Solid loading optimisation of extremely irregular particles

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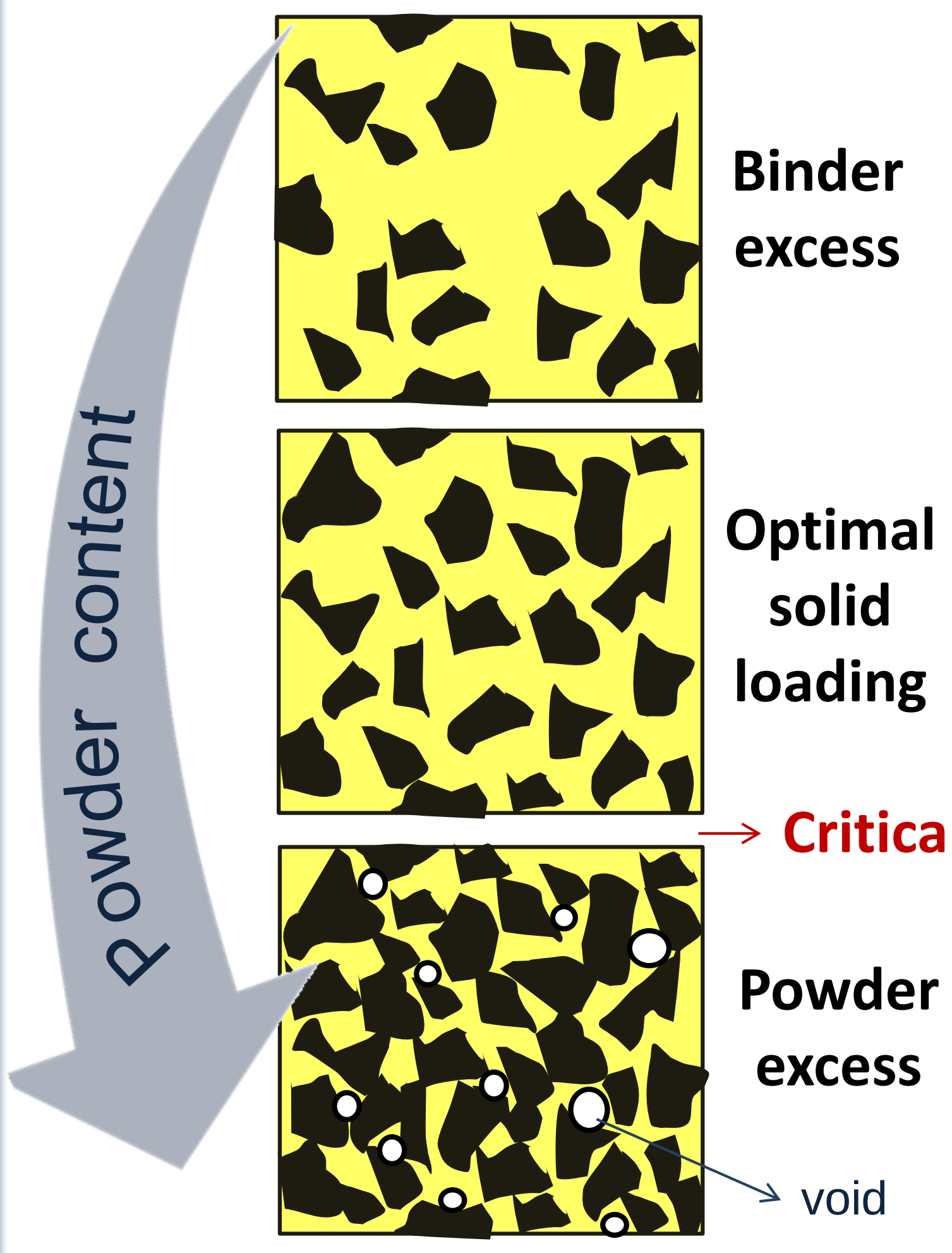
Objectives

1. To develop a PIM process based on an ***eco-friendy*** binder system and raw zircon powders.
2. To optimize the powder content of feedstock.

Introduction

Feedstock design and production become decisive for the success of the powder injection moulding process since following stages are widely influenced by its quality. An ideal feedstock is composed by the optimal balance of mixture of powder and effective binder.



Binder excess

- **Powder-binder separation during moulding** → *flashing* inhomogeneities, density gradients & distortion after sintering
- **Long time debinding cycles** →high risk of defects & *slumping*
- **Considerable shrinkage** → making tolerance control more difficult.

Optimal solid loading

- **Homogeneity**
- **Good rheological properties for injection moulding**
- **Tight dimension tolerance control**

→ **Critical Powder Volume Concentration (CPVC)** 2-5% higher than the optimal one

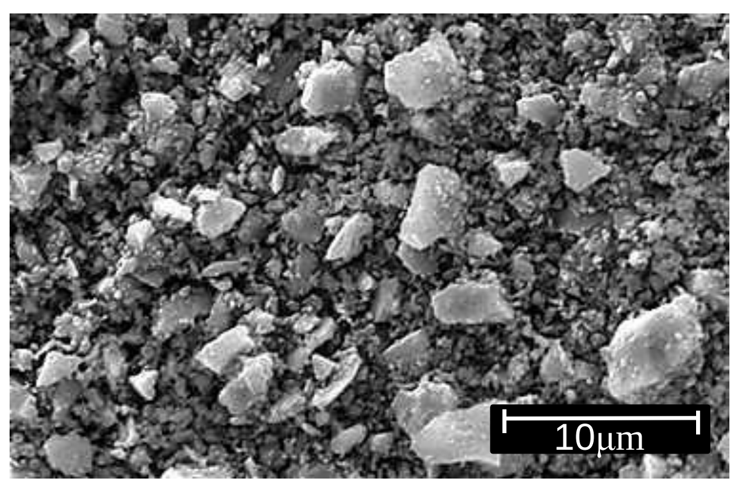
Powder excess

- **High viscosity** → not suitable rheological properties
- **Not enough binder to fill pores between particles**
- **High risk of contamination**

Different methods have been carried out to obtain the optimal solid loading of *zircon* ($ZrSiO_4$)

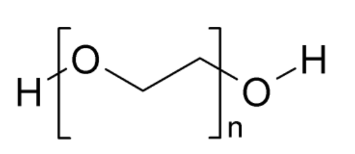
Materials

Powder Properties	Zircon
Morphology	Angular
Density($g·cm^{-3}$)	4.59
App. density (%TD)	19.05%
Tap density (%TD)	32.45%
D_{50}-D_{90}	1.9-5.5 µm

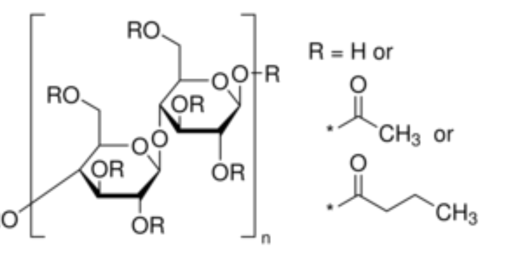


Binder system

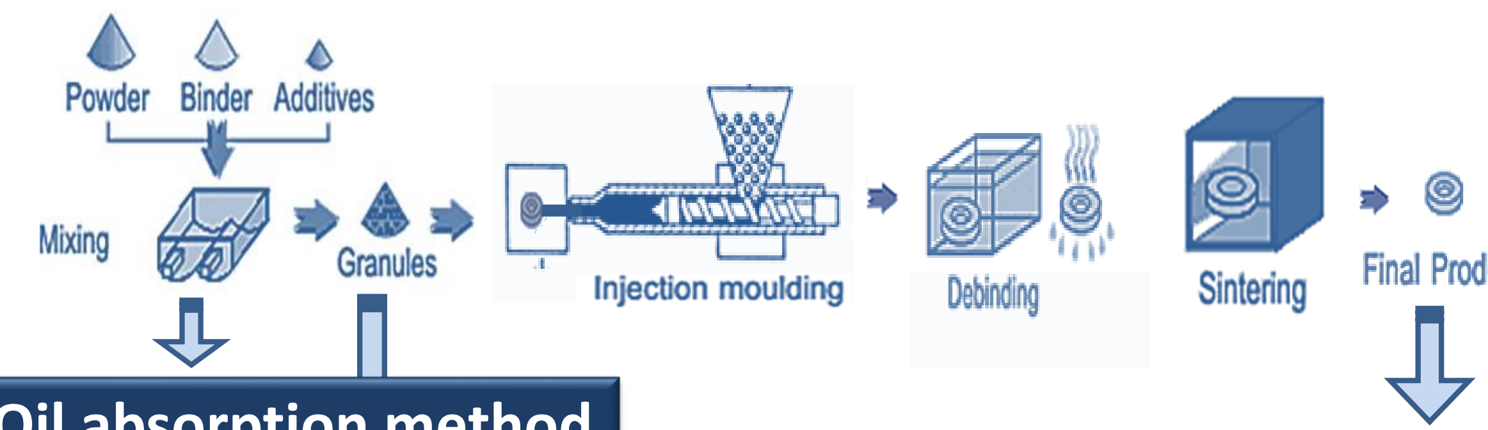
PEG



CAB



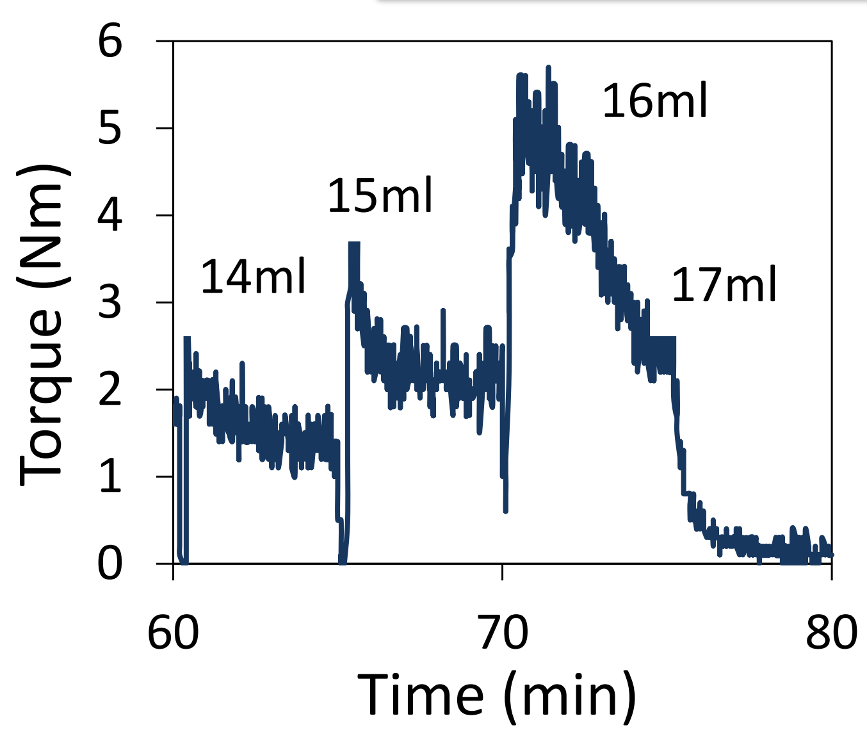
Experimental Procedure



1. Oil absorption method
2. Torque rheometry
3. Feedstock density
4. Rheological study
5. Dimension stability - distortion
6. Relative density
7. Flexural strength – Weibull
8. Fracture surface analysis

Results

1. Oil absorption method ($ZrSiO_4$)

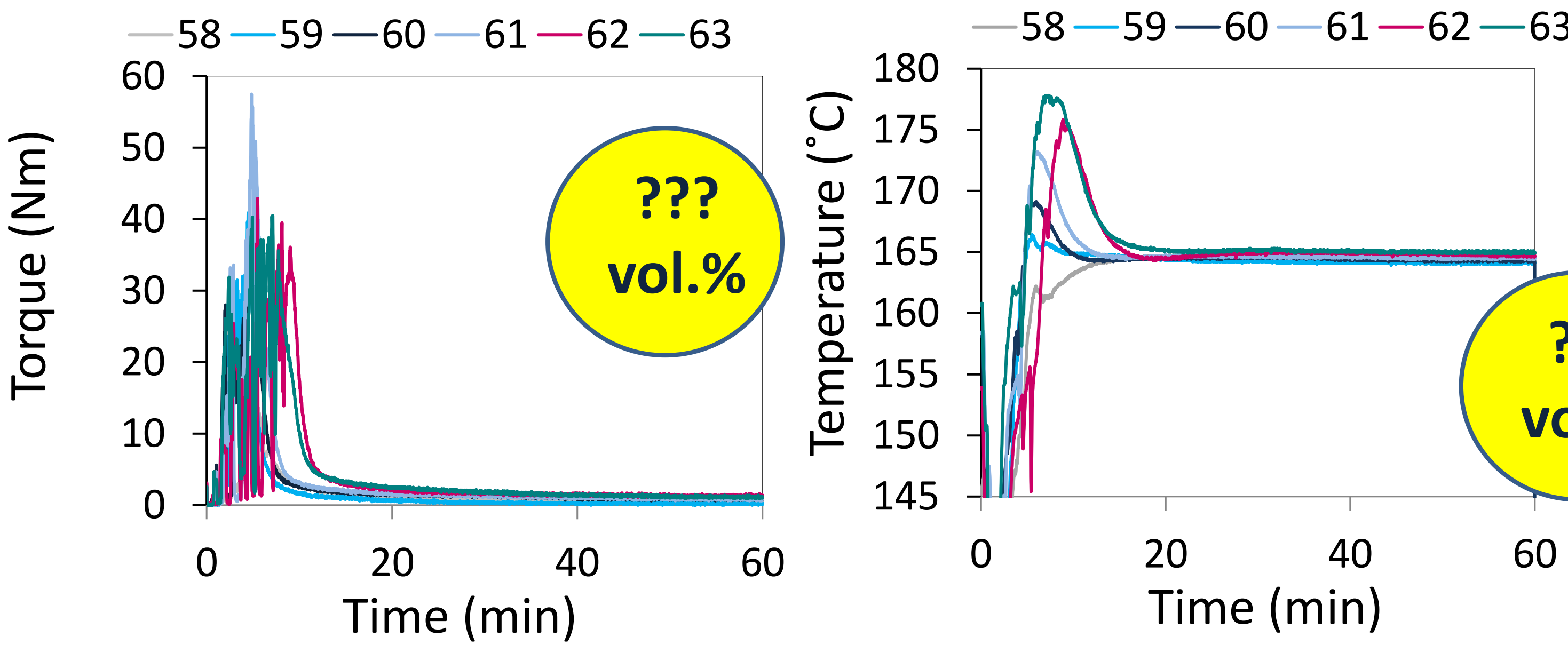


$$CPVC = \frac{V_P}{V_P + V_L}$$

CPCV 62vol.%

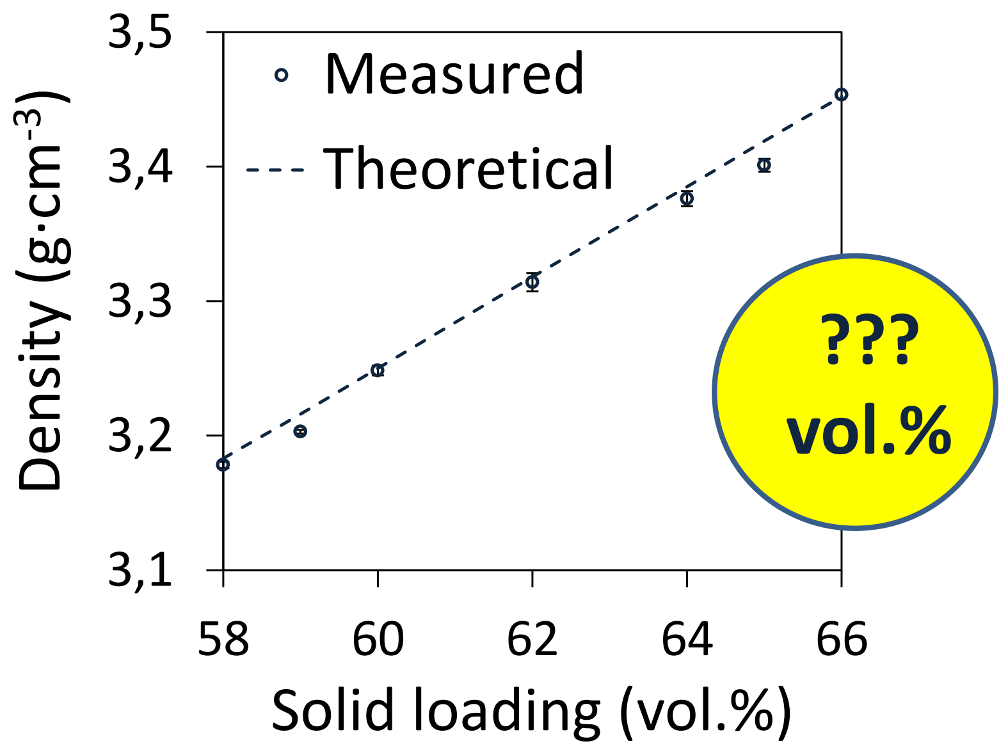
16ml of oil is the maximum value to obtain a coherent mix of 25.8 cm^3 of powders

2. Torque rheometry



Neither torque nor temperature (because of friction of particles) increase during mixing despite the higher solid loadings are tested

3. Feedstock density

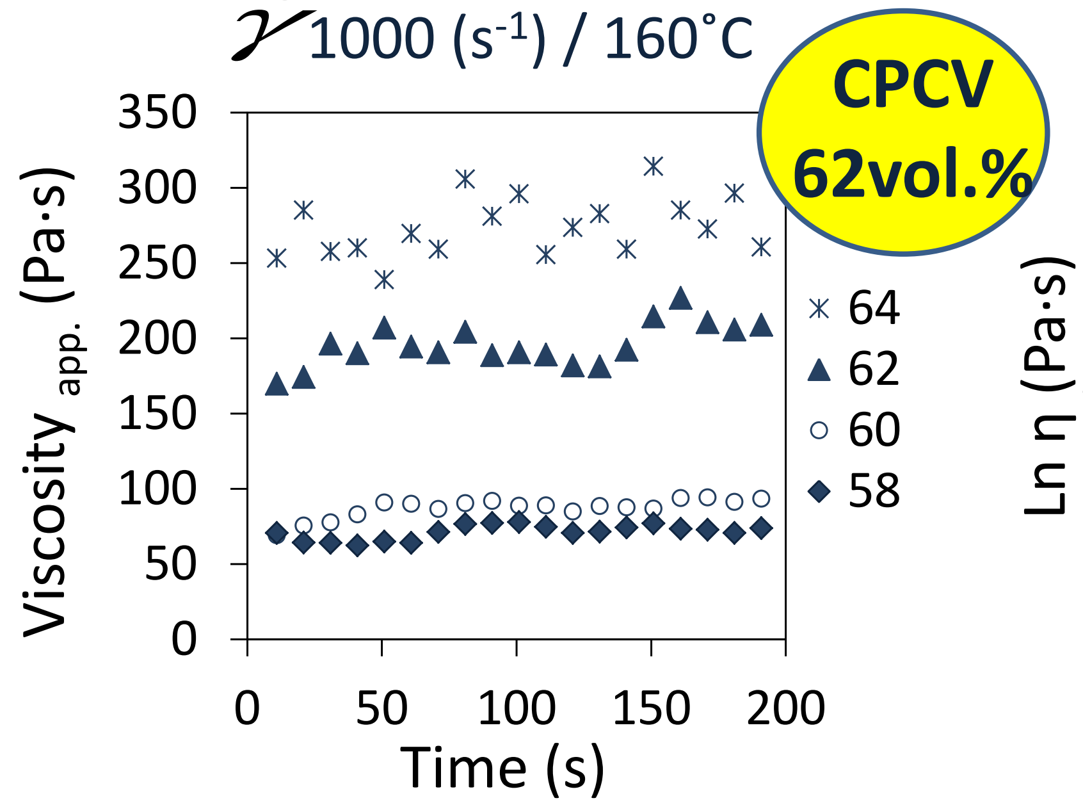


Experimental values are nearly the theoretical ones, even for high solid loadings, without showing pores evidence

4. Capillary rheometry

Homogeneity study

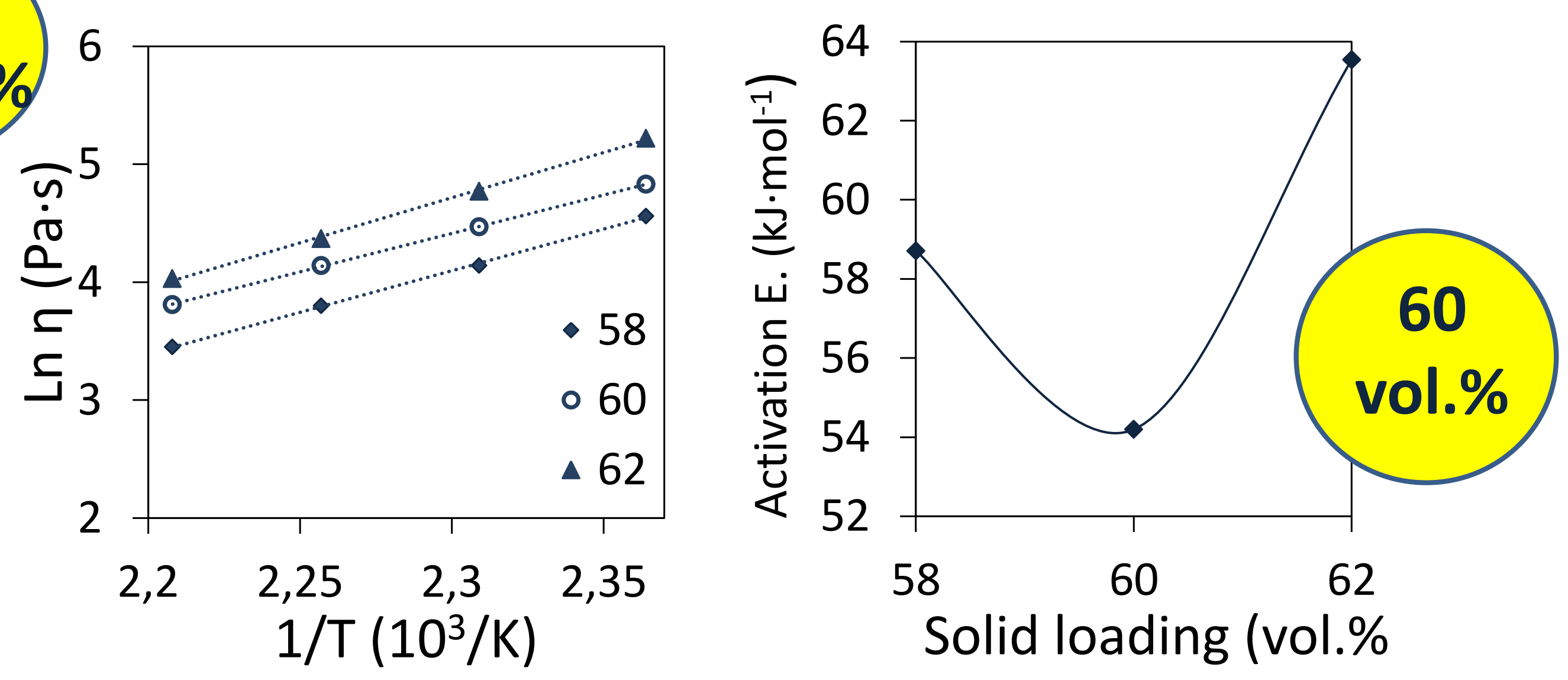
$\dot{\gamma}$ 1000 (s^{-1}) / 160°C



As from 62 vol.% of powder content, viscosity values show more dispersion due to the non homogeneity of feedstock

Temperature influence study

$\dot{\gamma}$ 1000 (s^{-1}) 150-160-170-180°C



$$\eta = B \cdot \exp\left(\frac{E_a}{R \cdot T}\right) \Rightarrow Ea$$

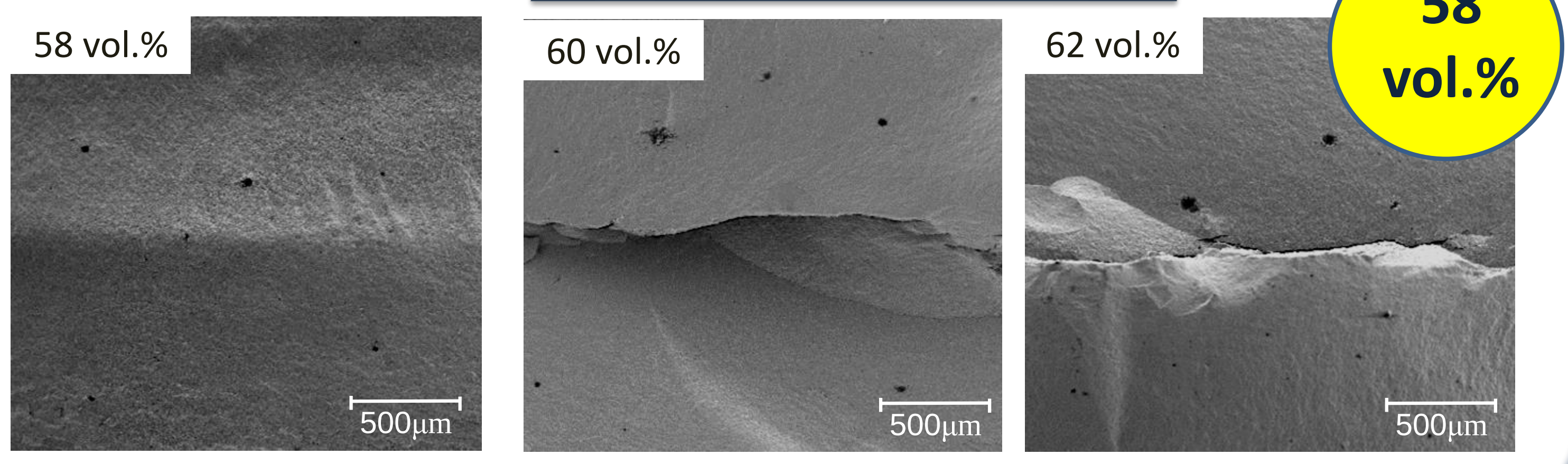
Minimum Ea value is achieved at 60vol%, indicating the lowest influence of T in viscosity

5. Distortion & dimensional behaviour 6. Relative density 7. Mechanical properties

Φ (vol.%)	Distortion (δ) l	Distortion (δ) w	Distortion (δ) t	Shrinkage (%) l	Shrinkage (%) w	Shrinkage (%) t	Relative density (%)	Flexural strength (MPa)	Weibull modulus
58	0.8	4.0	1.6	15.4	15.3	15.5	90.7	**131.7±15**	**10.4**
60	1.3	12.0	1.2	14.9	**12.0**	14.7	**92.2**	74.8±28	7.8
62	**2.4**	**18.5**	**1.8**	14.6	**10.6**	14.1	**92.0**	83.6±38	3.5

58 vol.% ⇒ lower distortion, isotropic shrinkage and higher flexural strength with a good weibull modulus are achieved

8. Fracture surface



Clear surface fracture for 58vol.% of solid loading

Conclusions

- A successful PIM process has been carried out with a raw powder of zirconium silicate with particles with non conventional properties used in this technology (irregular and low tap density) and using a water soluble binder system based on PEG and CAB.
- Many methods have been required to fully optimize the solid loading for this feedstock between 58-60 vol.% of powder.
- Two of the conventional methods used in PIM to obtain the critical solid loading (torque rheometry and density measurements) have not been useful for this feedstock based on extremely irregular particle.

Acknowledgements

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