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# FORMULATION OF OXIDE SUSPENSIONS FOR LIQUID PROCESSING OF CERAMIC MATRIX COMPOSITES

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## Introduction

### Context:

- Oxide-Oxide ceramic matrix composites can be used as high-temperature components for gas turbine engine technologies due to their good thermomechanical properties. [1]
- Liquid processes (*Resin Transfer Molding* and *Liquid Resin Infusion*) are more advantageous than CMC conventional ones.
- The formulation of suspensions represents the first essential step for liquid processing. [2]
- The suspension must be well dispersed, stable and have a low viscosity (< 1 Pa.s).

### Aim of this study:

- Formulate a bi-component suspension (with submicron alumina and colloidal silica) suitable for the liquid processing of oxide-oxide ceramic matrix composites

## Materials

- Mixture proportion: 59 vol.% of Al<sub>2</sub>O<sub>3</sub> and 41 vol.% of SiO<sub>2</sub>, corresponding to the mullite ratio

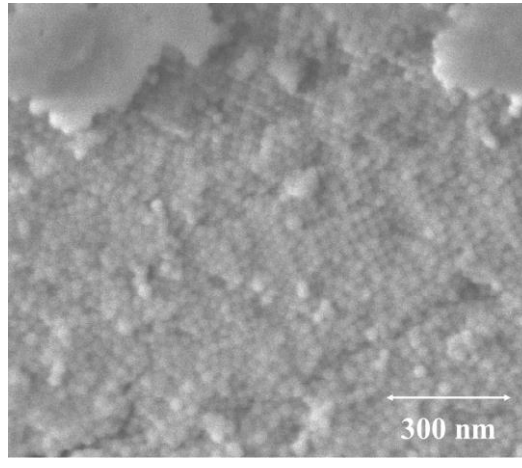
### Colloidal Silica

Particle size	20.0 – 24.0 nm
Surface area	129 – 155 m <sup>2</sup> .g <sup>-1</sup>
Volumetric mass density	2.2 g.cm <sup>-3</sup>

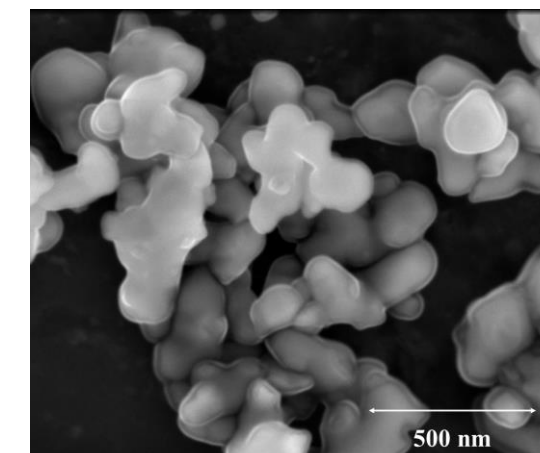
Ludox® AS-40, 40 wt.% suspension in water (Grace Davison, USA)

Technical Data

SEM Observation



### Submicron Alumina



AKP50, high purity alpha-alumina powder (Sumitomo Chemical, Japan)

SEM Observation

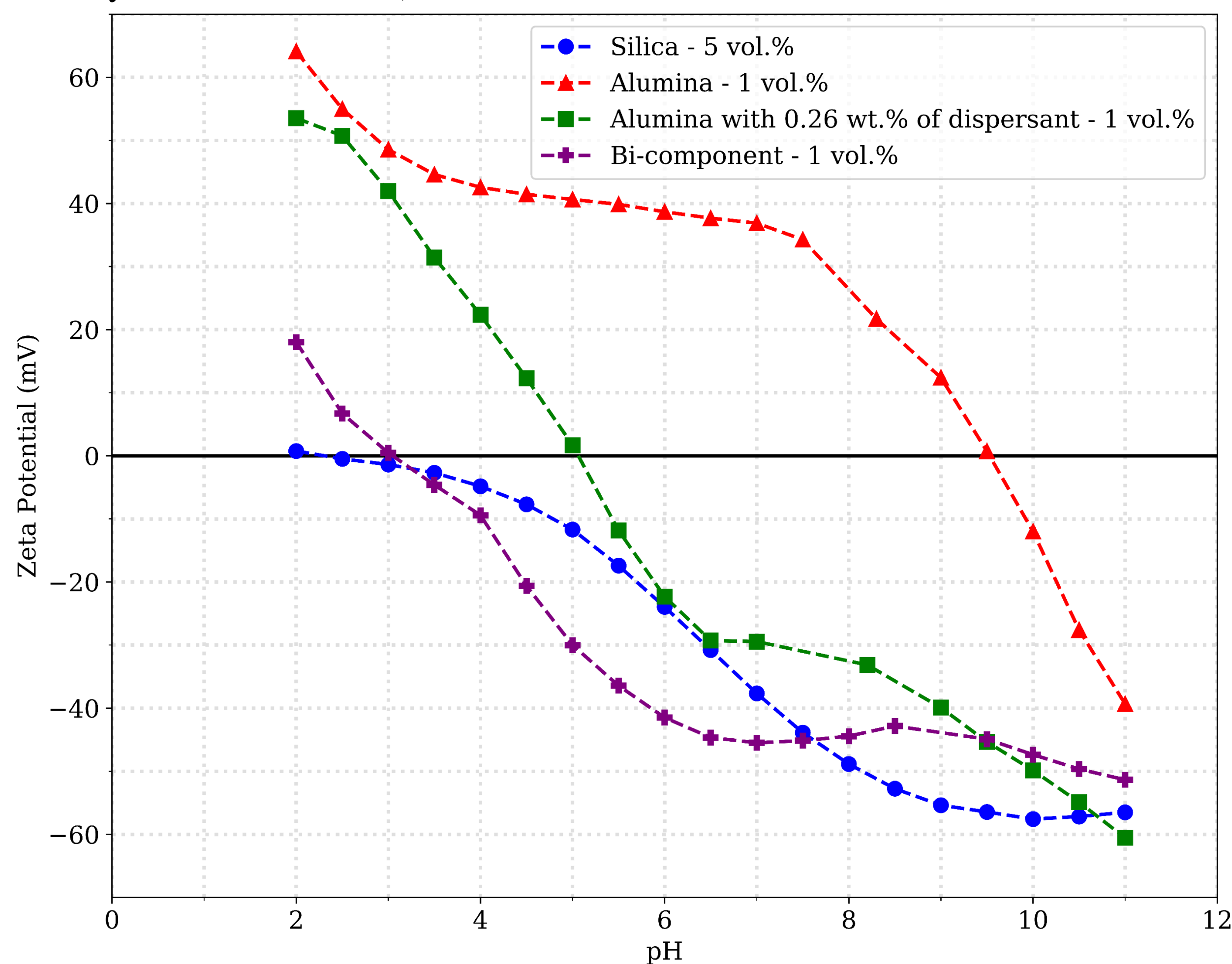
Technical Data

Particle size	200 nm
Surface area	10.3 m <sup>2</sup> .g <sup>-1</sup>
Volumetric mass density	3.98 g.cm <sup>-3</sup>

## Results

### Dispersion

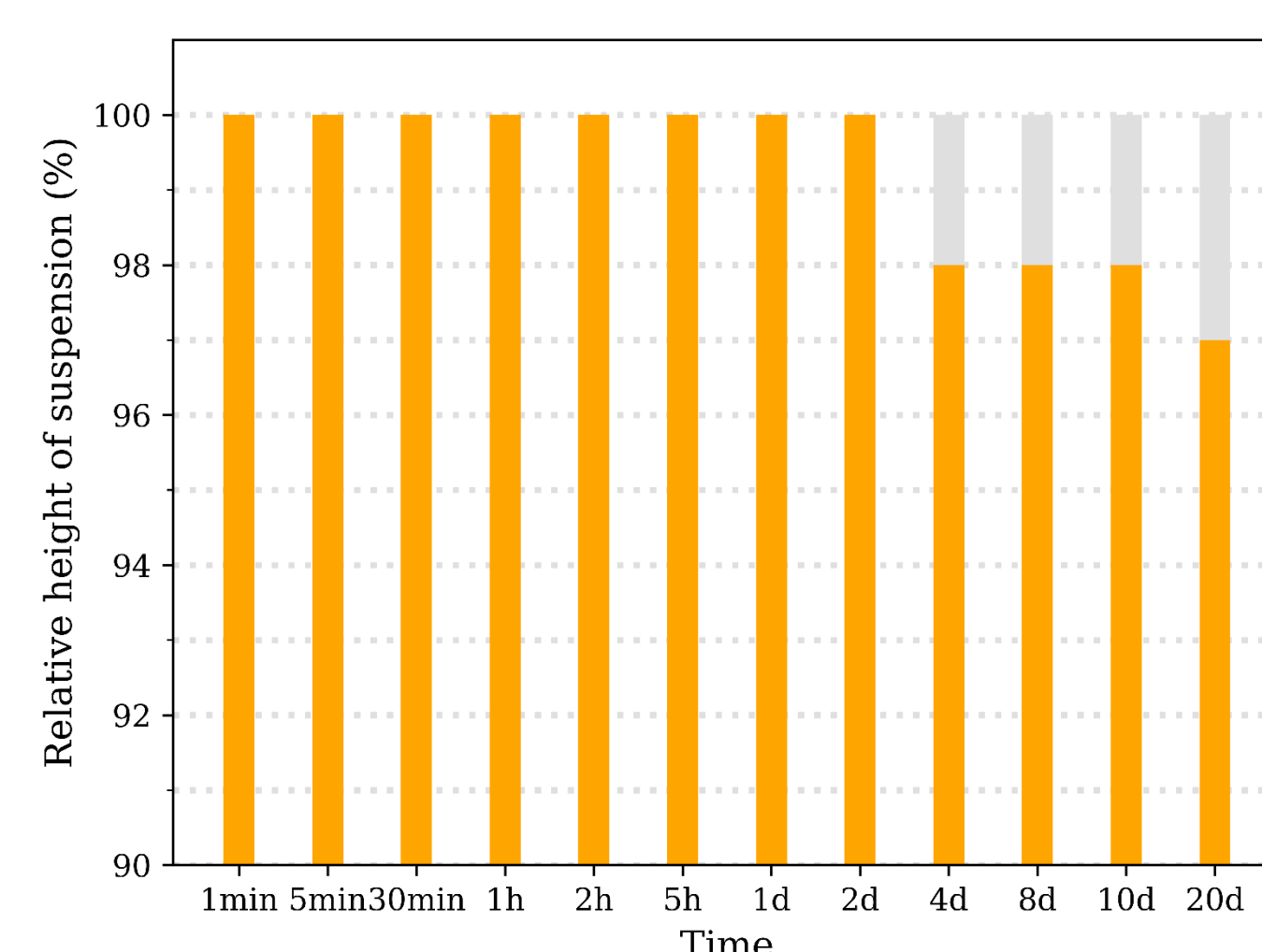
- Measurement of the zeta potential of diluted suspensions as a function of pH – Acoustosizer IIS (Colloidal Dynamics LLC, USA)



- The silica suspension possesses a negative zeta potential from pH = 2 to pH = 11.
- The zeta potential of the alumina suspension is positive ( $\zeta = 21.7$  mV) at natural pH (pH = 8.3) and its value is not sufficient to avoid particles agglomeration.
- This agglomeration can take place between particles of alumina (homo-agglomeration) but also between particles of alumina and silica (hetero-agglomeration).
- By adding 0.26 wt.% of dispersant (Darvan® C-N, Vanderbilt Minerals LLC, USA), an ammonium polymethacrylate, the isoelectric point (IEP) of the alumina is shifted from pH = 9.5 (without dispersant) to pH = 5.1.
- The bi-component suspension is defined as the mixture of the silica suspension and the alumina suspension with 0.26 wt.% of dispersant in the proportion given previously.
- This suspension is well dispersed because the zeta potential at natural pH (pH = 9.5) is measured at  $\zeta = -44.7$  mV.

### Stability

- Sedimentation tests were performed to study the stability of the suspension.
- A 40 vol.% bi-component suspension was considered.
- Sedimentation is observable after a time of 4 days.
- In consequence, the bi-component suspension is stable in comparison with the process time (less than one day).

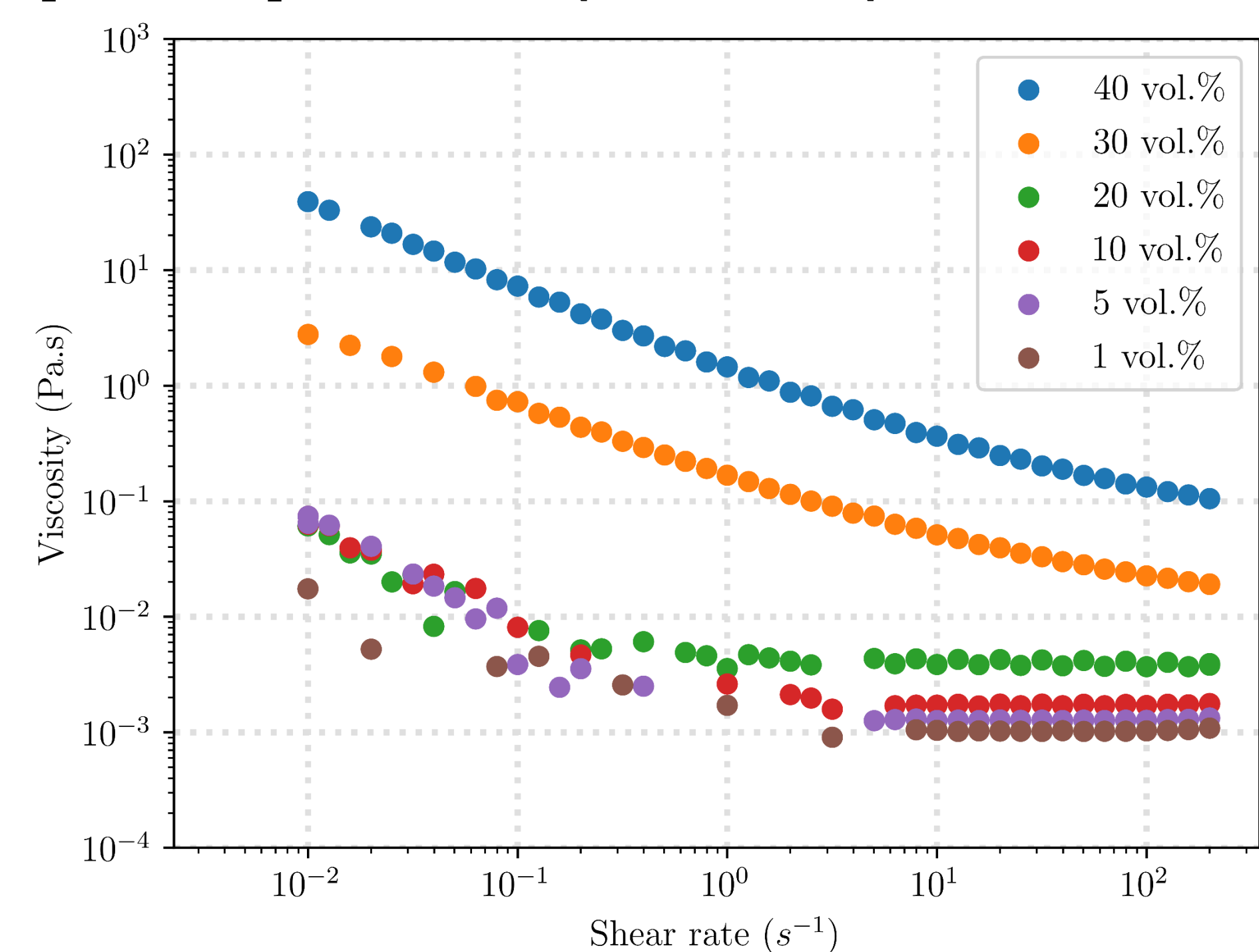


### Rheology

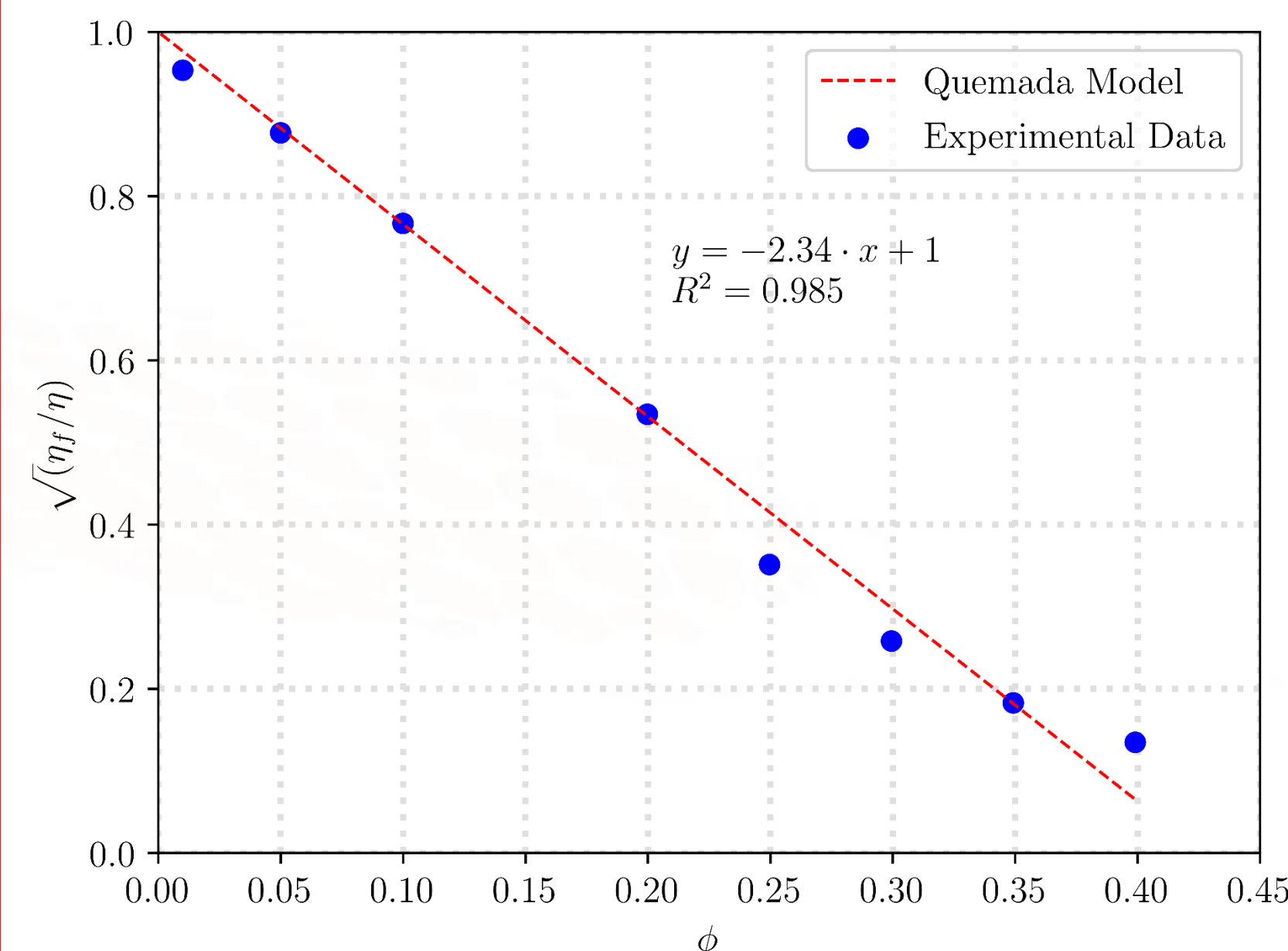
- Measurements performed using an AR-G2 rheometer (TA Instruments, USA) equipped with a cone-plate geometry.

- Rheological behavior of the bi-component suspension (from  $\dot{\gamma} = 10^{-2}$  s<sup>-1</sup> to  $\dot{\gamma} = 200$  s<sup>-1</sup>)

- The rheological behavior of the suspension is dominated by the colloidal and hydrodynamic interactions and by the brownian motions.
- At low shear rates, the behavior is shear thinning.
- Whereas at high shear rates, the viscosity is constant.
- Moreover, the solid concentration represents an essential variable regarding the rheology.



- Influence of solid concentration on suspension viscosity



- Quemada model:

$$\sqrt{\frac{\eta_f}{\eta}} = 1 - \frac{\phi}{\phi_{max}}$$

$\eta$  : viscosity of the suspension at the plateau (Pa.s)  
 $\eta_f$  : viscosity of the interstitial fluid – water (Pa.s)  
 $\phi$  : solid concentration (vol.%)  
 $\phi_{max}$  : maximum packing fraction (vol.%)

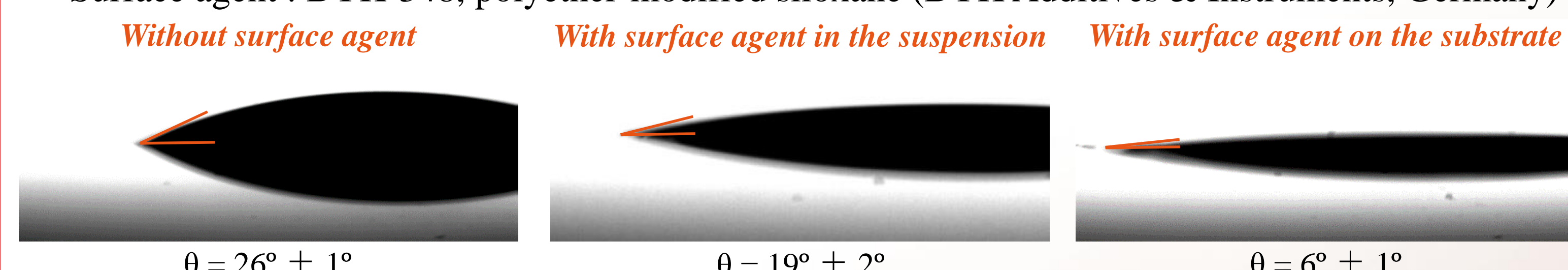
Identification of the model parameter  $\phi_{max}$  by linear regression

$\eta_f$	$\phi_{max}$
0.001 Pa.s	42.69 vol.%

- The influence of solid concentration on the suspension viscosity is well described by a Quemada model.

### Wetting

- Measurement of the contact angle of a 25 vol.% bi-component suspension drop on an alumina planar substrate
- Surface agent : BYK-348, polyether-modified siloxane (BYK Additives & Instruments, Germany)



- The contact angle decreases by using surface agent either in the suspension and on the substrate.
- The case for which the surface agent is deposited on the substrate surface is the most favorable.

## Conclusion

- A suspension of submicron alumina and colloidal silica has been formulated. It is well dispersed (no agglomeration of particles), stable (in comparison with the process time) and has a low viscosity (< 1 Pa.s). Moreover, the influence of solid concentration on the rheological behavior of the suspension is well described by a Quemada model. Furthermore, the wetting of the suspension can be promoted by using a surface agent.
- This bi-component suspension is thus suitable for the liquid processing of oxide-oxide ceramic matrix composites.

## References

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- Billotte, C., Fotsing, E.R. and Ruiz, E. (2017). Optimization of alumina slurry for oxide-oxide ceramic composites manufactured by injection molding. *Advances in Materials Science and Engineering*, vol. 2017.

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