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Manufacturing Dense Inorganic Solids Suspensions: Industrial Challenge Case Study

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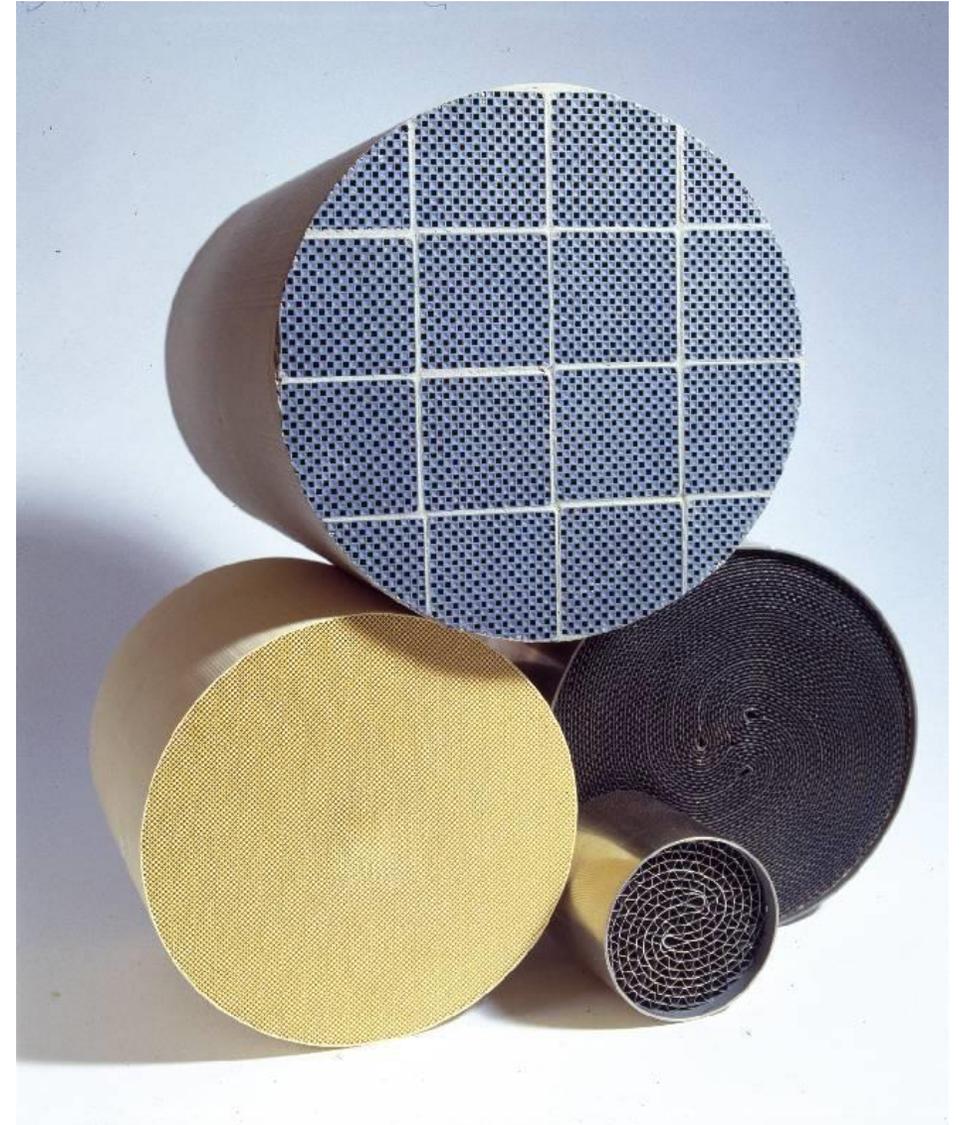
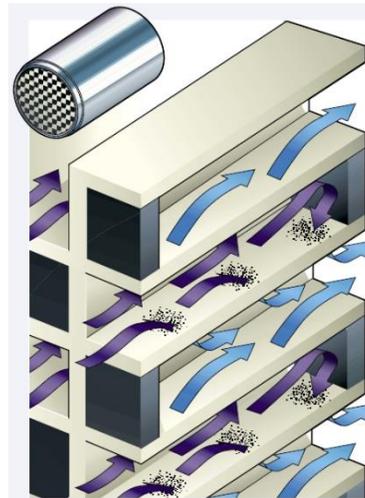
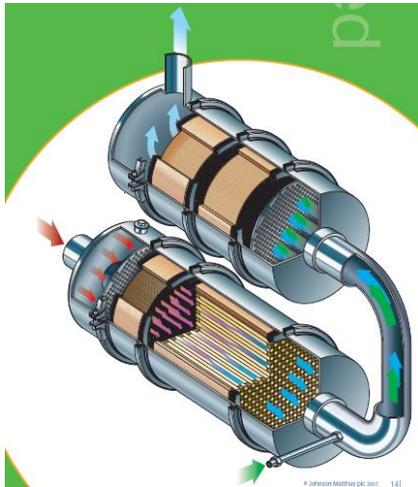
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JM Emission Control Catalyst Application (from Slurry)



Integrated Oxidation & NOx removal

Catalytic Soot Filter



Slurry mixing – Industrial Perspective Challenges

Manufacture of Washcoats & Inks for Coated Final Product Form

High solids content, Small Particles, Interactions

- Literature tends on focus on either
 - Low solids contents and/or
 - Large particles – no interaction
- **Evolving Rheology**
 - Process fluid changes significantly in rheology
 - Deliberate (rheology modifier) or “Accidental”
- **Changing liquid level**
 - Additions during the batch process
- **Quality of mixing**
 - 95% does not count

Final Product Demands

- Particle size: $d(50) < 10$ micron: coated layer: 20-100 micron
 - **Consistency requires across the coated layer**; say $50 (\mu\text{m})^3 = 12.5 \text{ pl}$

Manufacture

- **Vessel sizes vary** (100 litres – 10 m³); say 5 m³ = 5,000 litres
- Dosing volume per coating: order of 50 - 250 ml
 - **Uniformity from dose to dose and intra-dose**

Quality of Mixing Required: > 99.999% (viz. > 5S)

- Literature mixing times, $t(m95\%)$, are not relevant
- Literature scales of segregation are not relevant

Mixing Quality requirement: Washcoat & Ink Formulation (Example)

Scale of Mixing required: < 50 micron

- Ideally down to particle scale: which particle size; $d(50)$, $d(10)$?

Kolmogorov Considerations: example;

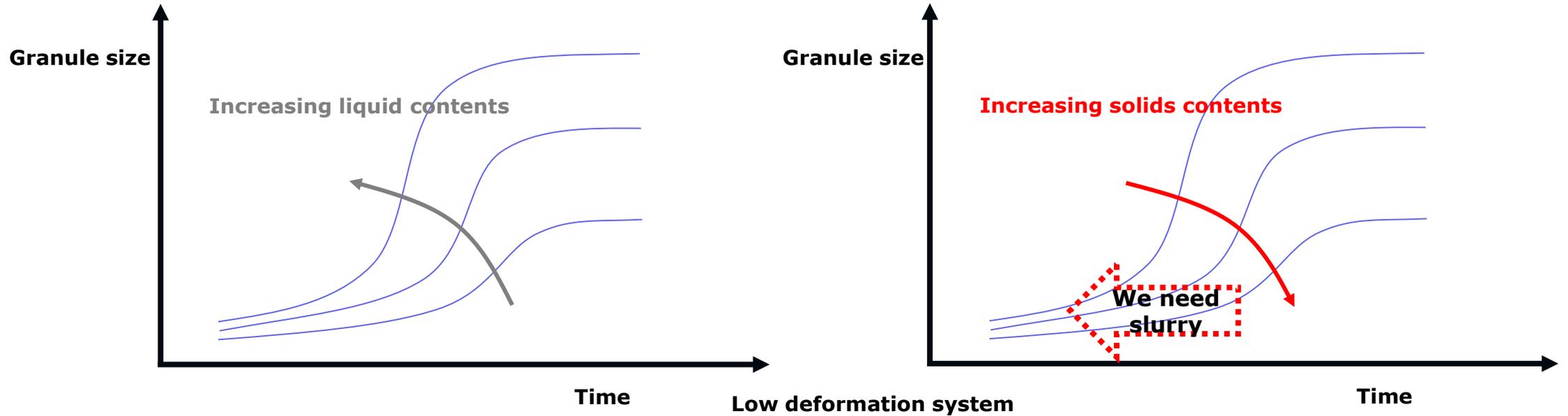
- Nominal viscosity: say 1,000 – 5,000 cP: Slurry density 1200 kg/m³
- Kolmogorov length scale:

	Water	Low Rheology	High Rheology
Viscosity (Pa.s)	1E-3	1	5
Density (kg/m ³)	1,000	1,200	1,200
Energy dissipation (W/kg)	1	1	1
Kolmogorov Length (mm)	32	5,000	16,000

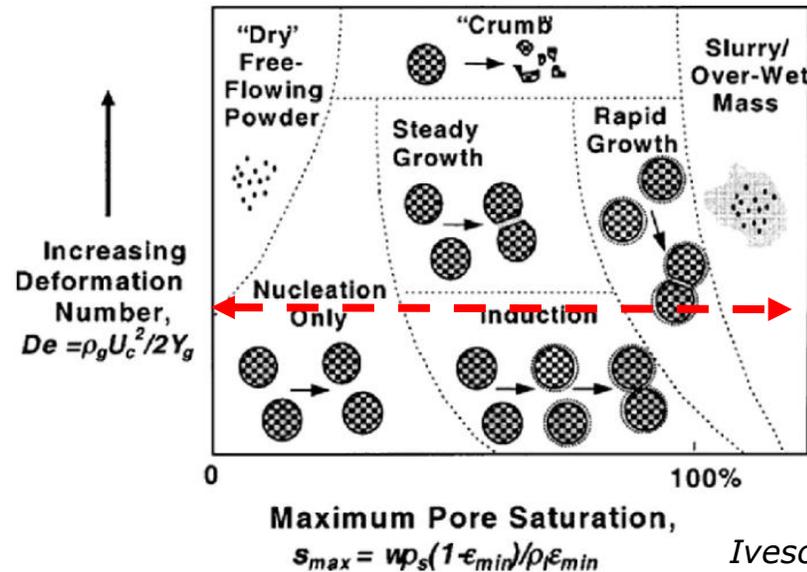
Scale of segregation << Kolmogorov length

Hydraulic shear alone will not achieve necessary scale of mixing

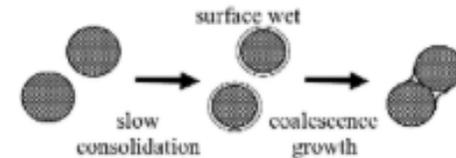
Powder, Liquid, Impeller, High shear to make one product



A round way when making slurry refer to regime



Happens when continuously adding powder & batching slurry!



Case Study: Manufacture of Dense Slurries

We add a **LOT** of powder

Literature focuses on geometric optimisations

- All at low solid contents (<1%)
- Are results applicable at high solids?

Typical literature is on floating solids

- Particle density < fluid density
 - Wet alumina density ca. 1,500 kg/m³
Titania, silica etc are similar
- **Not the case on many industrial processes**



“Floating” Solids

- 1) Lower density than fluid
- 2) Surface tension forces prevent solids from sinking
- 3) Agglomeration of particles traps air to a low bulk density



“Drawdown”

Must be drawn-down from surface before incorporation

‘Just drawdown’ condition when no powder spends more than **4 seconds** on surface



“Just Incorporated”



What happens at high solids contents?

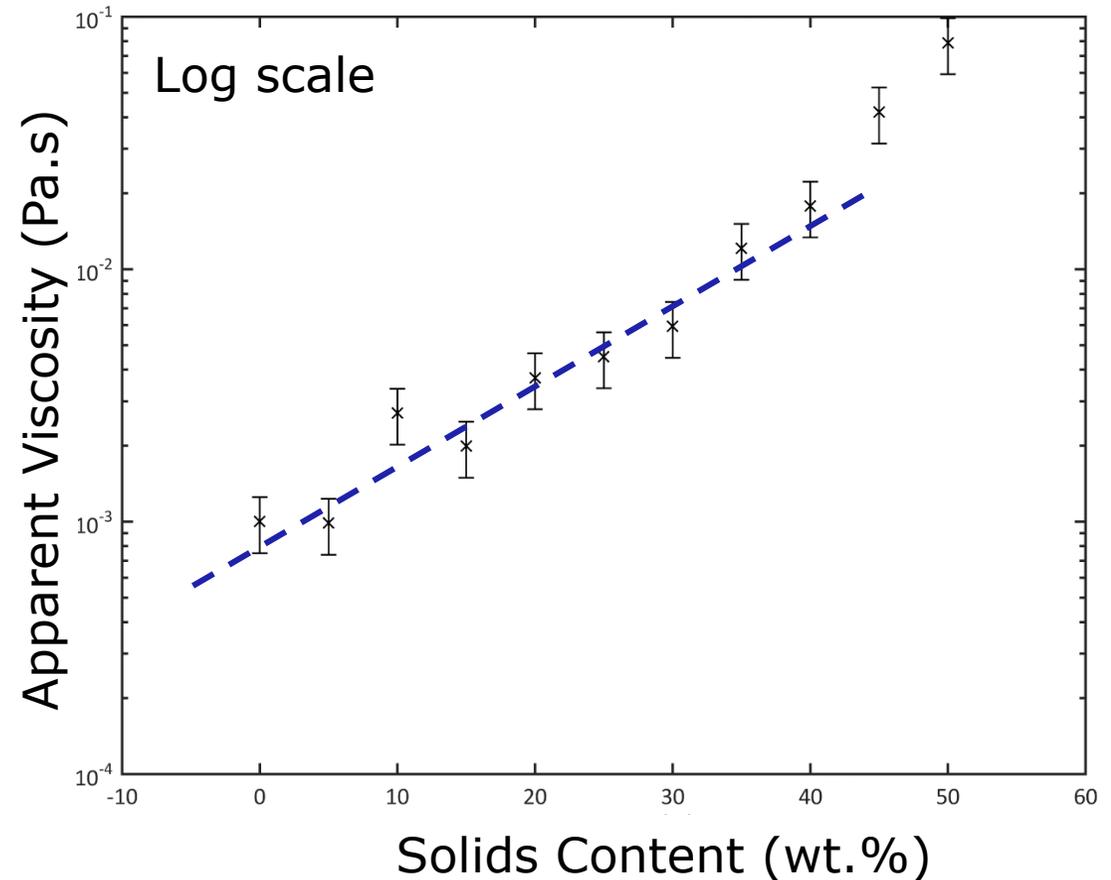
“Viscosity” increases: rheology becomes non-Newtonian

- Rheology difficult to measure due to sedimenting particles
- Apparent viscosity measured at a single shear rate: 200 s^{-1}
 - Parallel plate rheometer
- **Significant change in rheology through the batch addition**

E.g. at constant N:

- Re increases with wt.% solids

Noted:
Incremental specific energy needs increase through the process

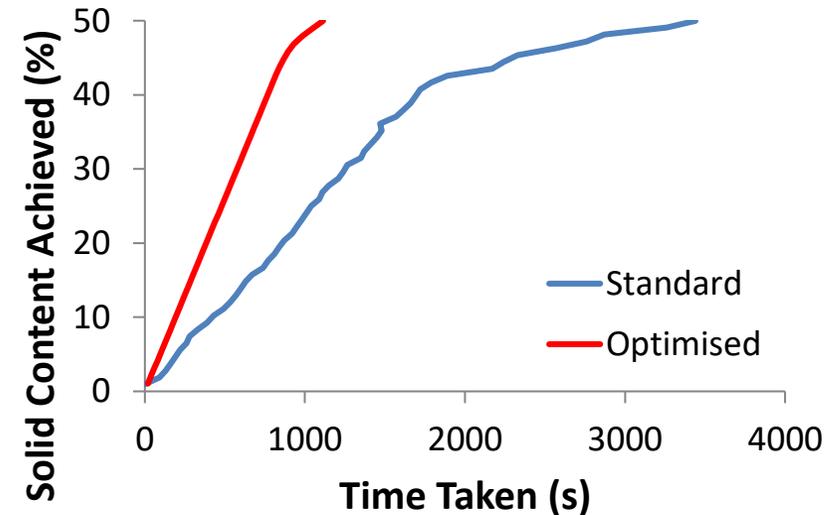
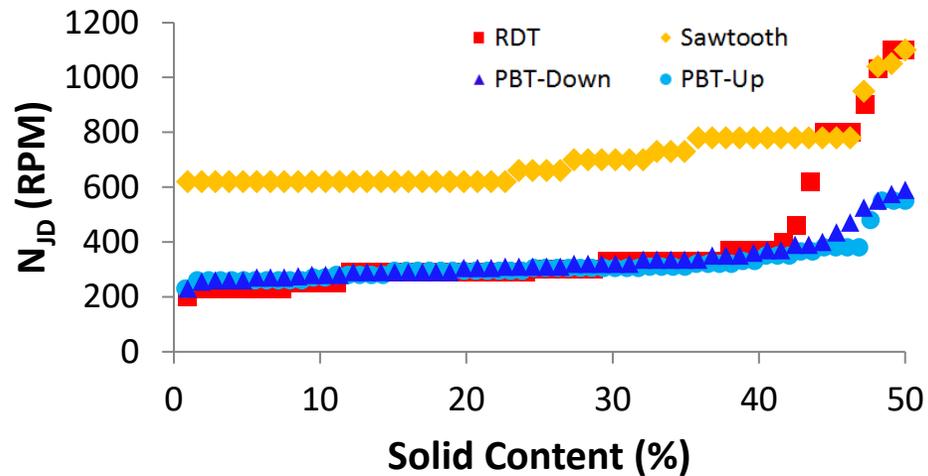
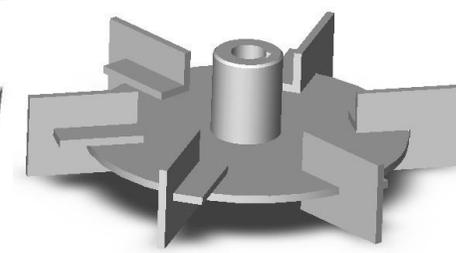
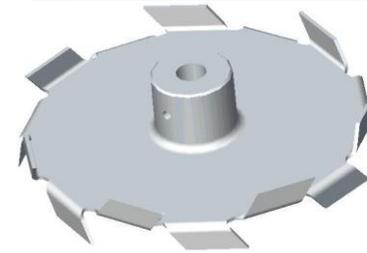
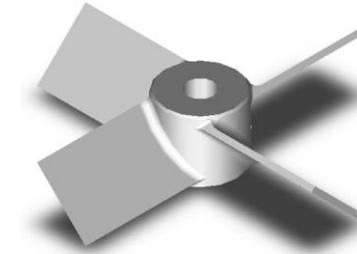


Batch Drawdown Optimisation

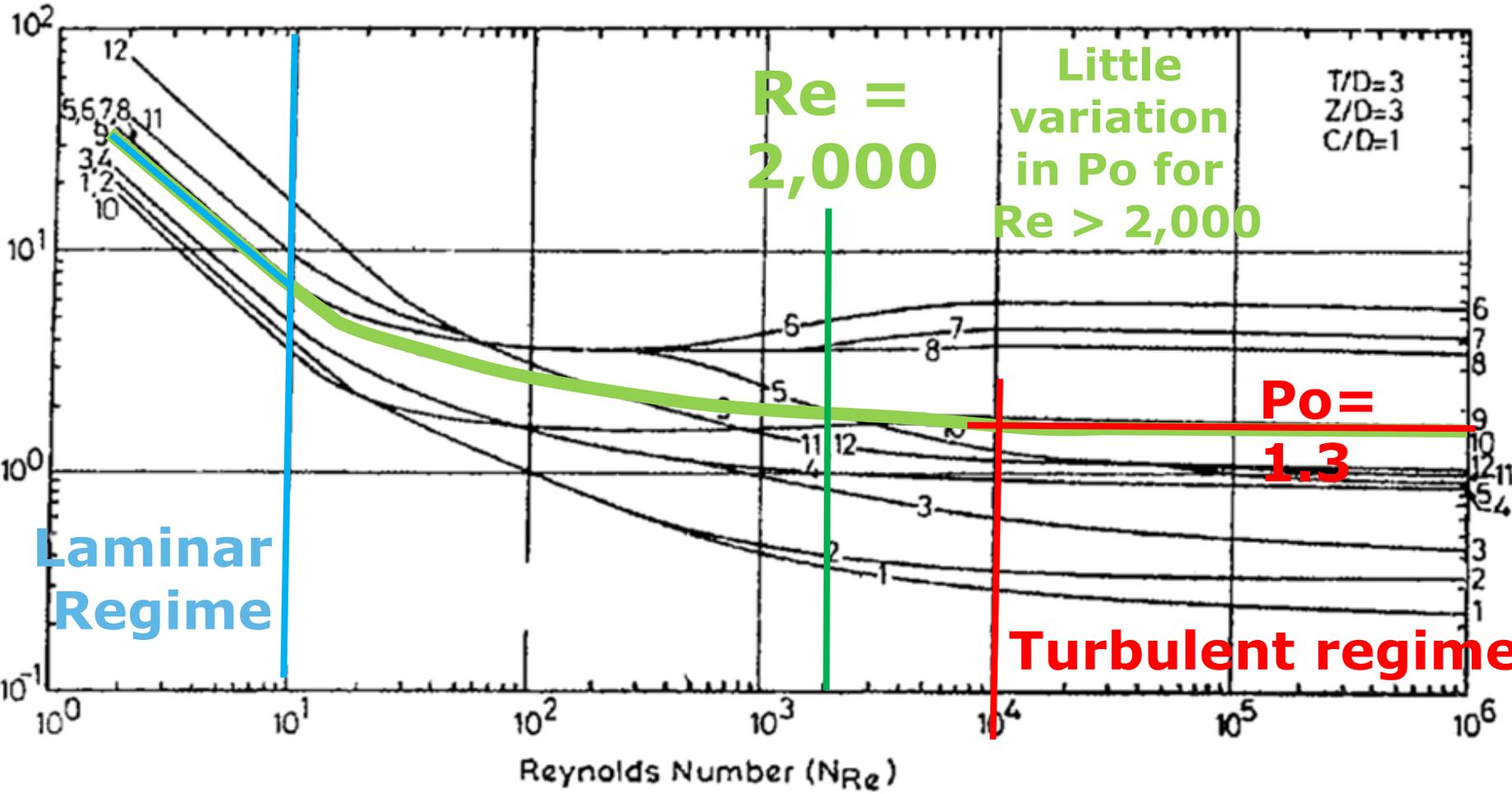
Stirred tank geometry optimisation

- Large diameter impellers
- Axial flow impellers
- Ideal submergence range $0.35 < S/T < 0.6$

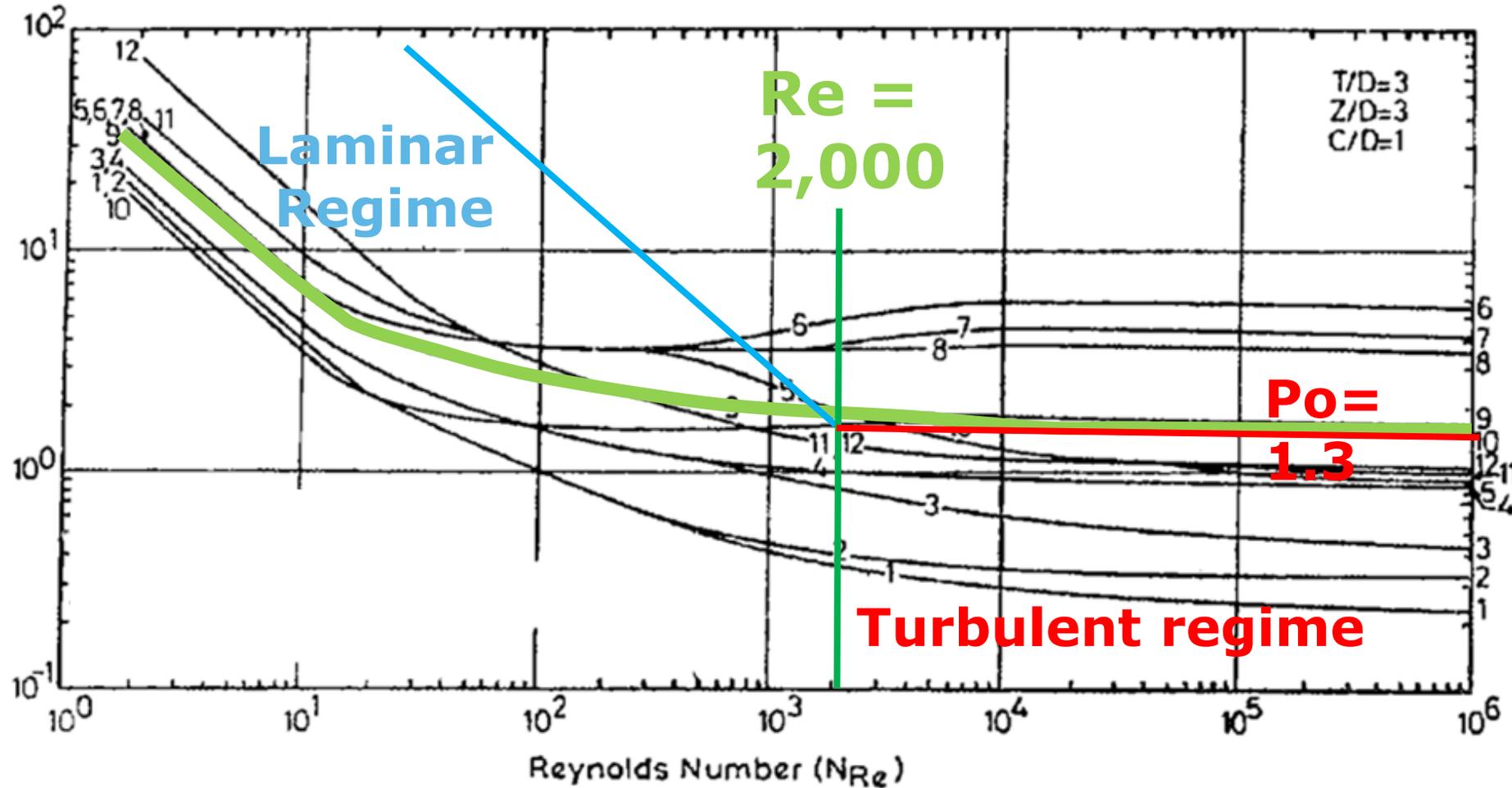
Significant improvement in time required to prepare slurry



What would we expect for a PBT ?

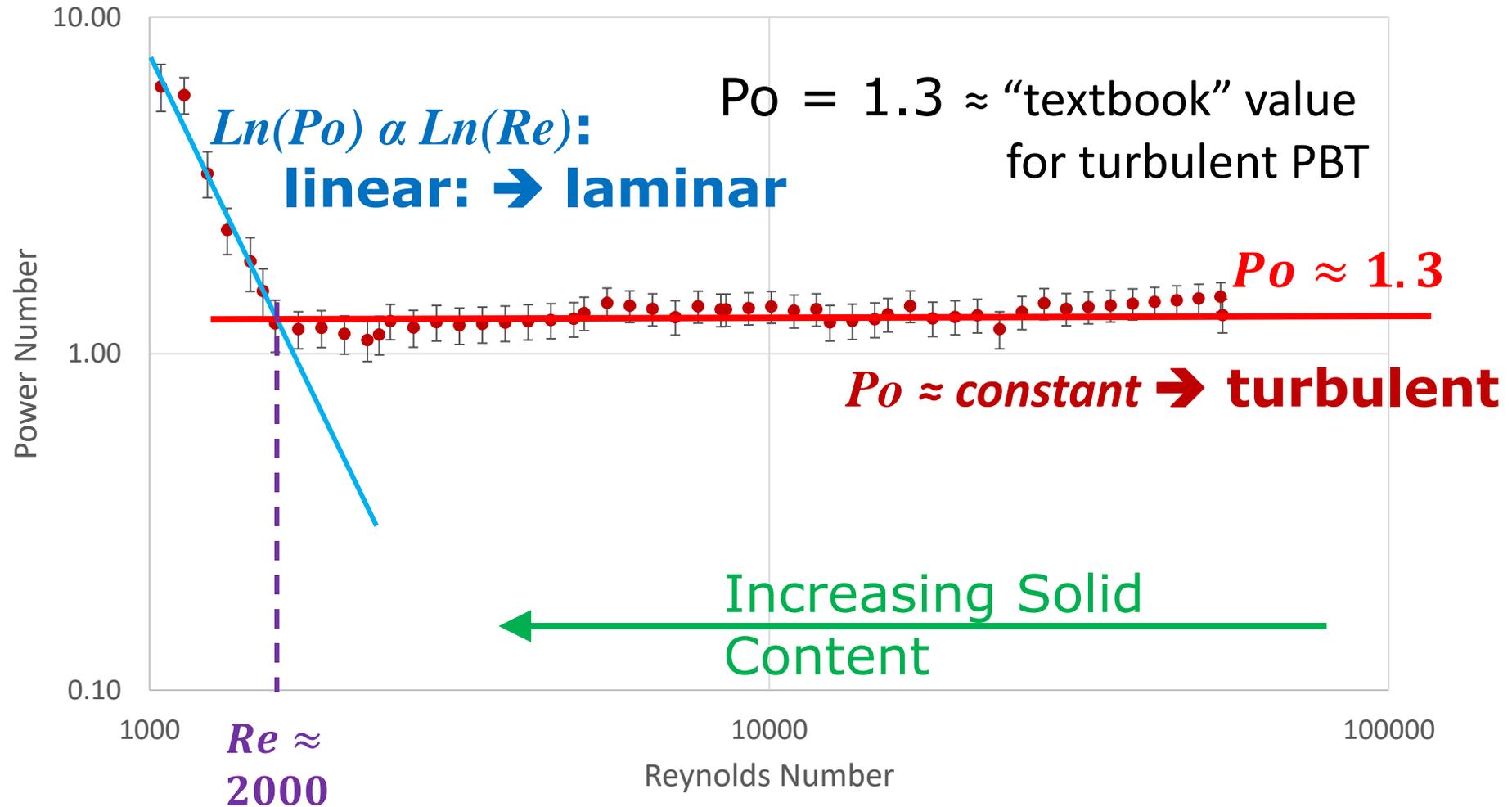


What we actually get in the transient process

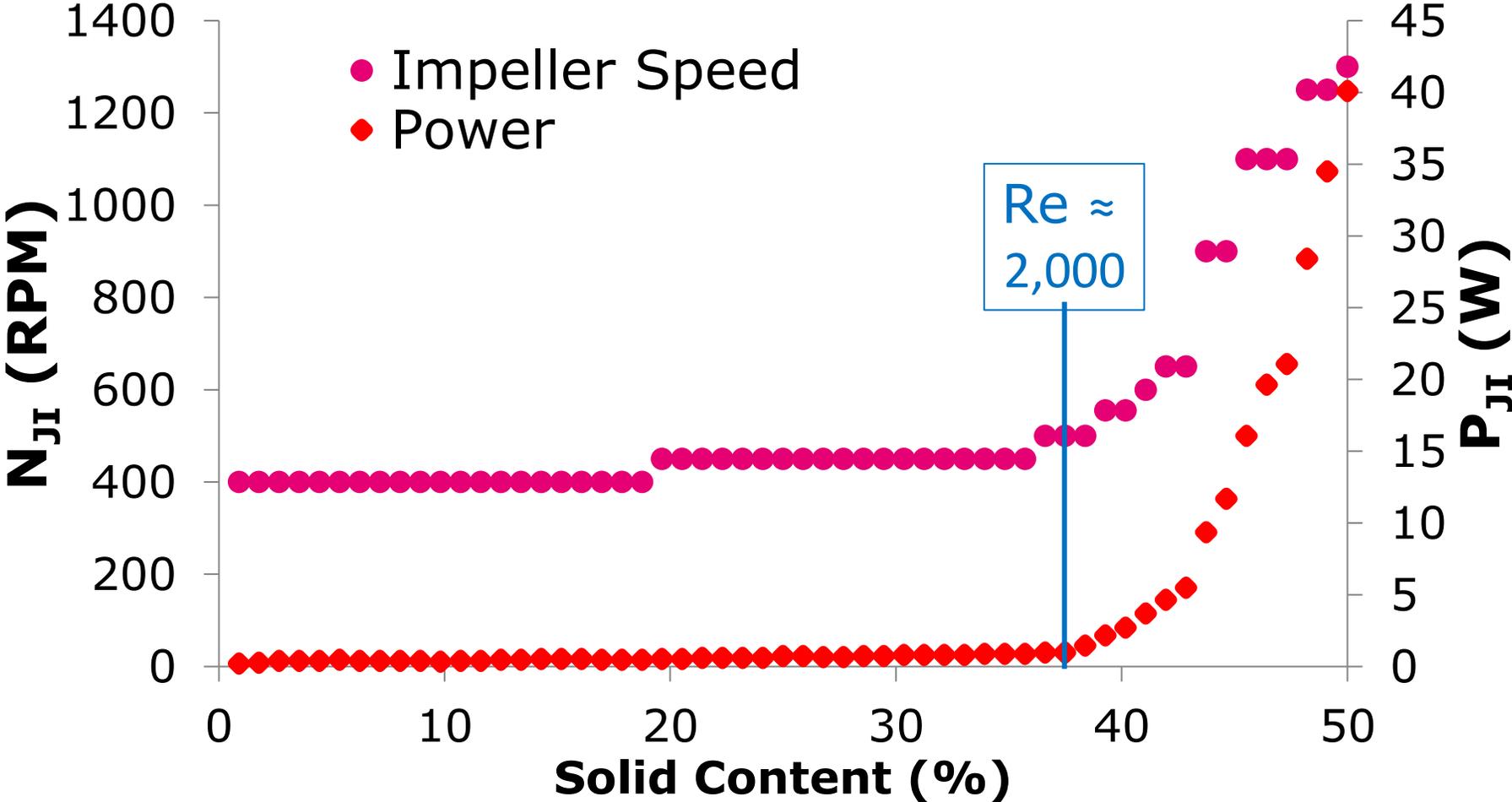


No transitional regime. High Power numbers in laminar regime

Power vs Reynolds Number (PBT-DP)



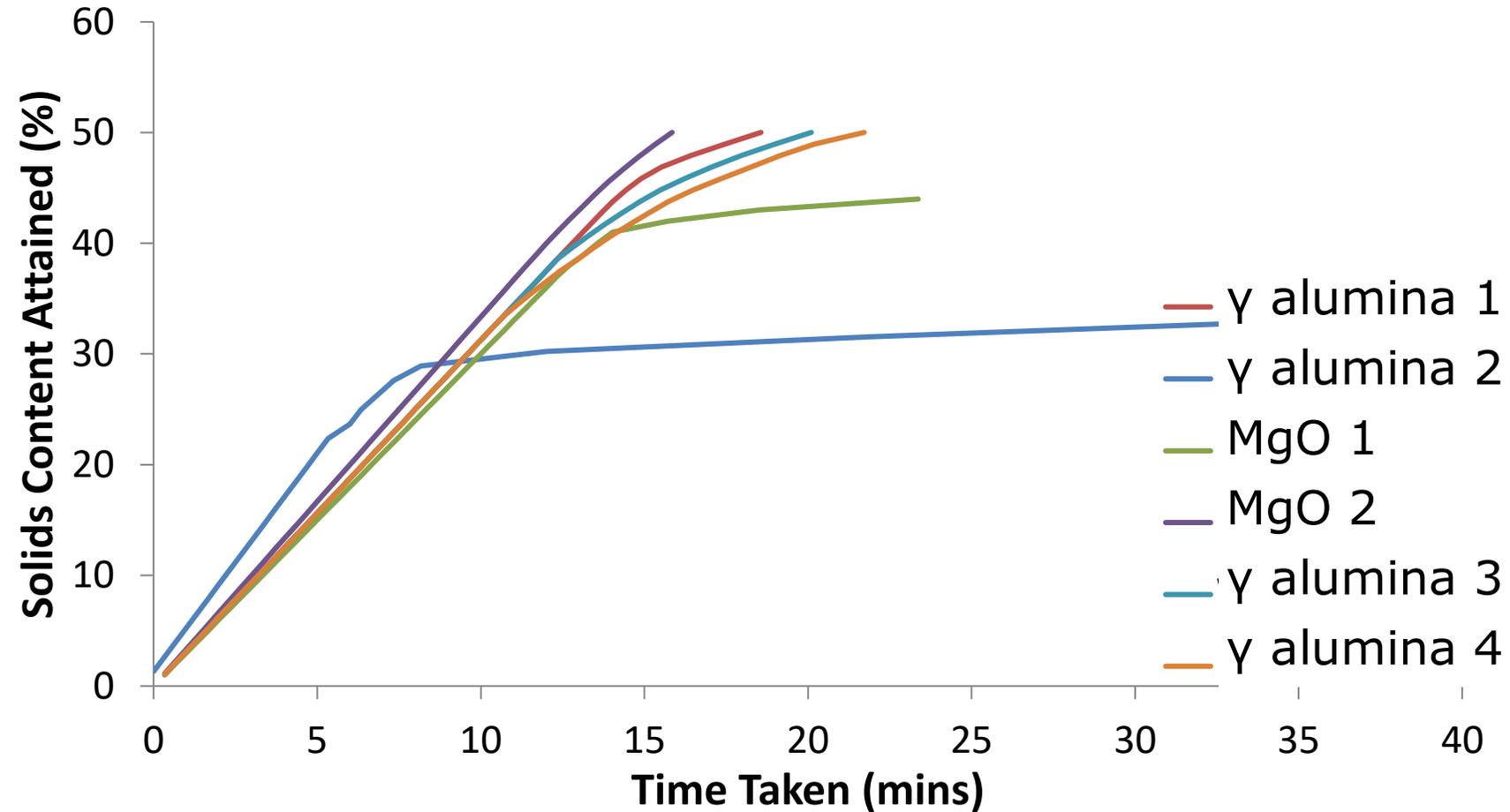
Speed & Power to "Just Incorporate" (PBT DP)



And other powders!

Different powders behave very differently

- Even those with nominally similar composition



Summary

- ❑ Over 50% solids added
- ❑ Fast batching of dense powder in liquid (about 4 seconds)
- ❑ Optimal drawdown design of blade used
- ❑ No transition regime observed from experiments compared to theory

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