



# International Electrokinetics Conference

## Elkin 2004



## Influence of the polyelectrolyte and base addition sequence on the suspension properties and electrophoretic deposition behaviour of ceramic powders

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# Outline

**Goal:** *Influence of sequence addition of BA and DC on the properties of  $Al_2O_3$  and Ce-ZrO<sub>2</sub> deposits*

**Suspension characterisation:** Viscosity  
BA content & sequence addition

 7.4 to 74 wt% BA; 100 to 200  $\mu$ l/g DC

**Electrophoretic deposition:** Yield, Deformability  
Drying  
Density

## Suspension preparation

- $\text{Al}_2\text{O}_3$  (Baikowski SM8) powder
- $\text{Ce-ZrO}_2$  (Daiichi CEZ-12) powder
- Ethanol (to avoid gas evolution during EPD)
- Darvan C (DC) ammonium polymethacrylate

MW= 10000 - 16000

BA (base)  $\uparrow$   $\rightarrow$  degree of dissociation ( $\alpha$ )  $\uparrow$ :



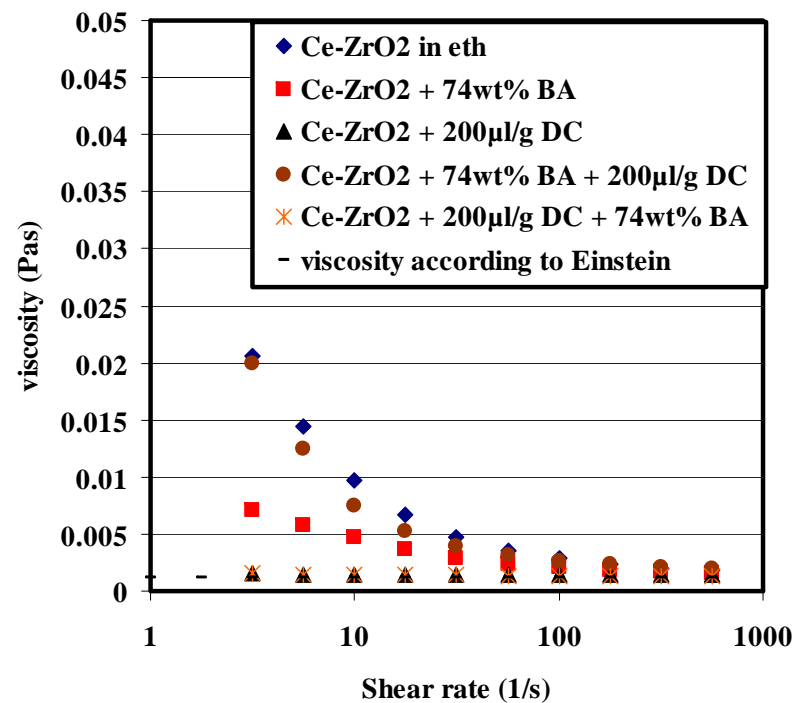
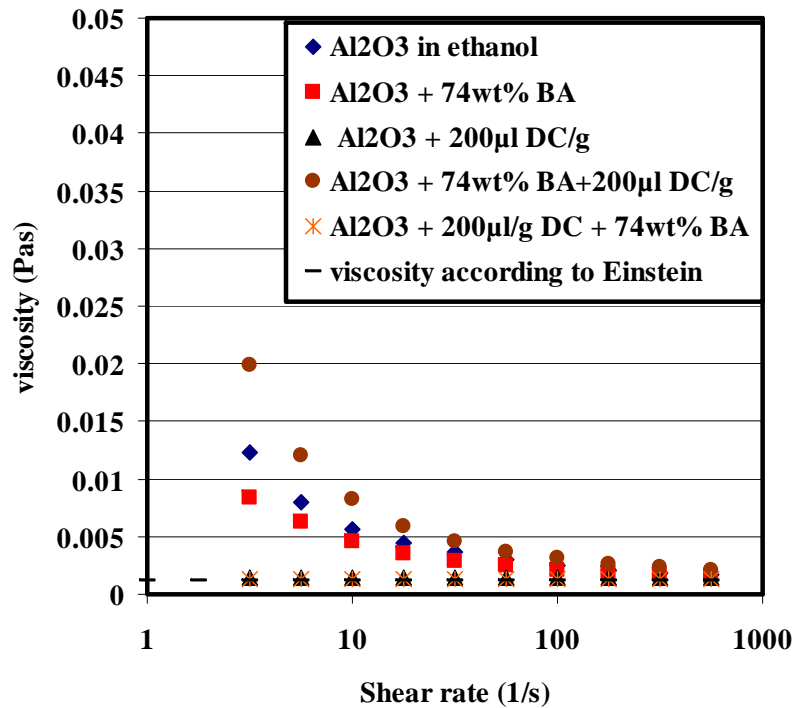
- n-Butylamine (BA) (for DC dissociation)

➔ Suspension viscosity investigated as function of the addition sequence of BA and DC

# Suspension characteristics

Influence of **BA** and **DC** addition sequence on  $\eta$  at  $\uparrow$  BA content

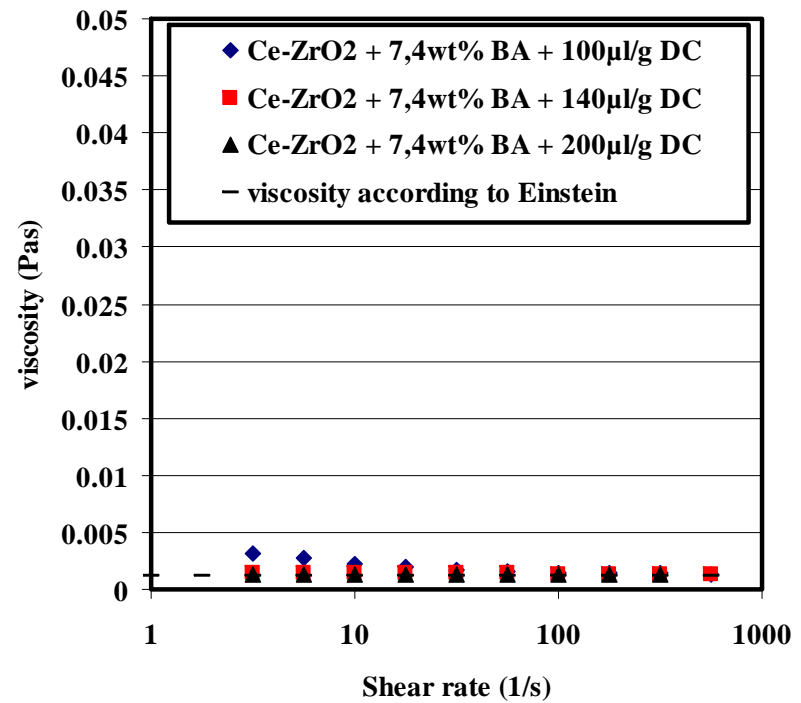
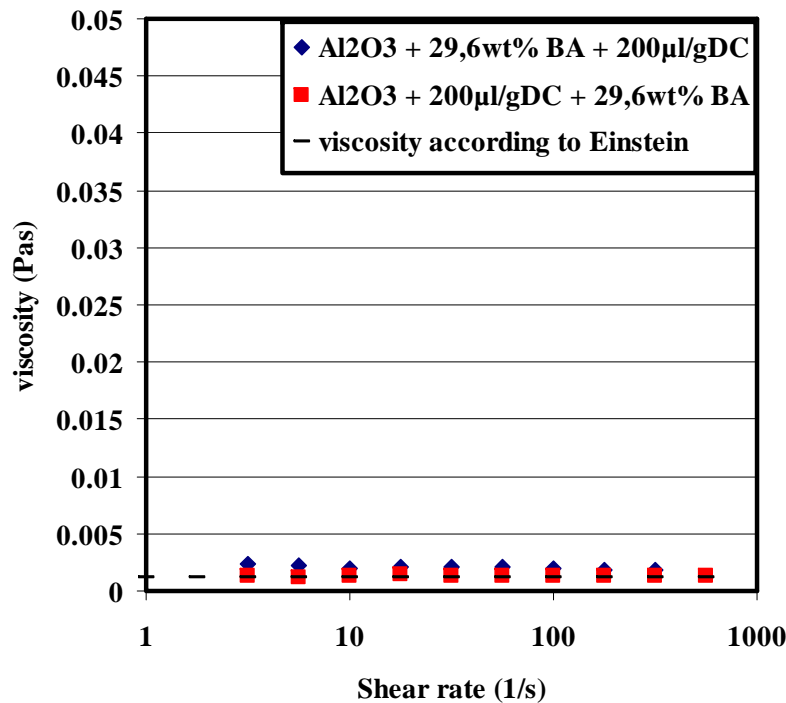
Suspensions: 2.5 vol %  $\text{Al}_2\text{O}_3$ , 1.5 vol % Ce-ZrO<sub>2</sub>



When **BA** first added compound  $\rightarrow \uparrow \eta$ , unstable suspension  
 When **DC** first added compound  $\rightarrow \downarrow \eta$ , stable suspension

# Suspension characteristics

## Influence of BA and DC addition sequence on $\eta$ at $\downarrow$ BA content

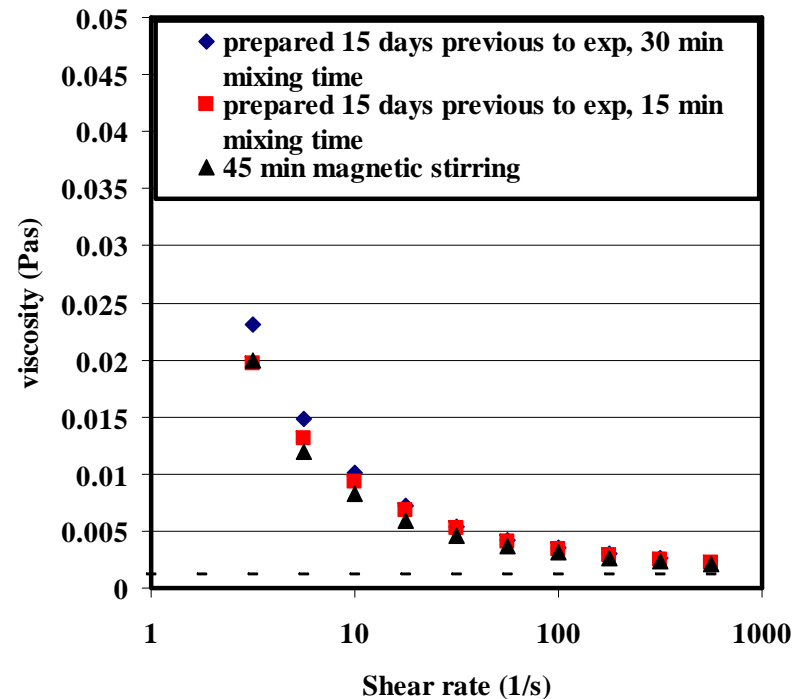
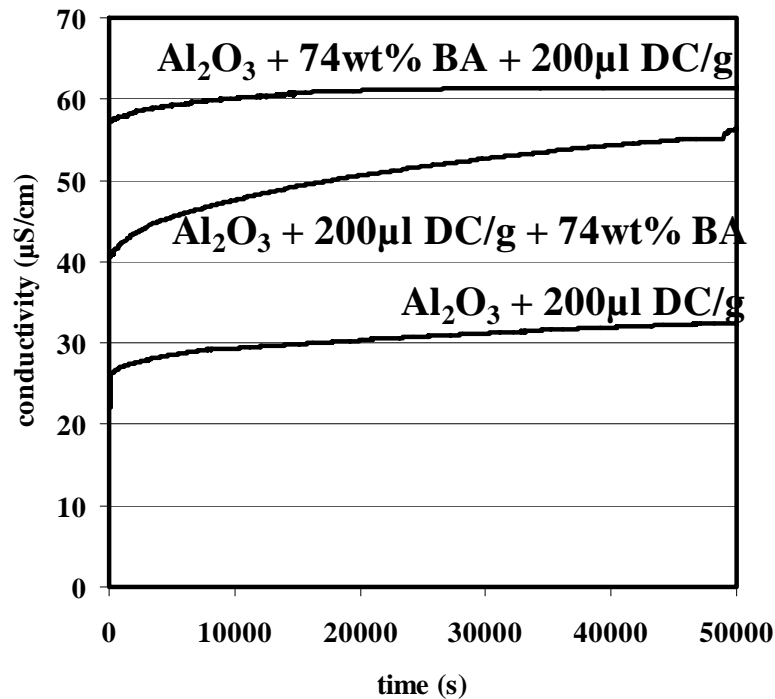


$\downarrow \eta$  with  $\downarrow$  BA, lower BA content, stable suspensions

BA content influences  $\eta$  more than the DC content and the addition sequence

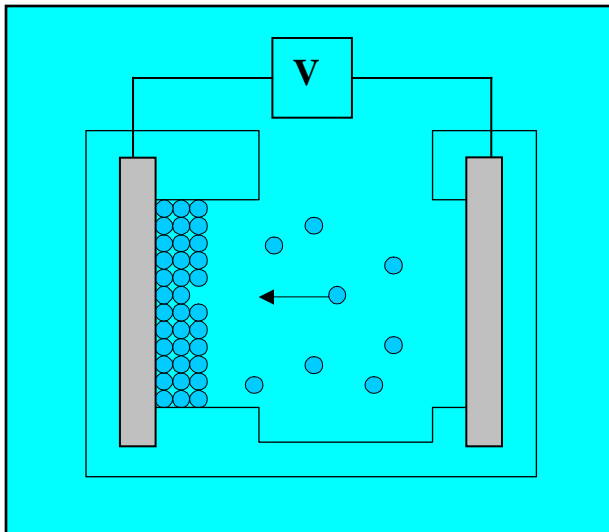
# Suspension characteristics

## Suspension for a deformable deposit: chemical stability in time



Constant conductivity and  $\eta$  in time  $\rightarrow$  good chemical stability

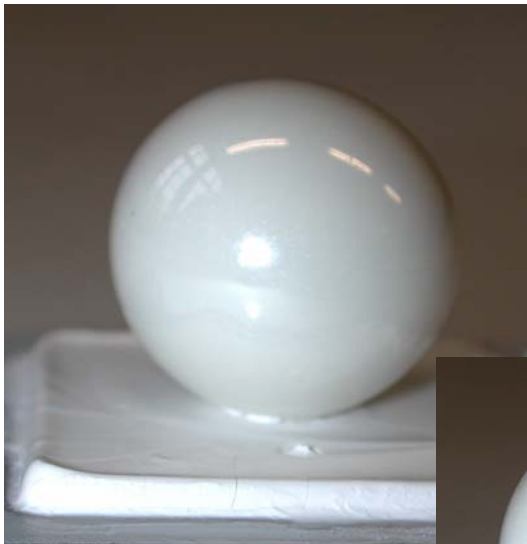
## EPD conditions



- **300 V**
- **Magnetic stirring during EPD**
- **Vertical electrodes**
- **Stainless steel electrodes**  
distance 3.5 cm  
surface 9 cm<sup>2</sup>

# Deposit characteristics

| Powder                         | First added compound | Second added compound | Viscosity (Pas) at 3.16 s <sup>-1</sup> | Deposit characteristics |
|--------------------------------|----------------------|-----------------------|---|-------------------------|
| Al <sub>2</sub> O <sub>3</sub> | 74wt% BA             | 200μl/g DC            | 0.0199                                  | Deformable              |
| Al <sub>2</sub> O <sub>3</sub> | 200μl/g DC           | 74wt% BA              | 0.0013                                  | Rigid                   |
| Ce-ZrO <sub>2</sub>            | 74wt% BA             | 200μl/g DC            | 0.0199                                  | Deformable              |
| Ce-ZrO <sub>2</sub>            | 200μl/g DC           | 74wt% BA              | 0.0015                                  | Rigid                   |
| Al <sub>2</sub> O <sub>3</sub> | 29.6wt% BA           | 200μl/g DC            | 0.0023                                  | Deformable              |
| Al <sub>2</sub> O <sub>3</sub> | 200μl/g DC           | 29.6wt% BA            | 0.0013                                  | Rigid                   |
| Ce-ZrO <sub>2</sub>            | 7.4wt% BA            | 100μl/g DC            | 0.0031                                  | Deformable              |
| Ce-ZrO <sub>2</sub>            | 7.4wt% BA            | 140μl/g DC            | 0.0015                                  | Deformable              |
| Ce-ZrO <sub>2</sub>            | 7.4wt% BA            | 200μl/g DC            | 0.0013                                  | Deformable              |



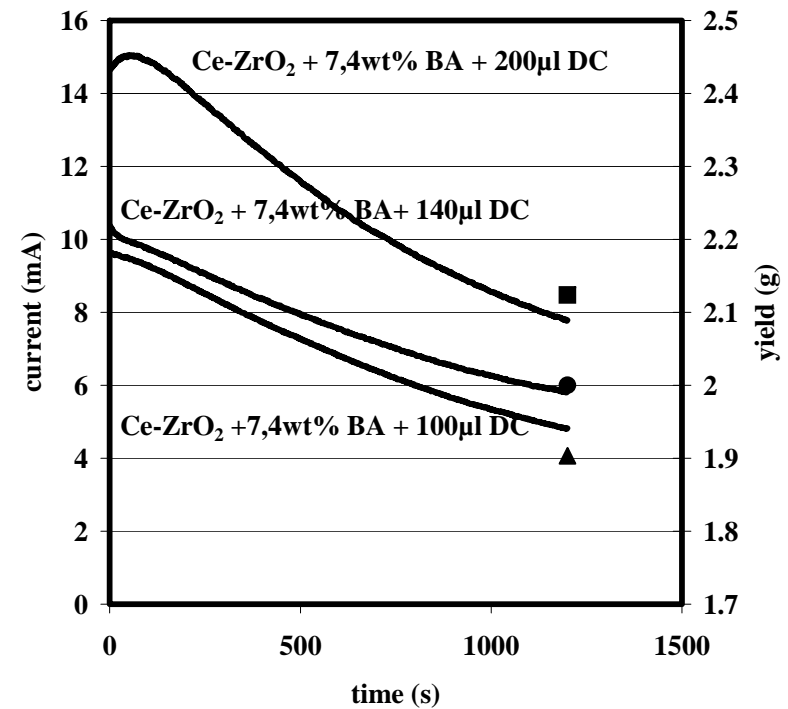
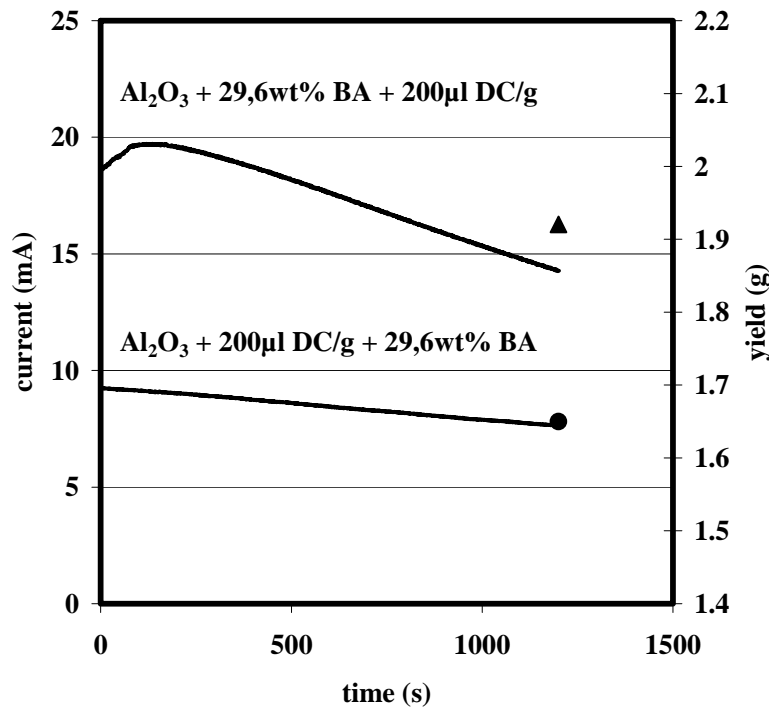
**BA first added compound** →  
**deformable deposit**

**DC first added compound** →  
**rigid deposit**

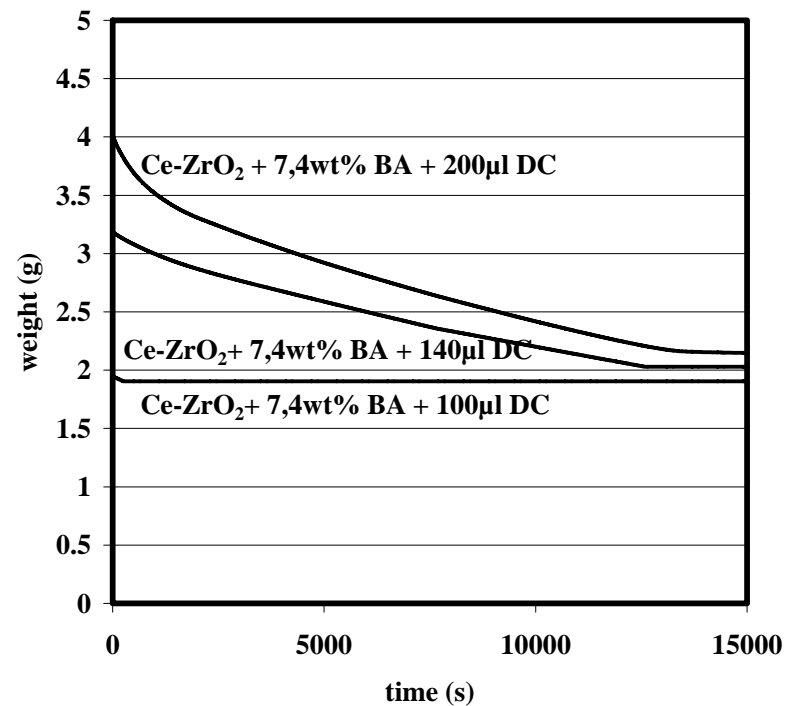
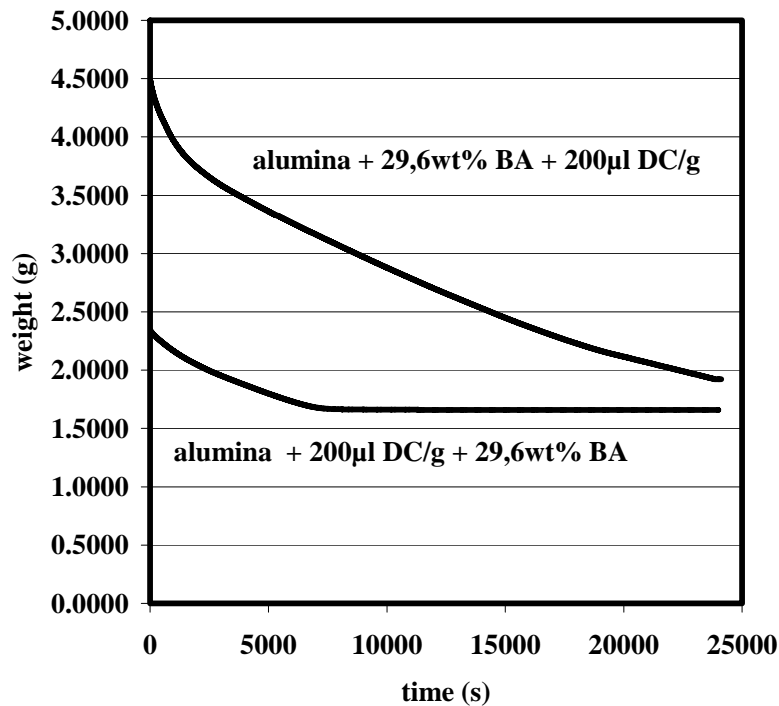


# Electrophoretic deposition

**Current: BA first added > DC first added**  
**BA constant and DC  $\uparrow$   $\rightarrow$  Current  $\uparrow$ , Yield  $\uparrow$**



# Drying



**Drying time** when BA added first >> DC added first  
**BA first added compound**

**BA constant drying time ↑ with ↑ DC content**

## Density

- Small influence of BA & DC addition sequence or content
- Large influence of particle characteristics

Relative density  $\text{Al}_2\text{O}_3$  deposits ~40%

Relative density Ce-ZrO<sub>2</sub> deposits ~20%

$\text{Al}_2\text{O}_3$  is  $\mu\text{m}$  sized (0.6  $\mu\text{m}$ ) → packing ↑

Ce-ZrO<sub>2</sub> is nm sized → packing ↓

# Conclusions

## ➤ Suspension stability

- At  $\uparrow$  BA content:

**BA first added:** unstable suspensions (possible network formation by free polymer bridges)

**DC first added:** stable suspensions

- At  $\downarrow$  BA content:

**no influence of addition sequence, stable suspensions**

# Conclusions

## ➤ Deposit characteristics

- BA first added → deformable deposit
- DC first added → rigid deposit
- drying time ↑ as BA and/or DC ↑
- Similar behavior of  $\text{Al}_2\text{O}_3$  and  $\text{Ce-ZrO}_2$  suspensions



# Acknowledgements



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