	0.1	0.7	4.9	0.1	45.6

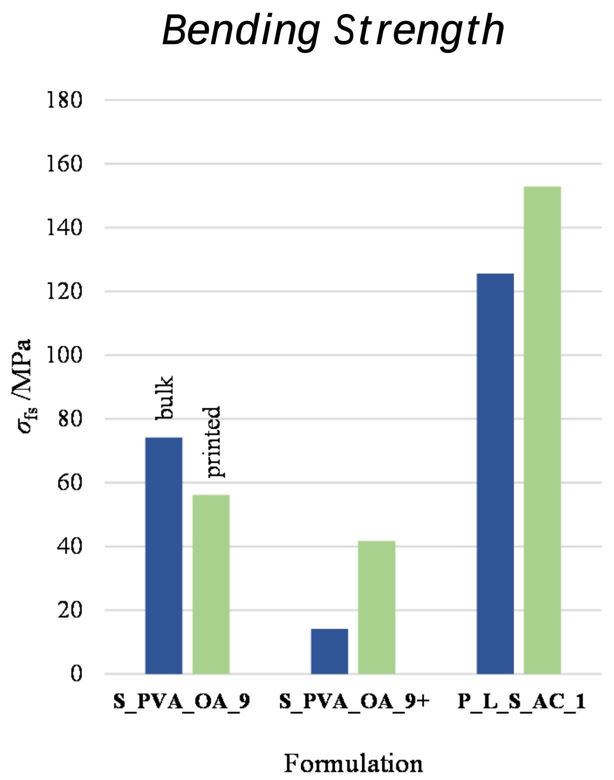
Rheology and hardness of the formulations

Formulation	Hardness /kg	Viscosity in 0,5-20 s^{-1} range /Pa.s	Printing behaviour
S_PVA_OA_9	1,8	91 to 4349	Poor adhesion to the building platform
S_PVA_OA_9+	< 1	17 to 3187	Prints well
P_L_S_5	-	159 to 11195	-
P_L_S_AC_1	2,9	159 to 8870	Prints with upper limit reservoir pressure

- Formulation hardness is proportional to viscosity
- Formulations with hardness higher than ca. 3 kg are not recommended for processing

Sintered porosity and bending strength





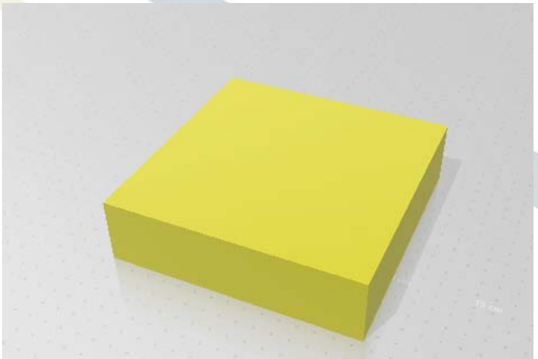
- Higher porosity (33 & 37%) in formulation based on higher powder particle size (D50 = 4μm)
- Higher strength in sintered formulations with lower porosity

Cofinanciado por:

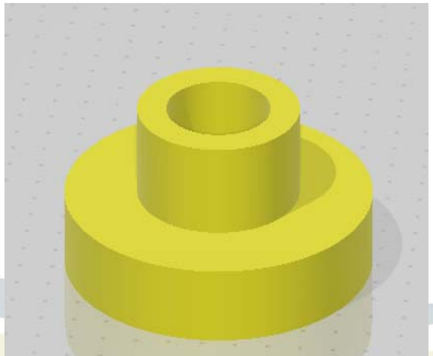
Prototype production

Formulation S_PVA_OA_9+

Formulation P_L_S_AC_1



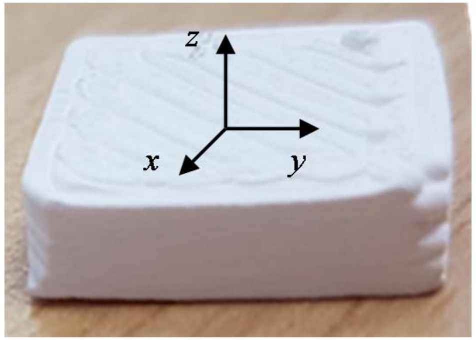
Green: 15x15x4mm



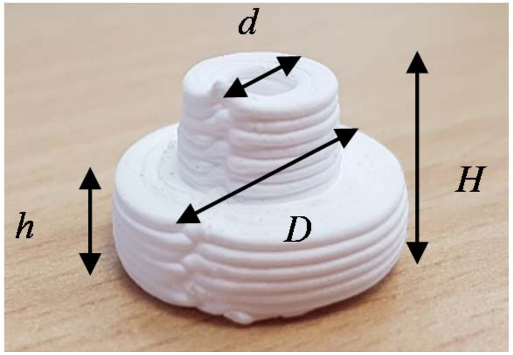
Green: 16x16x10mm

Sintering linear shrinkage

Plate



Spacer



Formulation	Plate			*Spacer*			
	$R_{L,x}$ /%	$R_{L,y}$ /%	$R_{L,z}$ /%	$R_{L,D}$ /%	$R_{L,d}$ /%	$R_{L,H}$ /%	$R_{L,h}$ /%
S_P_OA_9	17,7	18,3	17,6	18,4	17,3	20,2	19,3
S_P_OA_9+	4,8	4,6	6,0	3,8	3,9	6,8	7,4
P_L_S_AC_1	18,	18,55	25,5	18,2	16,4	21,8	18,9

- Higher shrinkage in formulations with lower particle size and high densification (S_P_OA_9 and P_L_S_AC_1)
- Higher shrinkage in Z direction

Application prototyping

Formulation S_PVA_OA_9 (alumina D=0.4μm)

printing a gas dosing valve disc

Application prototyping

Green parts:

- Injection moulding (left)
- Robocasting (right)

- Green parts (top)
- Sintered parts (bottom)

Conclusion

- Comparing to AM of plastics and metals, ceramics are in the very beginning but with a huge potential

- Technology and application should be developed together

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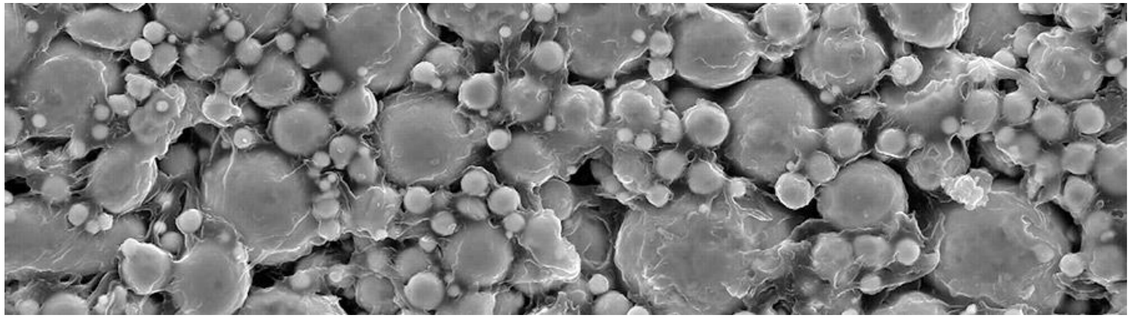
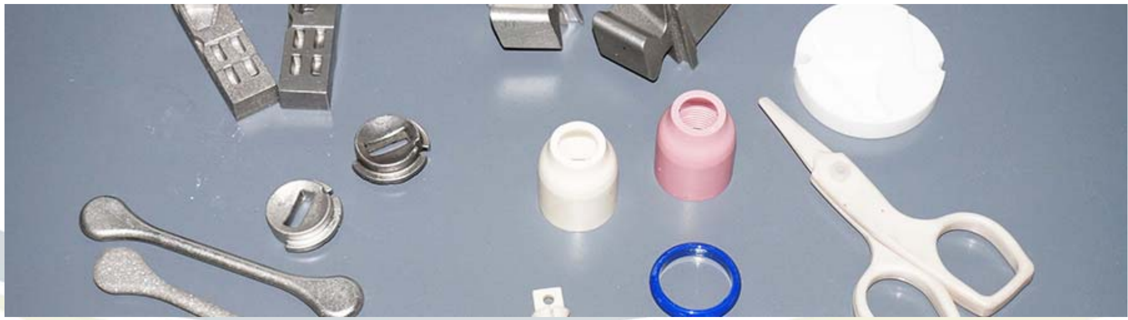
CTCV materiais

Thank you





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