



Manufacture of porous cordierite samples using gelcasting of polysaccharides

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INTRODUCTION

	Cell Size / nm	<i>PROCESSING - Shaping</i>
Microporous	<2	Sol-Gel, Sacrificial Templates
Mesoporous	2-50	Sol-Gel, Sacrificial Templates
Macroporous	>50	Replica, Sacrificial Templates, Freeze Drying, <u>DIRECT FOAMING</u>

Requirements: a “gel” to fix the structure

INTRODUCTION

Cordierite, $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$

Thermal shock resistance material



APPLICATIONS

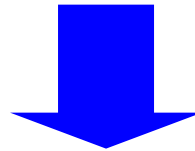
Honeycomb catalyst supports in automobiles
Thermal Insulation
Kiln furniture
Filters

SYNTHESIS

High temperature reaction in solid state
Chemical methods { **Coprecipitation reactions**
Solution combustion
Sol-gel

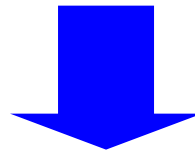
OBJECTIVE

Cordierite Materials with Tailored Porosity



Colloidal Processing

Direct Foaming



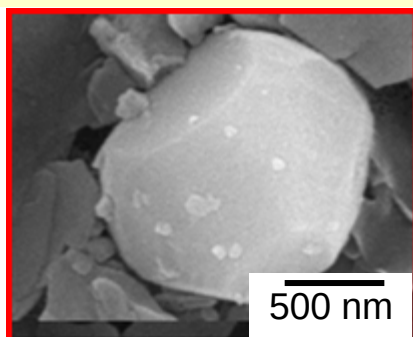
Gelcasting of polysaccharides

EXPERIMENTAL

Cordierite, $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$

molar ratio: $\text{MgO}, \text{Al}_2\text{O}_3, \text{SiO}_2$
22.2 / 22.2 / 55.6
weight ratio: 13.8 / 34.9 / 51.3

Alumina, 16.2 wt%

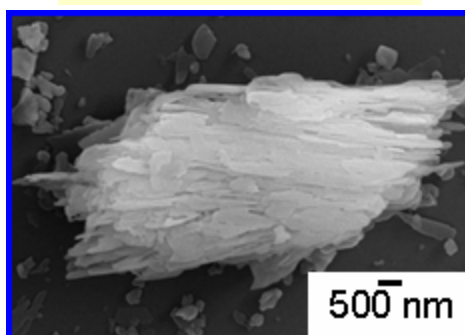


ALCOA, USA

1000SG	CL3000FG
S_s 10.2 m ² /g	S_s 0.7 m ² /g
$\bar{d}_{50}$ 0.6 μm	$\bar{d}_{50}$ 5.7 μm
ρ 3.98 g/cm ³	ρ 3.98 g/cm ³

100:0, 85:15, 15:85

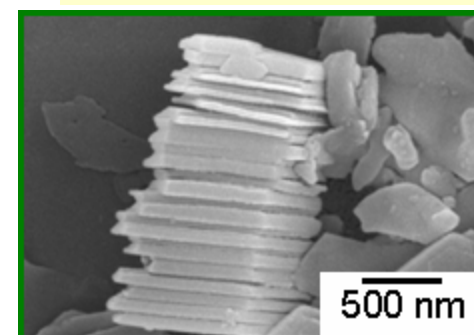
Talc, 43.8 wt%



S_s 20.6 m²/g
 ρ 2.76 g/cm³

Colorminas S.A., Criciúma, Brazil

Kaolin, 40.0 wt%

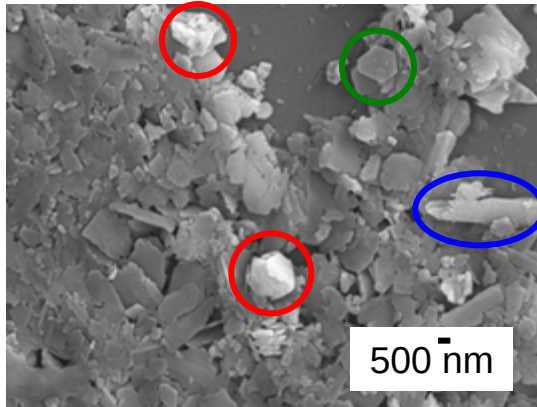


S_s 9.5 m²/g
 ρ 2.58 g/cm³

Raw Materials



EXPERIMENTAL



Raw Materials

Water, % Dolapix PCN (Zschimmer-Schwarz, Germany), pH, fine and coarse alumina, ageing...

Suspension

Slip casting
Sintering

Residual Porosity

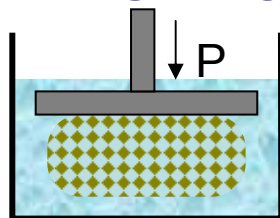


Milling
(t planetary mill, 15-45 min)

Slip casting
Sintering

Dense

SPONGE
IMPREGNATION



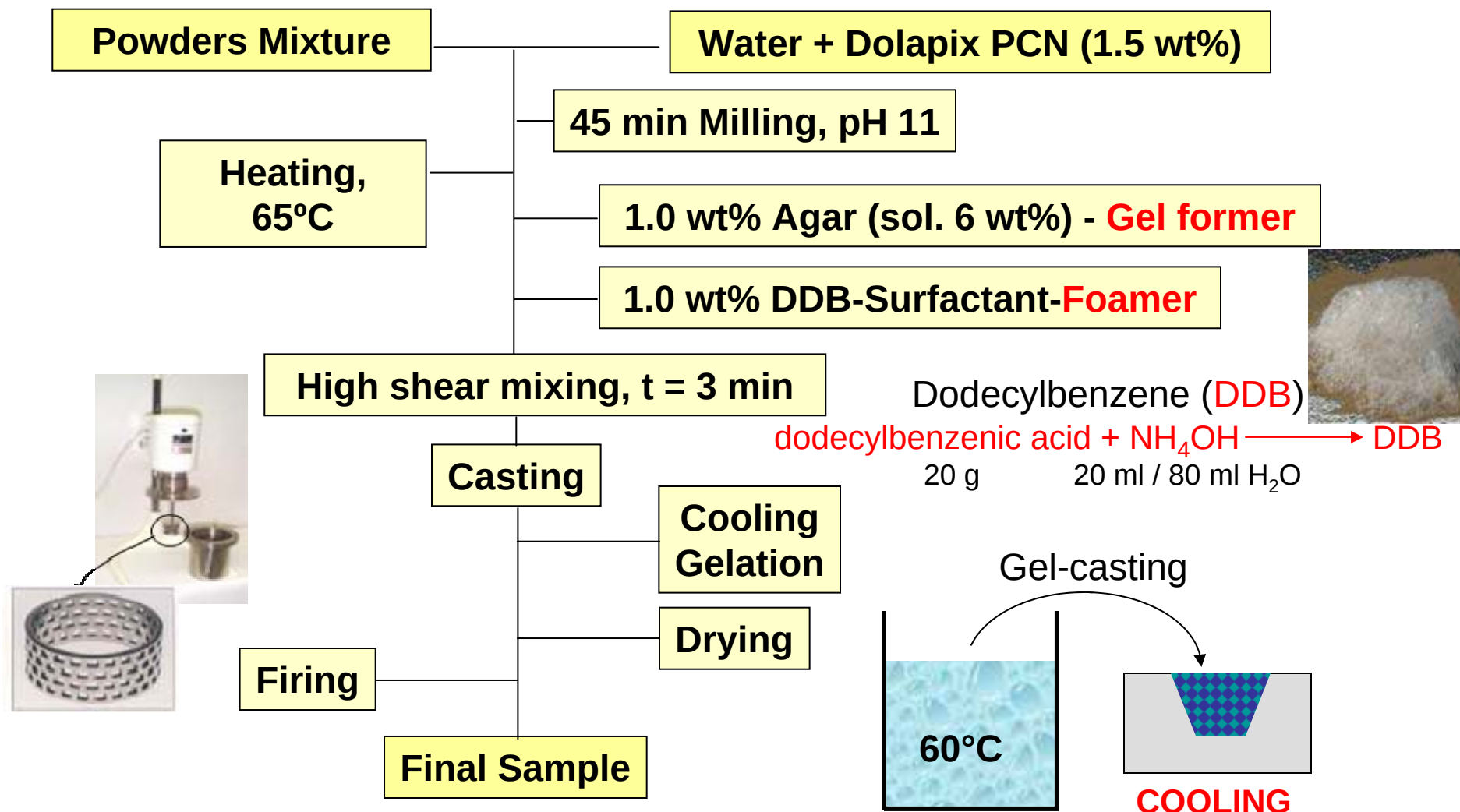
Replica
Sintering

Direct Foaming /
Polysaccharides
Sintering

Porous samples

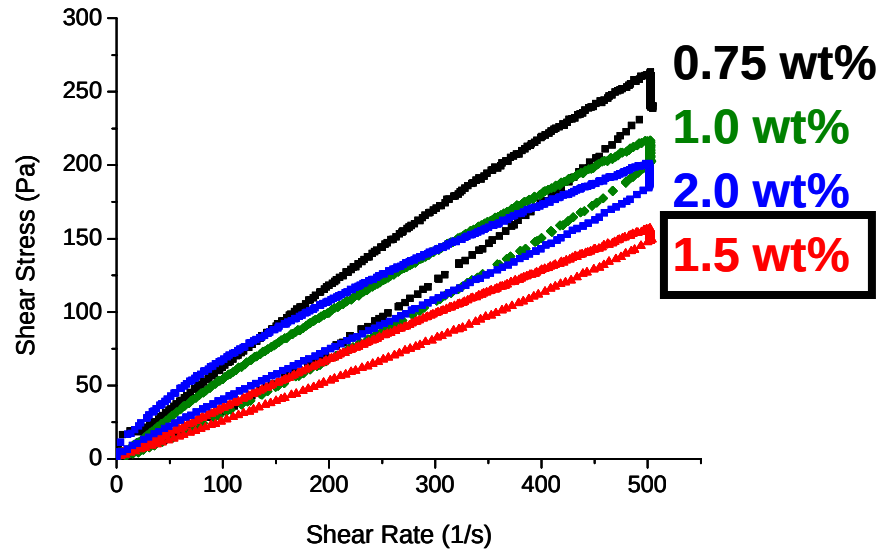
EXPERIMENTAL

Direct Foaming (Polysaccharides)

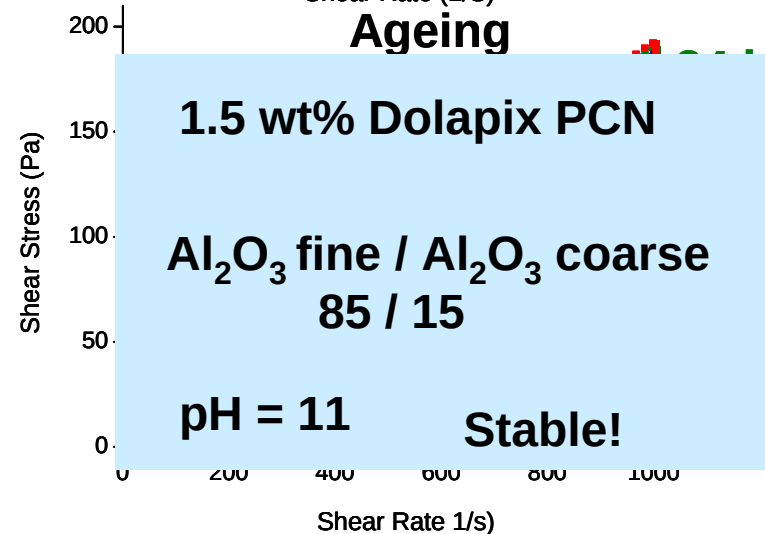
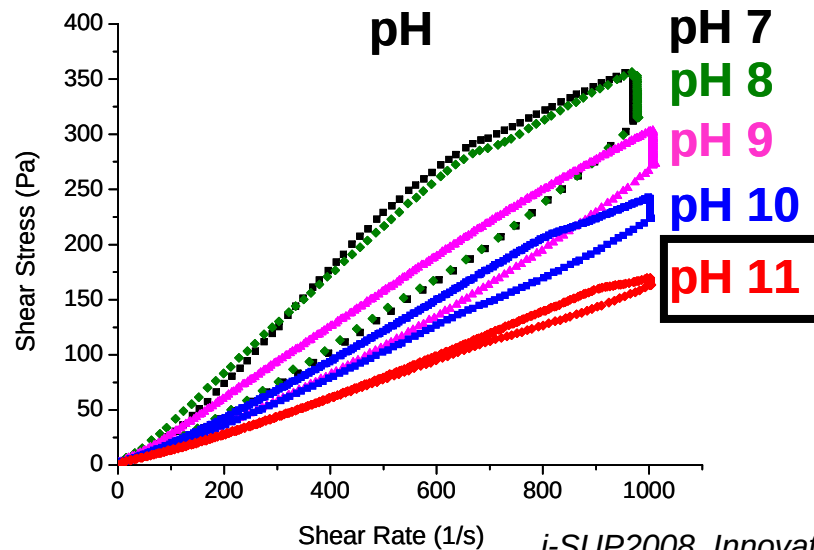
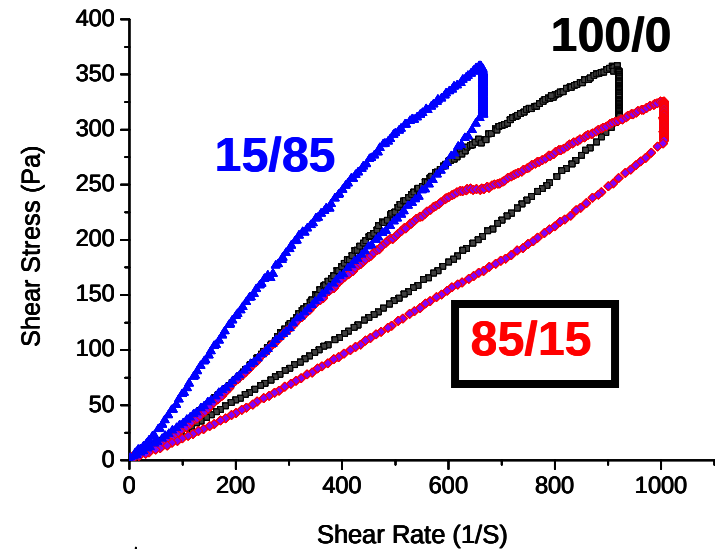


SUSPENSION, 40 vol%

Dolapix content

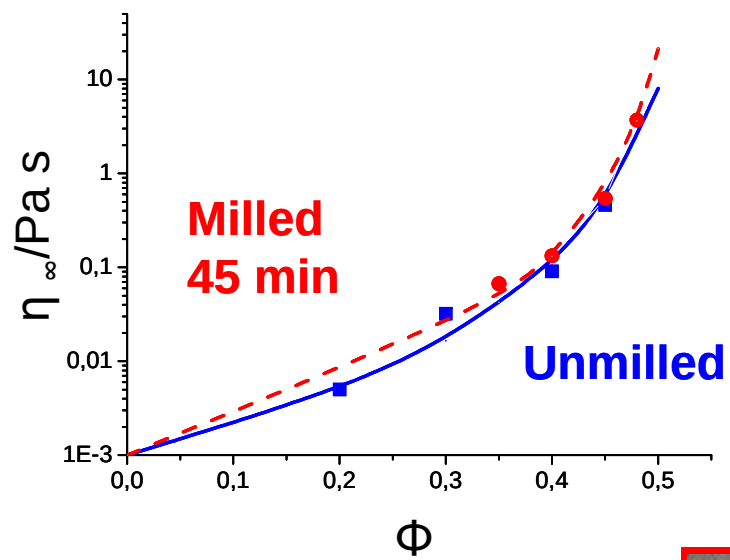


Al₂O₃: fine (0.6 μm) / coarse (5.7 μm)

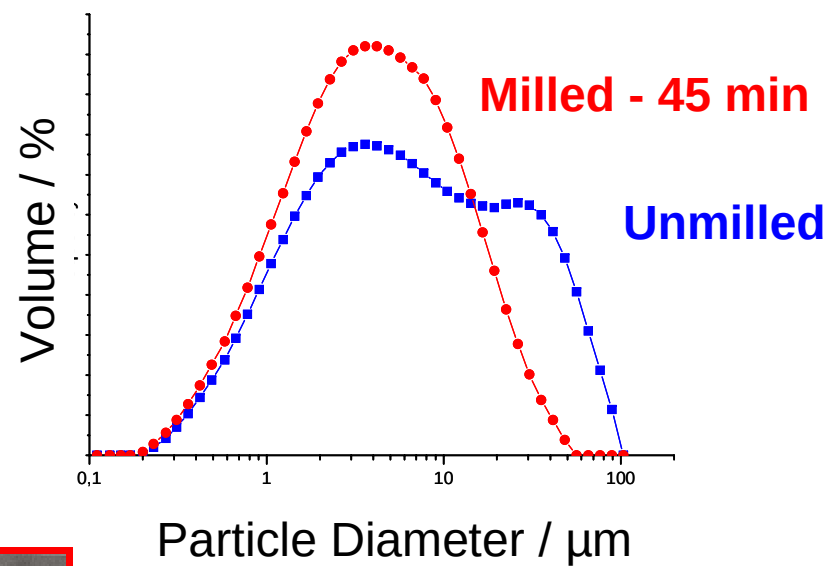


MILLING

Krieger-Dougherty

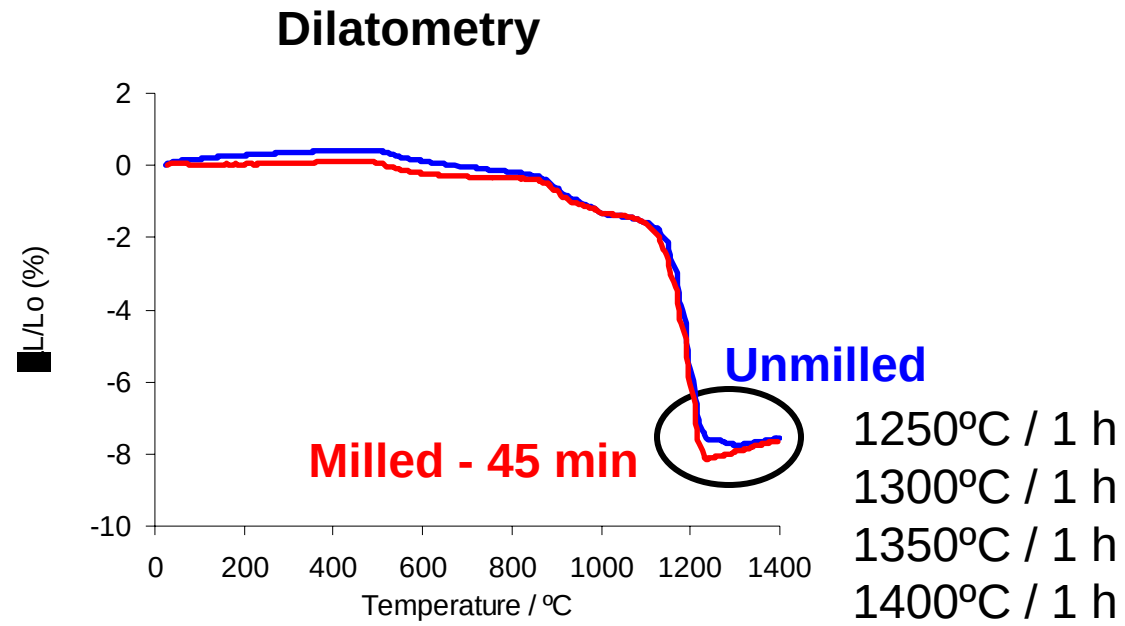


Size distribution

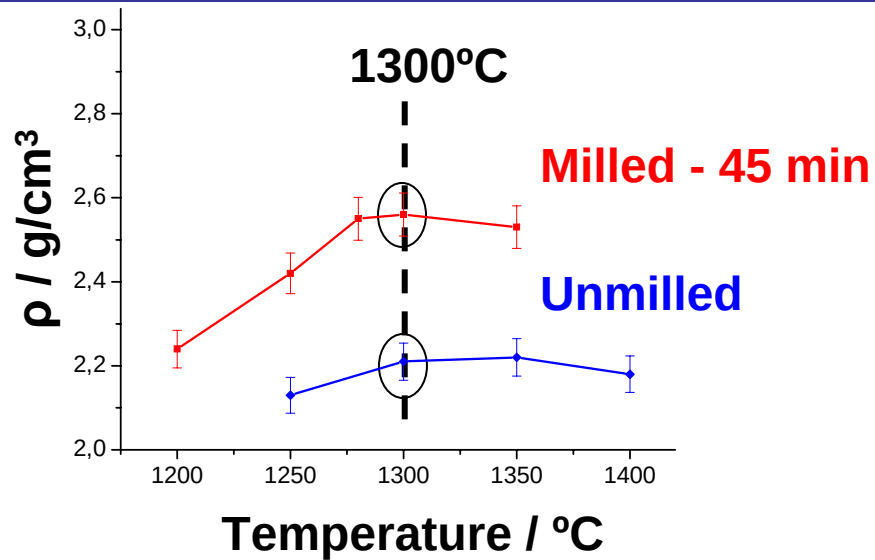


Slip casting

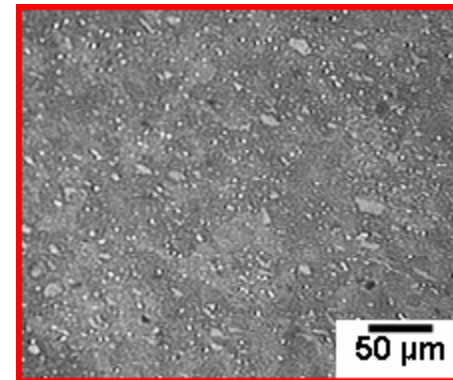
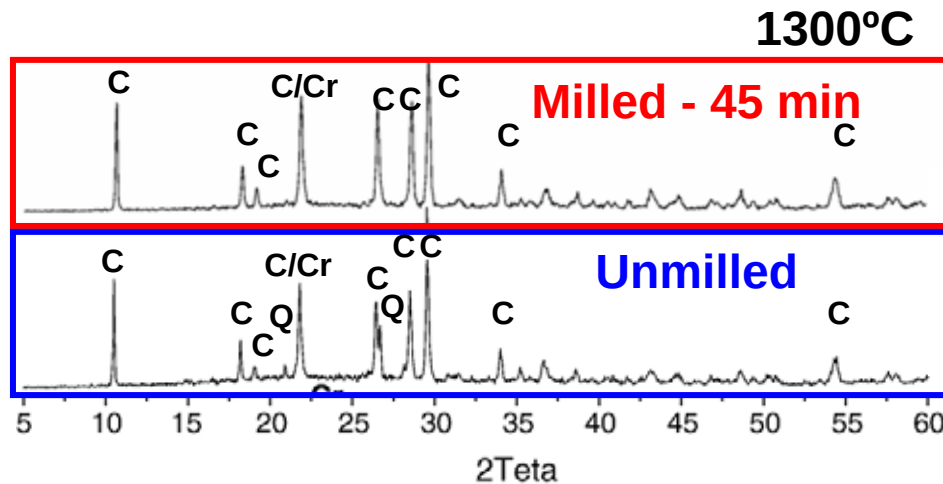
DYNAMIC SINTERING STUDIES



STATIC SINTERING, XRD

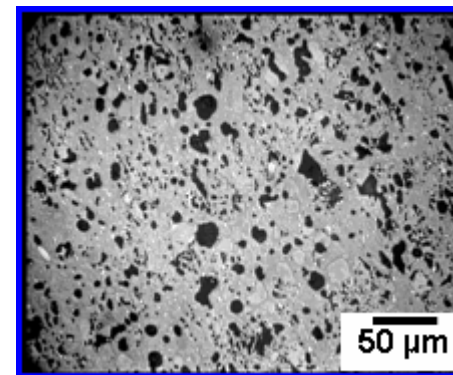


$T < 1300^{\circ}\text{C}$: Cordierite is not yet the major phase



DENSE

2.6 g cm⁻³



RESIDUAL POROSITY

2.2 g cm⁻³

CONTROLLED POROSITY

Dense

Milled - 45 min

Porous samples

Replica

Direct Foaming /
Polysaccharides

Parameters

Solids Loading

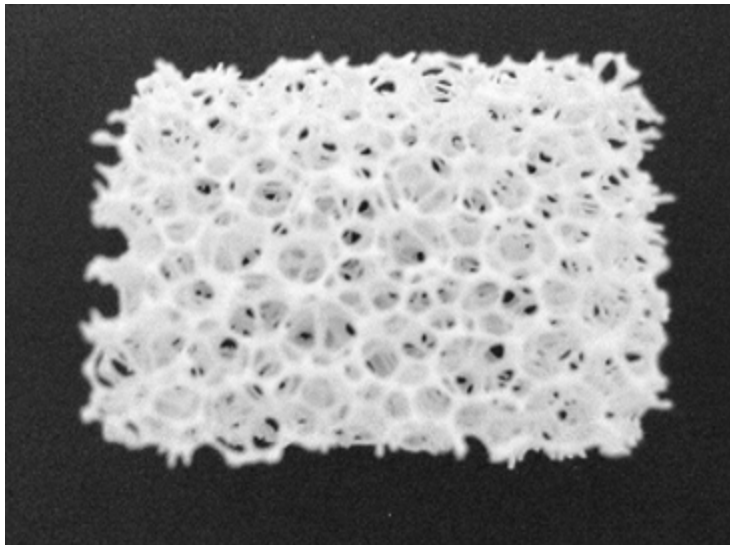
Temperature

Viscosity!!

"Gelling time"

Direct Foaming

Replica



DIRECT FOAMING

Milled Suspension +
+Gelling agent + Foaming agent

Solids Loading

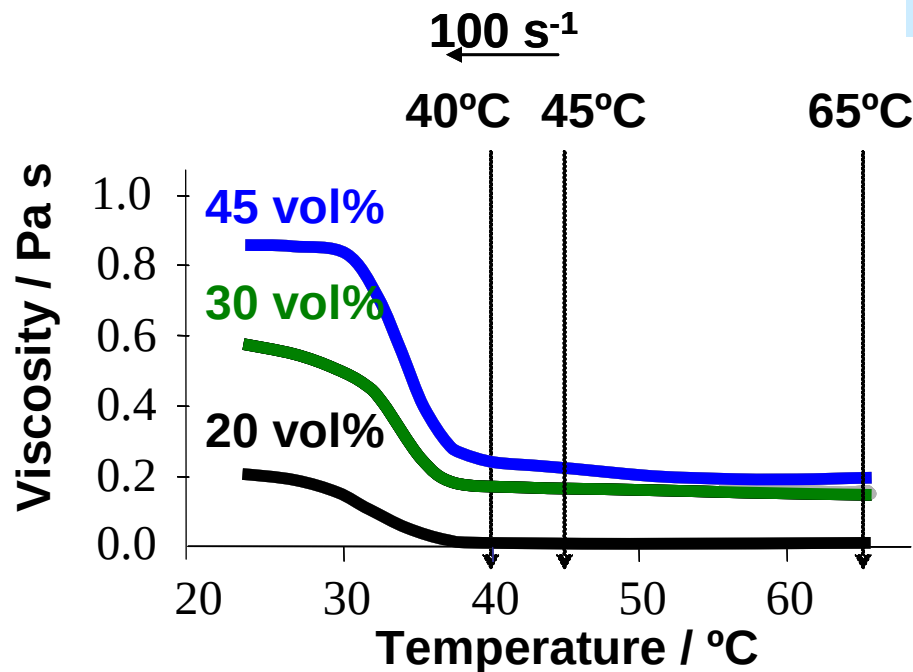
Temperature



Viscosity!!



"Gelling time"



Viscosity / mPa s

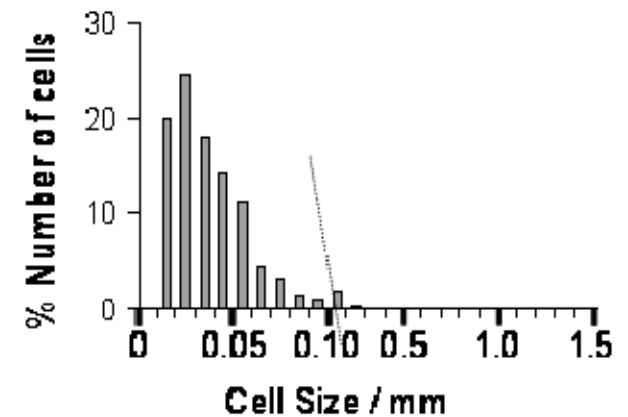
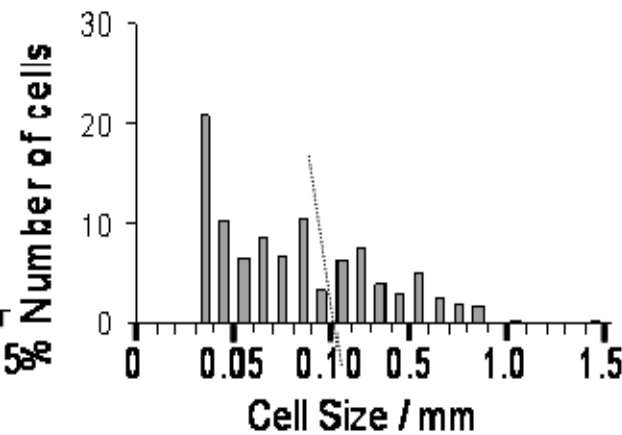
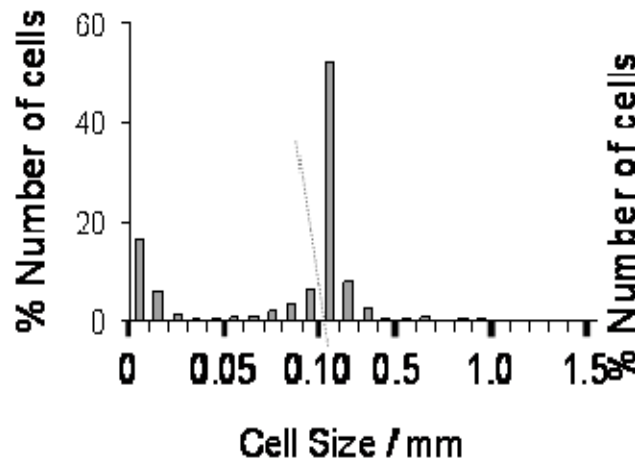
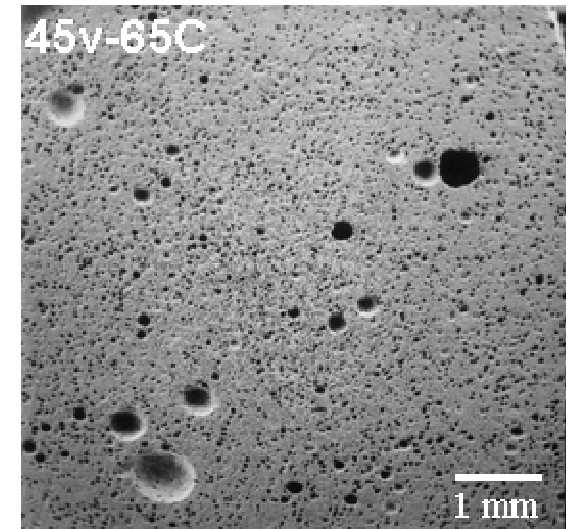
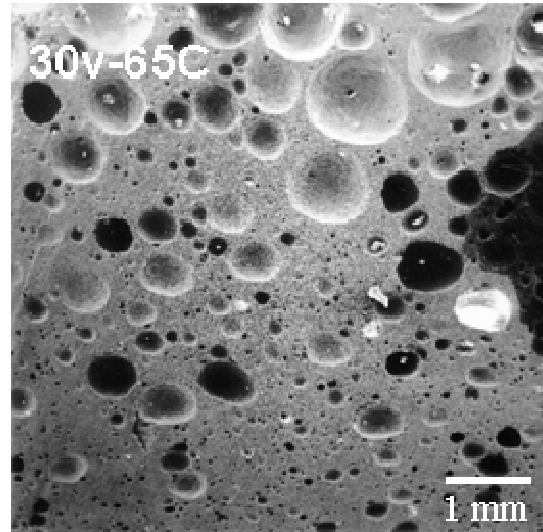
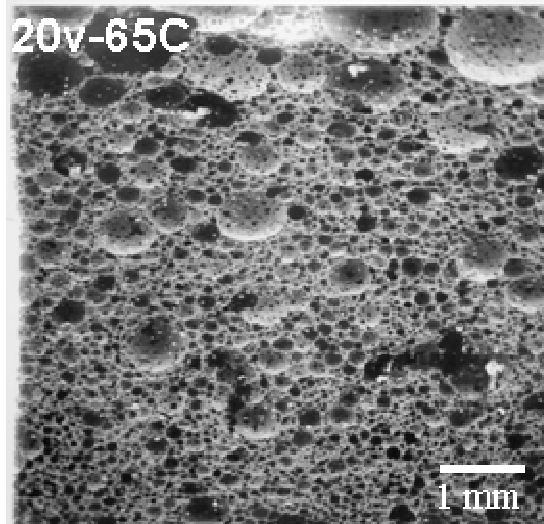
45 vol% 190 - 220

30 vol% 160 - 170

20 vol% 10 - 15

DIRECT FOAMING

T = 65°C



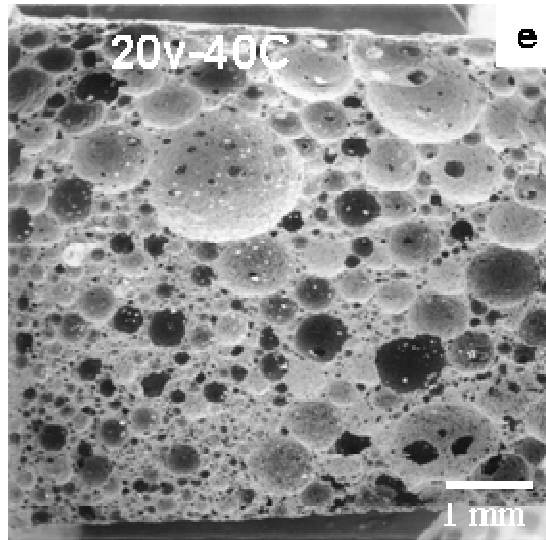
Average cell diameter
125 μm

90 μm

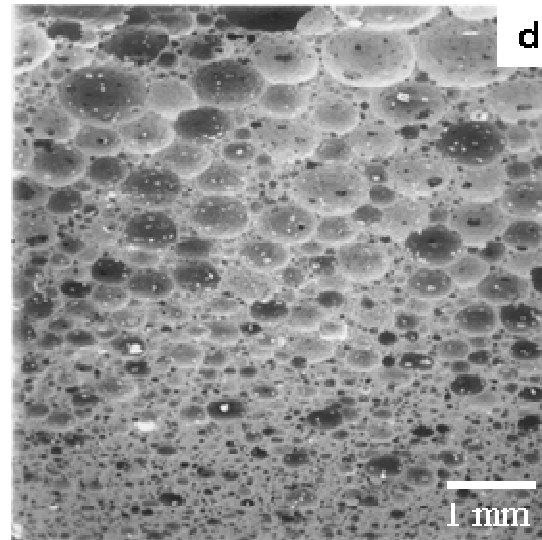
40 μm

DIRECT FOAMING

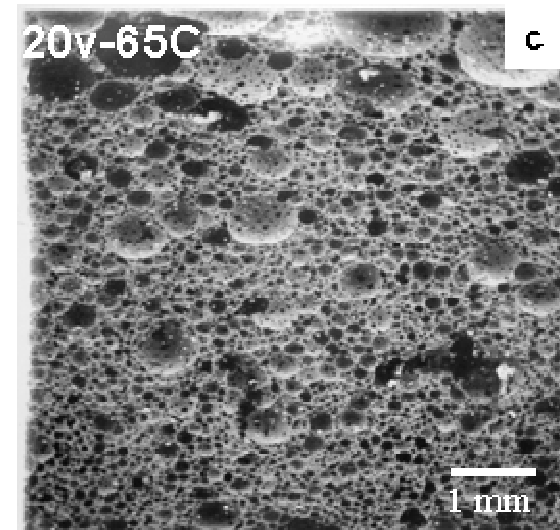
20 vol%



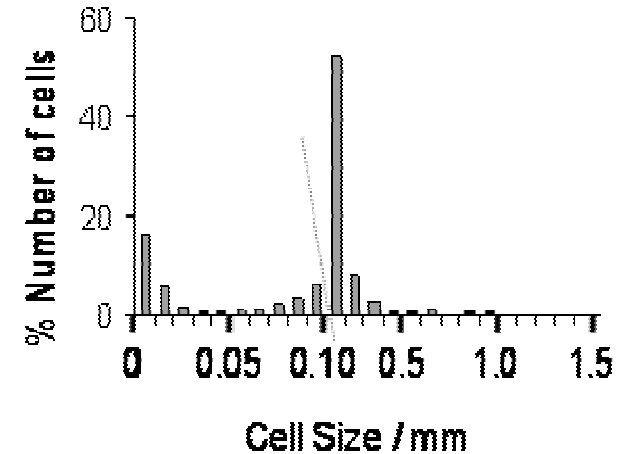
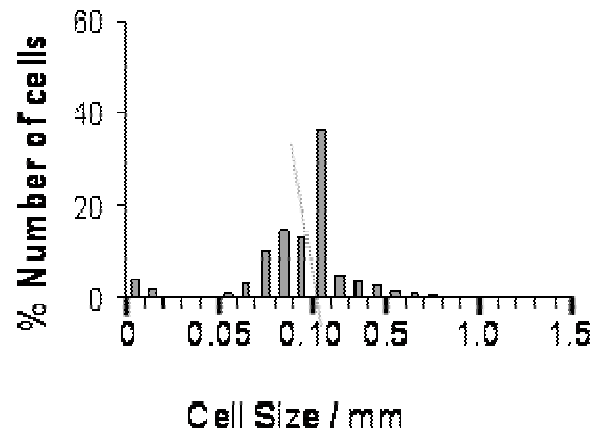
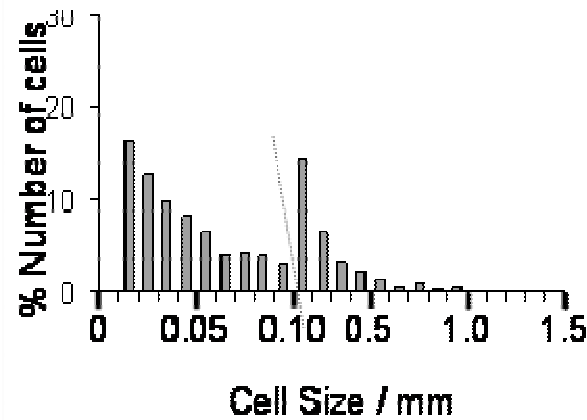
T = 40°C



T = 45°C



T = 65°C



DIRECT FOAMING

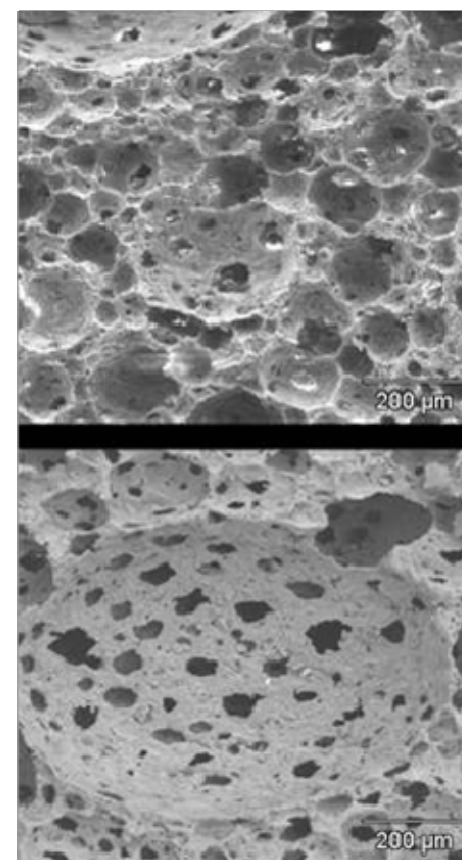
SAMPLE	Viscosity at 100 s ⁻¹ mPa s	Apparent density ±0.1 g cm ⁻³	Open porosity Vol%
20 vol%-40°C	15	0.6	73.9
20 vol%-45°C	13	0.7	74.0
20 vol%-65°C	10	0.6	76.5
30 vol%-40°C	170	1.8	15.1
30 vol%-45°C	162	1.7	18.4
30 vol%-65°C	159	1.5	29.4
45 vol%-45°C	221	1.9	1.8
45 vol%-65°C	186	2.0	1.2

Average cell
diameter

→ 125 µm

→ 90 µm

→ 40 µm



SUMMARY

- Homogeneous aqueous suspensions of kaolin/talc/alumina mixture have been prepared: 1.5 wt% Dolapix PCN, 85/15 fine/coarse aluminas and pH 11. The effect of the milling was studied.
- Sintered unmilled samples showed large remain porosity, but milled ones lead to dense structures due to the activation of the raw materials by milling.
- Cordierite phase only appears as main phase at $T \geq 1300^{\circ}\text{C}$.
- Materials with tailored porosity have been prepared from milled suspensions by sponge impregnation and by Direct Foaming (gelcasting of polysaccharides in combination with a foaming agent).
- The solid loading of the precursor slip is a critical parameter in the manufacture of porous samples by Direct Foaming which is related to the slips viscosity.

***Porous Coatings
Work in elaboration!!***



THANK YOU VERY MUCH FOR YOUR ATTENTION!