

PA11 Powder Flow Characterisation: Assessing Powder Particle Processability

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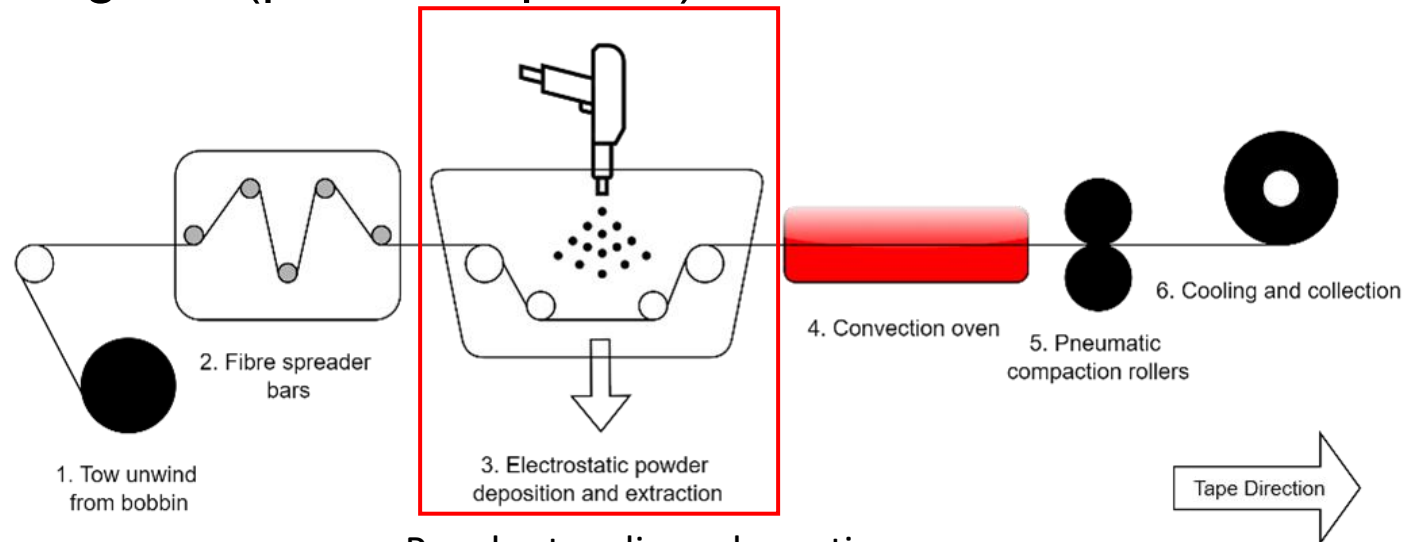
1. Introduction
 1. Broader context
 2. Motivation and aim
2. Methodology and Results
 1. Particle size and shape analysis
 2. Hygroscopicity behaviour
 3. Powder rheology
3. Summary and Conclusion

Broader Context

- **What?**
 - Thermoplastic composite tapes
 - Commonly processed via wet processing (solution, slurry, melt impregnation)
 - Our solution, **dry powder processing for better control ∴ aids impregnation**
- **Why powder?**
 - Better heat transfer → Lower viscosity inhomogeneity → Better consolidation
- **How?**
 - Powder tape processing line (powder tapeline)



Typical thermoplastic tape

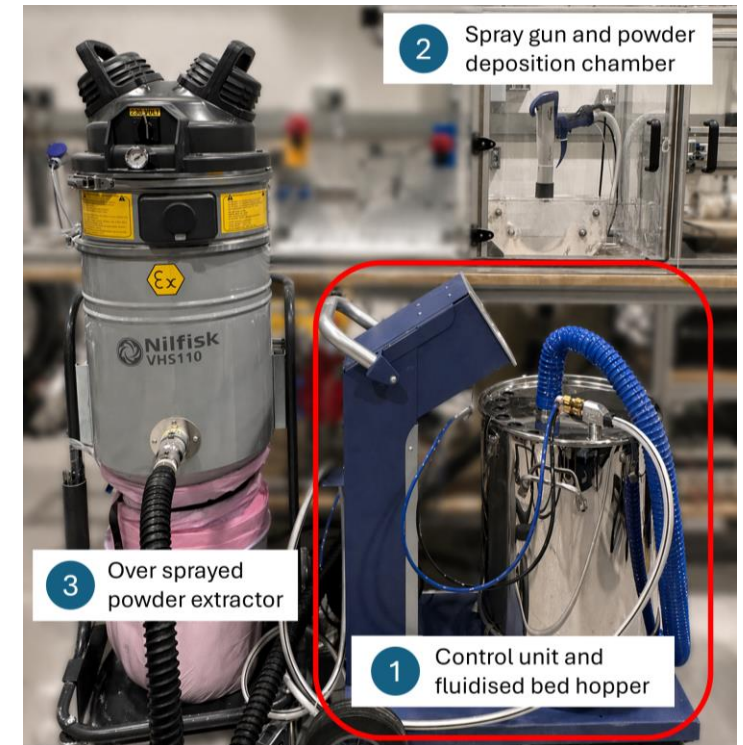
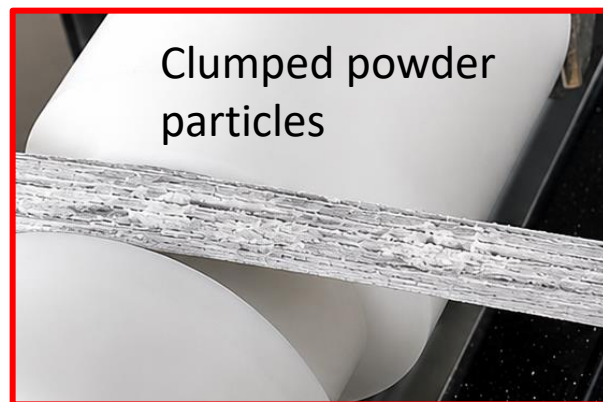


Powder tapeline schematic

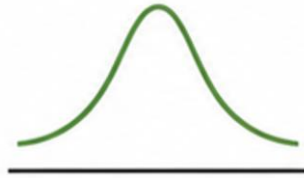
Motivation and Aim

- Powder deposition is a complex system, governed by:
 1. Powder fluidisation and venturi transport
 2. Electrostatic interaction
 3. Wrap-around effect to carbon fibre tow
 4. Over spray extraction
- Powder deposition **influences FVF** from inconsistent powder spray

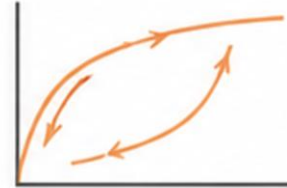
Aim: Robust methodology of powder screening for powder tapeline suitability



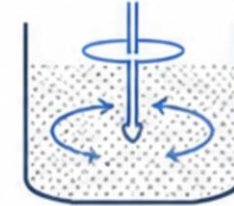
Factors influencing powder flow:



Particle size and morphology



Moisture content



Intermolecular forces/attraction

1 Intrinsic powder characterisation



Particle Size and Shape Analyser

2 Hygroscopicity behaviour assessment



Dynamic Vapour Sorption

3 Flow behaviour assessment



FT4 Powder Rheometer

1. PA11 ES NAT

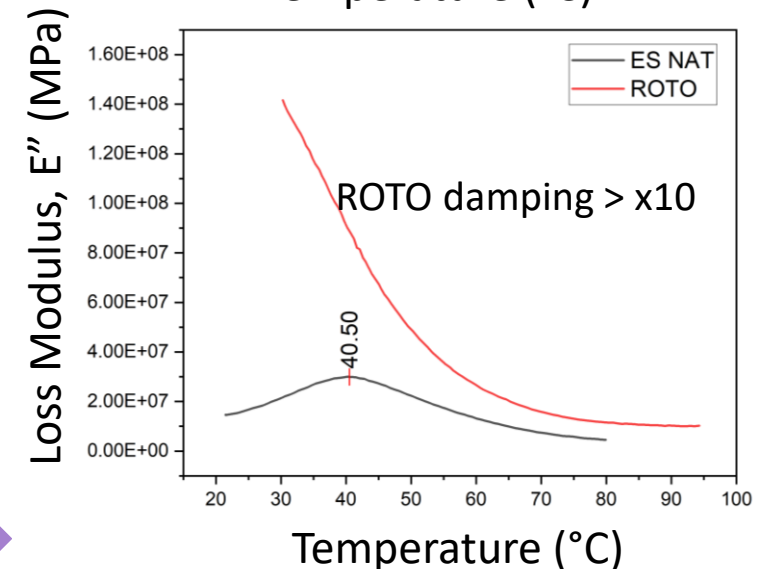
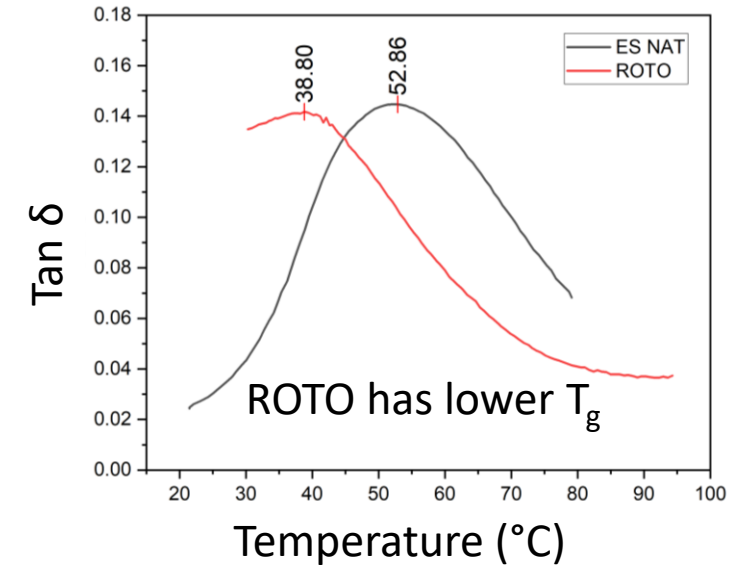
1. $D_{50} \approx 30 \mu\text{m}$
2. Additive free
3. $T_g \approx 52.86 \text{ }^\circ\text{C}$

2. PA11 ROTO

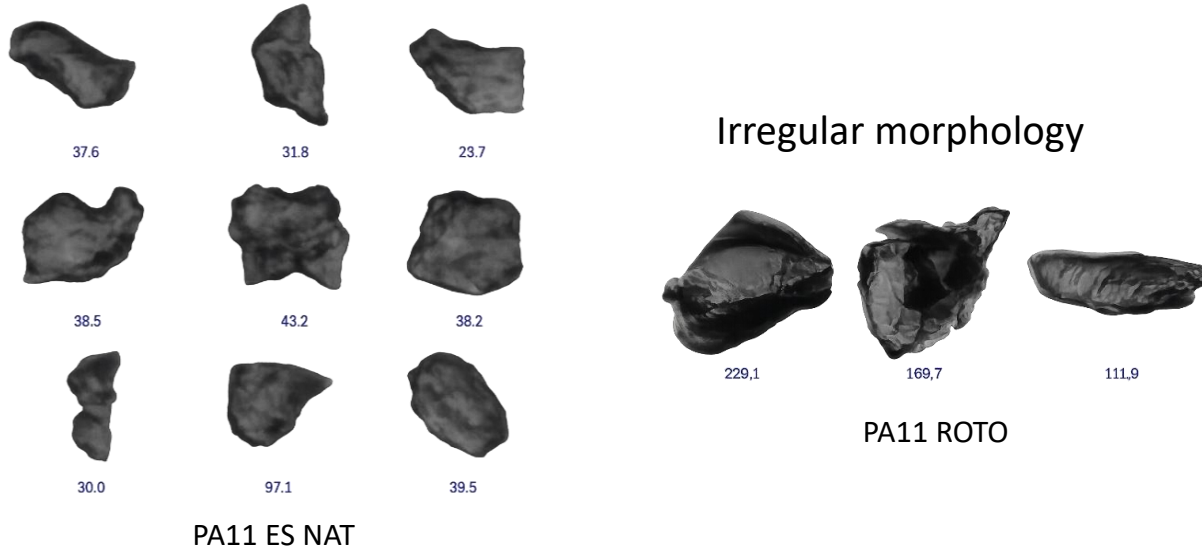
1. $D_{50} \approx 300 \mu\text{m}$
2. Plasticizer, heat stabiliser, light stabiliser additives
3. $T_g \approx 38.80 \text{ }^\circ\text{C}$



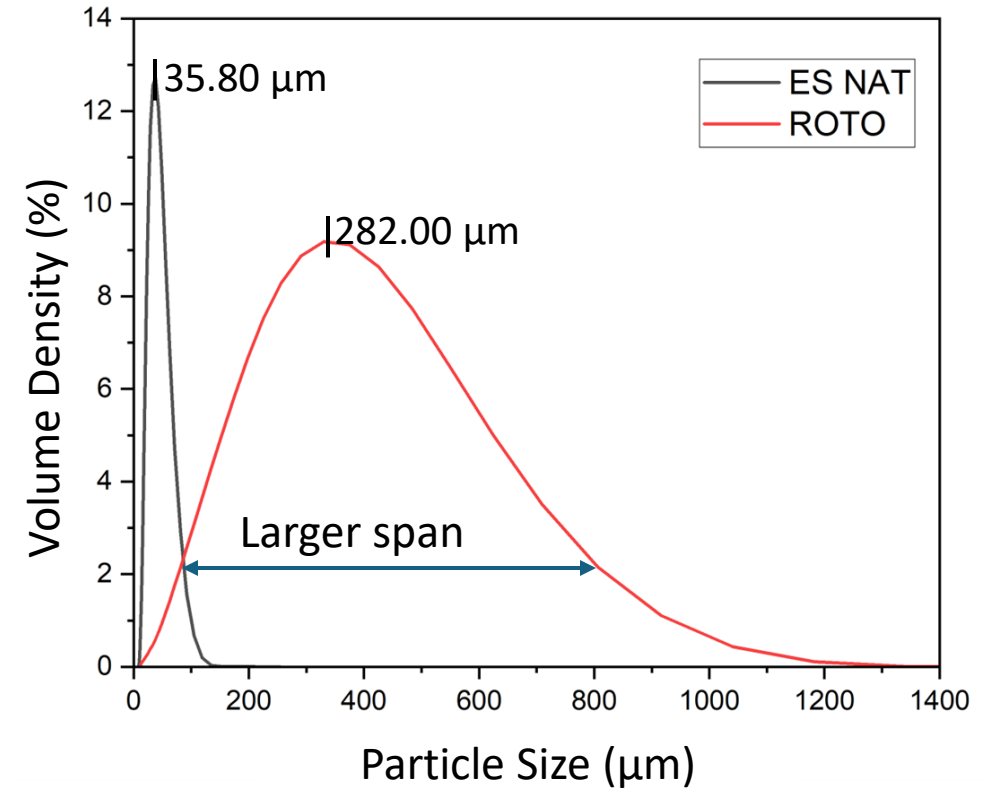
DMA in accordance with ISO 6721:11:2019



1. Particle size and shape analysis



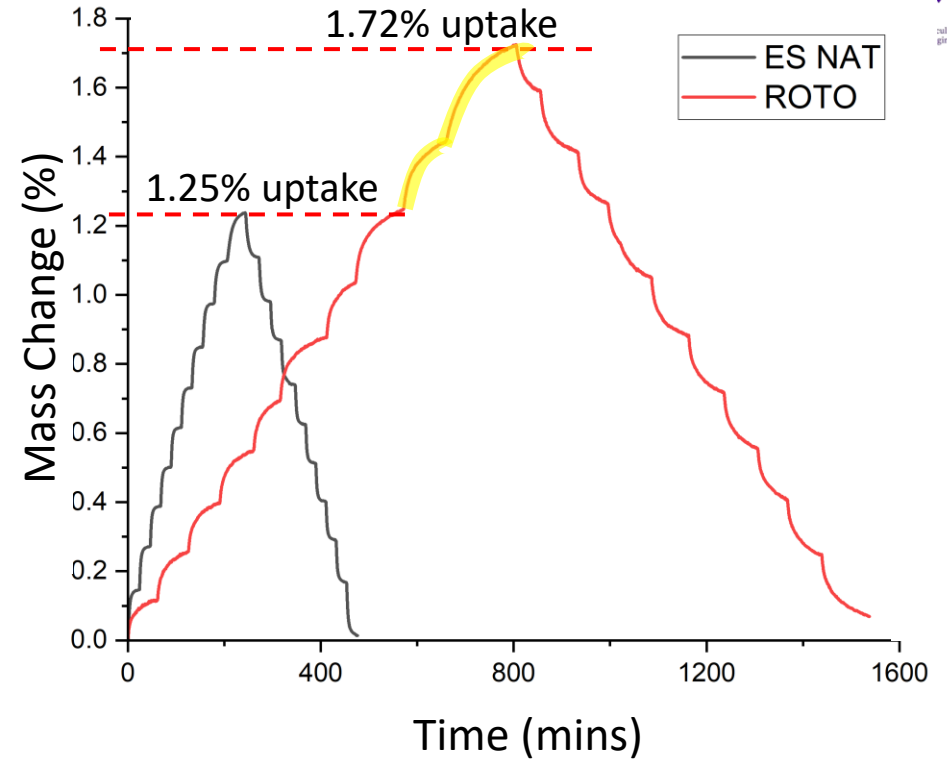
Parameter	ES NAT	ROTO
Volume Mean Diameter (D10), μm	19.6	92.2
Volume Mean Diameter (D50), μm	35.80	282
Volume Mean Diameter (D90), μm	64.4	589
Span	1.25	1.76
Smoothness	0.44	0.35
Circularity	0.60	0.54



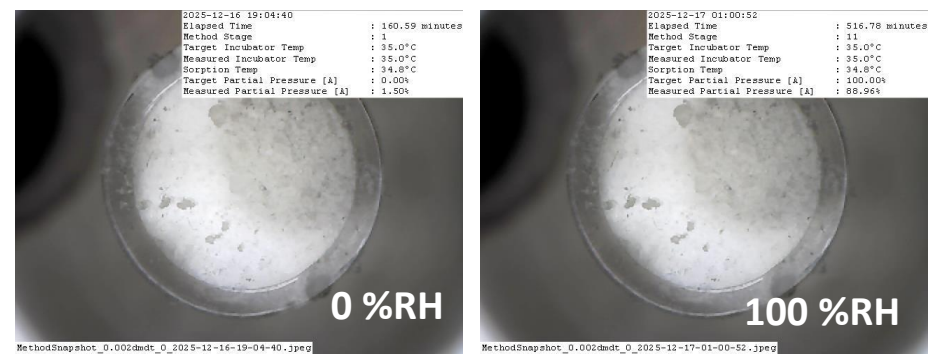
$$Span = \frac{D90 - D10}{D50}$$

2. Hygroscopicity behaviour

- Physical property of material to adsorb or retain moisture
- ROTO has **higher adsorbed moisture, slower diffusion rate**
- Initial increase → surface adsorption**
- Upward sloping → multilayer adsorption**
 - at higher RH%, **higher diffusion** through polymer chain into **bulk adsorption sites**
- Both shows no sudden jump, **no deliquescence** (powder did not dissolve)



DVS curves for PA11 powders at 10% stepwise increments of RH measured over a full humidity cycle (0–100% RH) at 25°C.



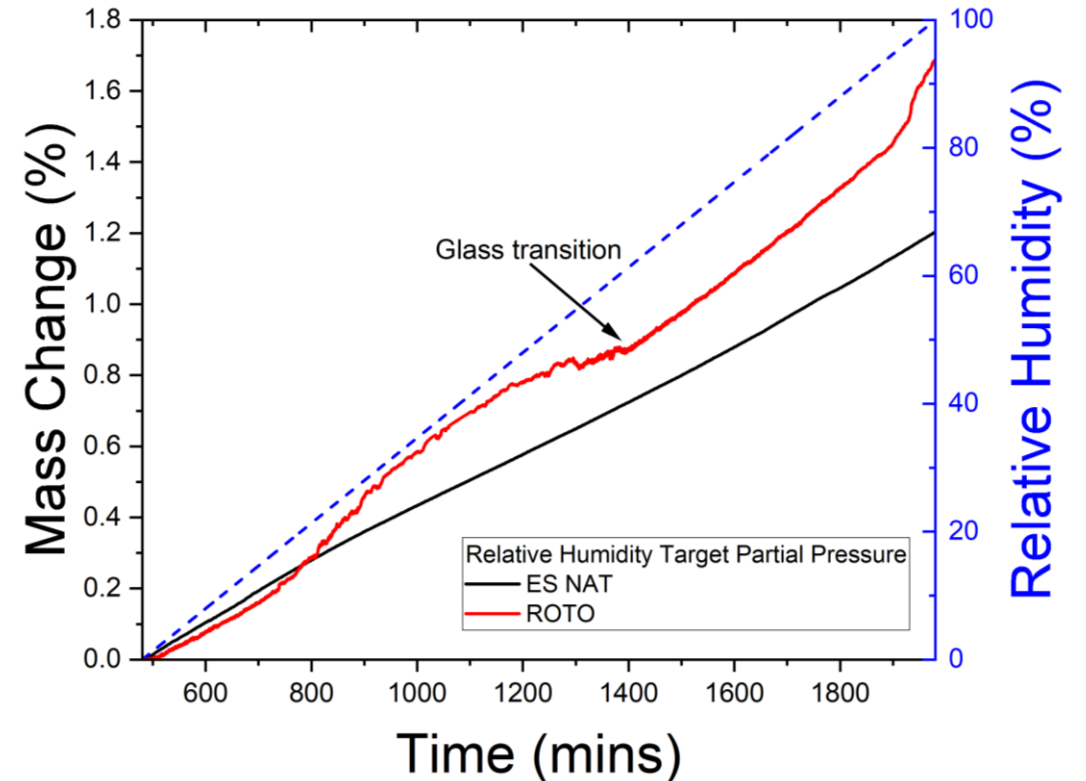
2. Hygroscopicity ramp

- ES NAT smooth → no glass transition
- ROTO slope change → **glass transition** at ~50 %RH
 - **amorphous** region becomes **rubbery**
- **Water acts as plasticiser** for polymer, lowering the T_g
 - Increase **chain mobility** → **promotes water uptake** → steeper slope

Takeaway: ES NAT has a more robust hygroscopicity behaviour

DMA glass transition temperatures, T_g :

- ES NAT; $T_g \approx 52.86 \text{ }^\circ\text{C}$
- ROTO; $T_g \approx 38.80 \text{ }^\circ\text{C}$



Ramp measurement for PA11 powders at 4%RH/h rate from 0–100% RH at 25 °C.

3. FT4 Powder rheology – working principle

FT4 modes of testing



Bulk properties



Dynamic properties



Aeration properties



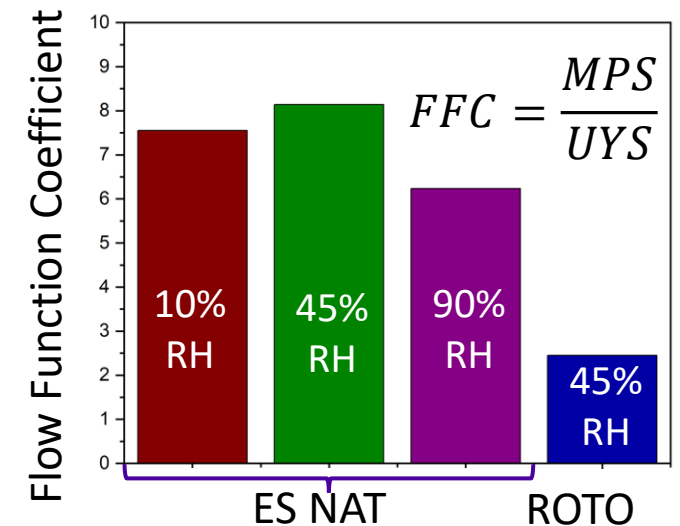
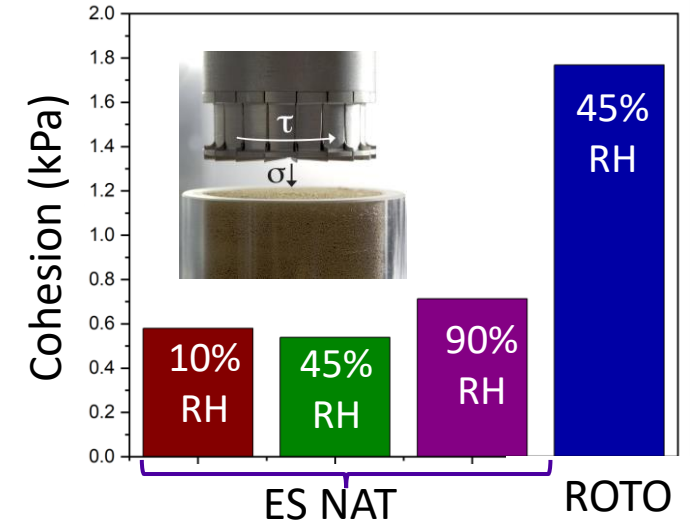
Shear properties

- Characterise **powder flow behaviour** by measuring **energy of powder resistance** under controlled test conditions
- Characterise:
 - bulk properties (compressibility & permeability)
 - dynamic properties
 - aeration properties
 - shear (quasi-static) properties
- **Influence of humidity only on ES NAT**

Conditions	Relative Humidity ($\pm 5\%$)	Duration (Hr)
Low-RH	10–20	24
Mid-RH	50	24–72
High-RH	80–85	48–96

3. Powder rheology – quasi-static properties

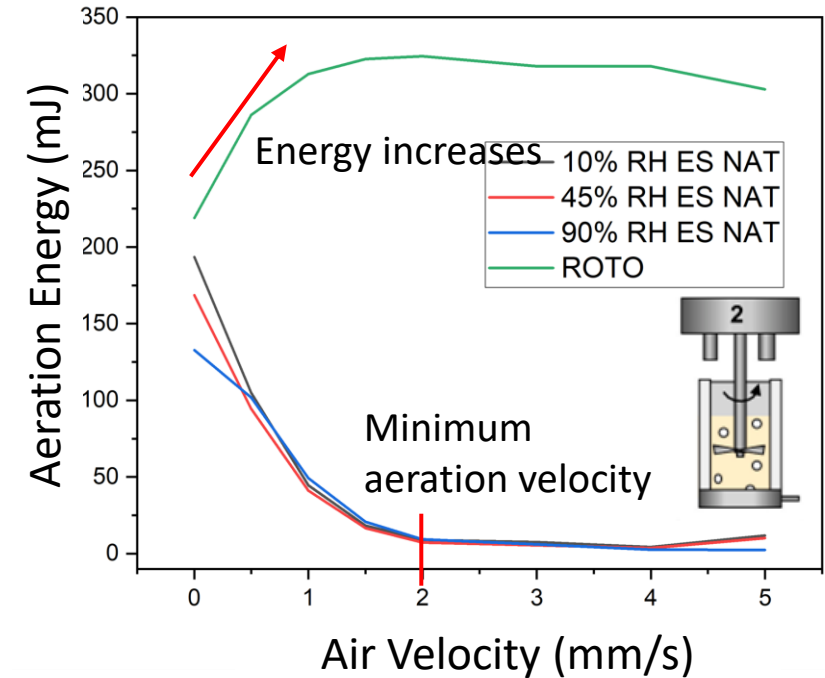
- Quantify **powder strength** under **consolidated powder bed**
- ROTO has higher cohesion (cohesive forces)
 - Softer powder, viscous damping
 - Despite the particle size advantage (classical theory)
 - **Low humidity** has more **static influence**
 - **High humidity** acts as **lubricant**
- ROTO has higher flow function coefficient, **FFC** (industrial relevant dimensionless quantification of powder flow behaviour)
 - ES NAT = easy flowing
 - ROTO = cohesive



3. Powder rheology – aeration properties

- **ES NAT** energy decrease as expected
 - **Fluidise** at ~ 2 mm/s
- **ROTO** behaviour is unusual (**doesn't fluidise**)
 - Poor powder bed expansion
 - Governed by **particle rearrangement**
- ROTO behaviour in line with cohesive powder
 - **Wider PSD** → powder **segregation** and promotes **channelling**
 - **Viscoelasticity** behaviour → softer powder → higher contact area → **cohesion**

Takeaway: ES NAT is a more robust system for powder tapeline

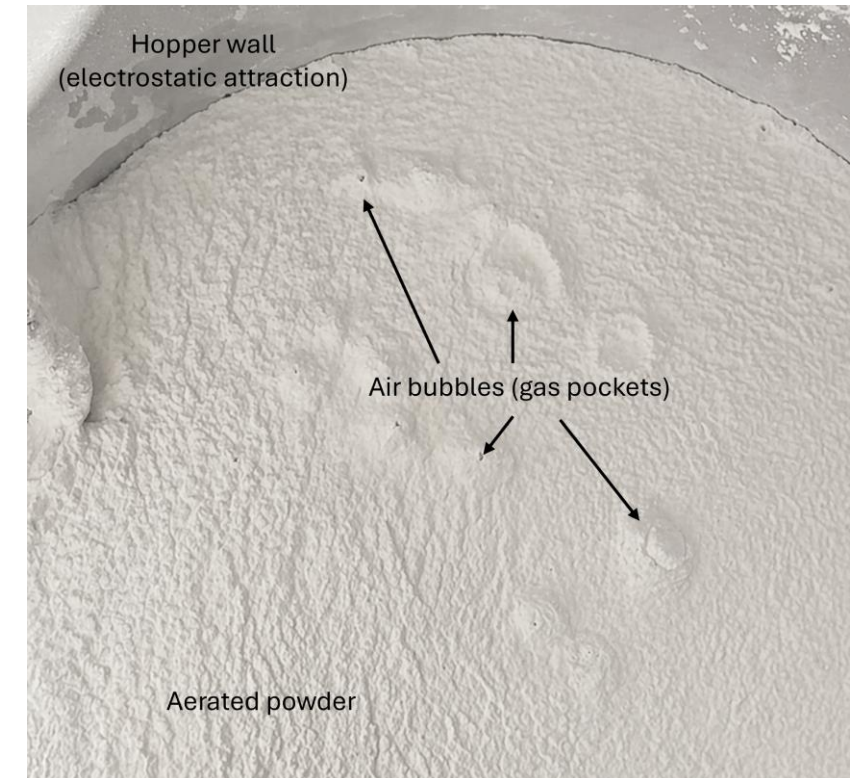


Summary and Conclusions

- Established a comprehensive characterisation framework for understanding powder flow behaviour
 - Influence of particle size **intrinsic properties** (size and shape)
 - Influence of **environmental condition** with hygroscopicity behaviour
 - **Flow performance** with powder rheology
- **Classical expectation** that larger = better flow, **isn't always true**
 - **No single test provide a complete picture** of powder flow behaviour
 - ES NAT is a more robust system compared to ROTO

Summary and Conclusions

- For tapeline powder screening,
 - Hygroscopic behaviour → Shear Cell → Aeration
 - Consistent powder deposition → consistent tape **FVF** → better final part **quality**
- Beyond tapeline,
 - Powder characterisation → reliable **prediction of powder behaviour** → **improves processability**



Fluidised PA11 ES NAT powder in powder tapeline hopper



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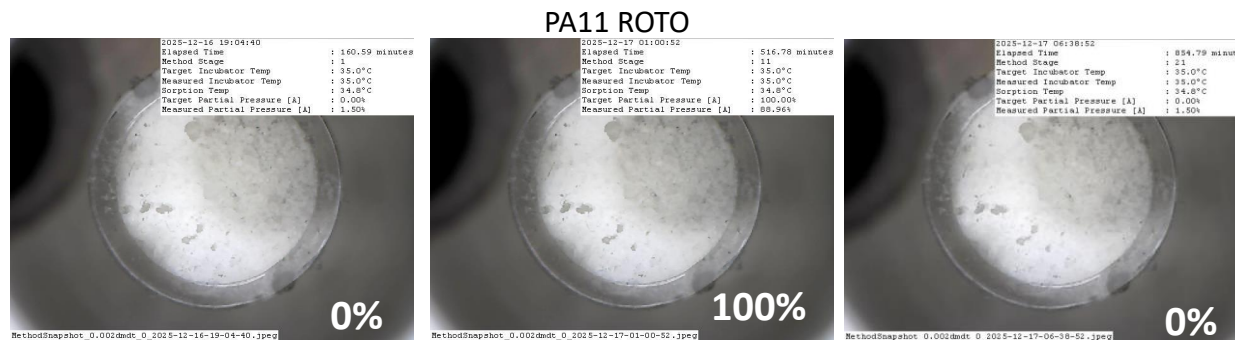
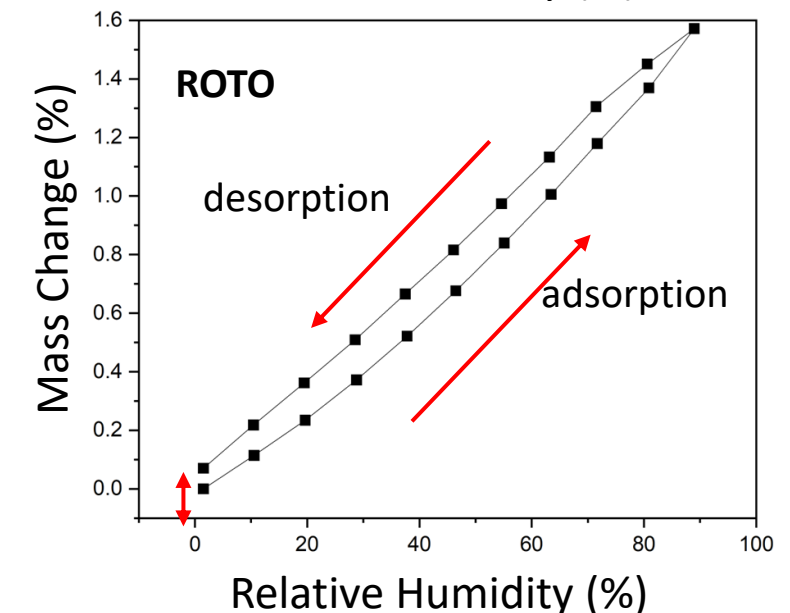
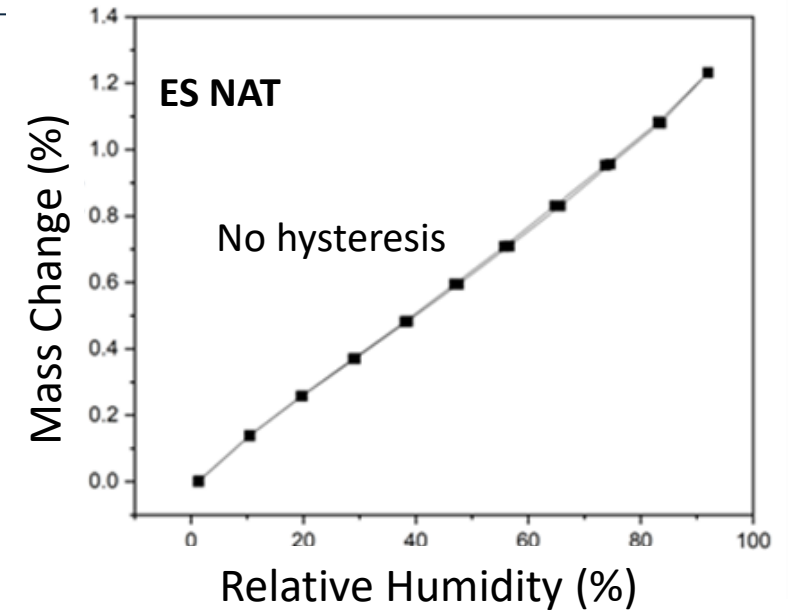


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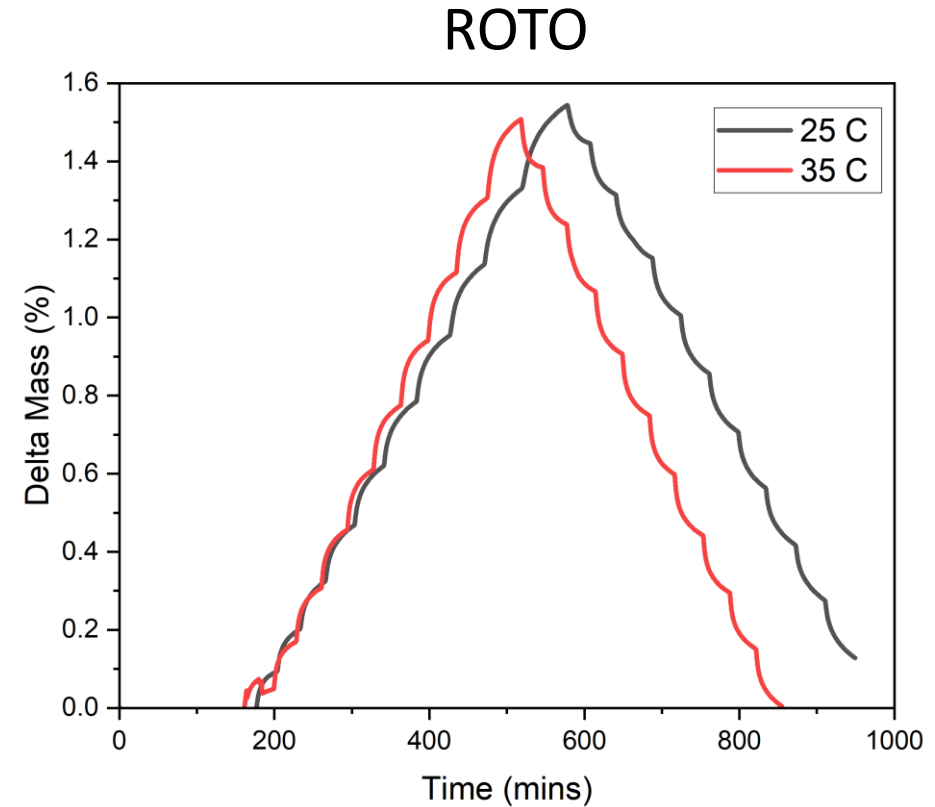
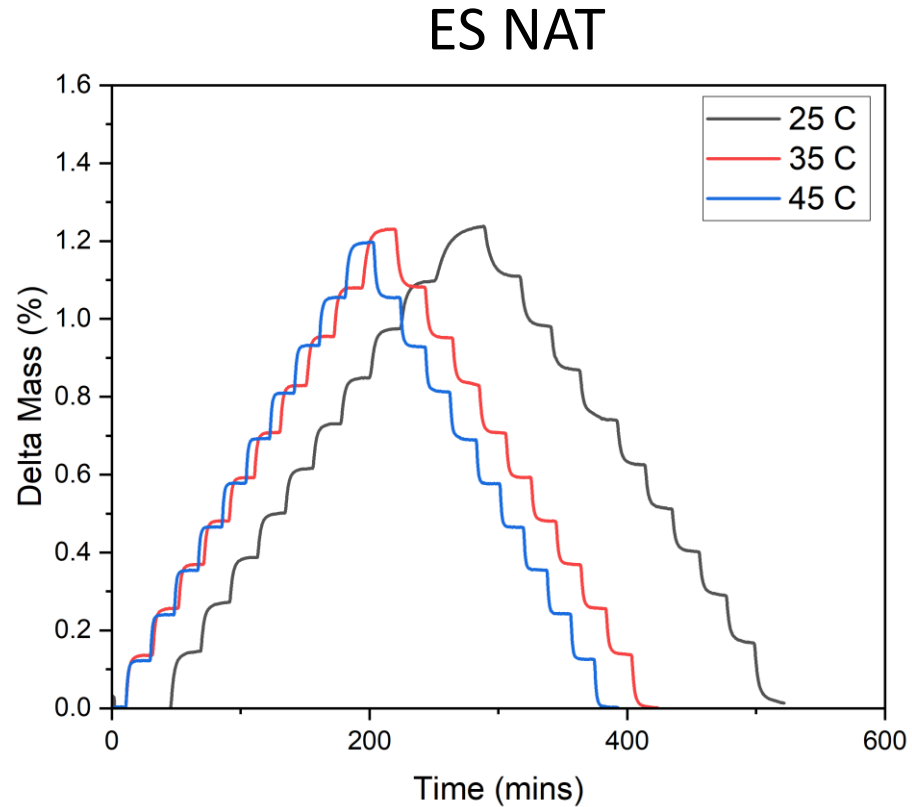


Appex. A1 - Hygroscopicity isotherm

- Type 2 adsorption behaviour
 - Multilayer adsorption**
 - Non-porous/macroporous solid** (common for PA11)
- ROTO exhibit open hysteresis
 - Moisture retention
 - May promote **liquid bridging** under cyclic loading
- No physical change



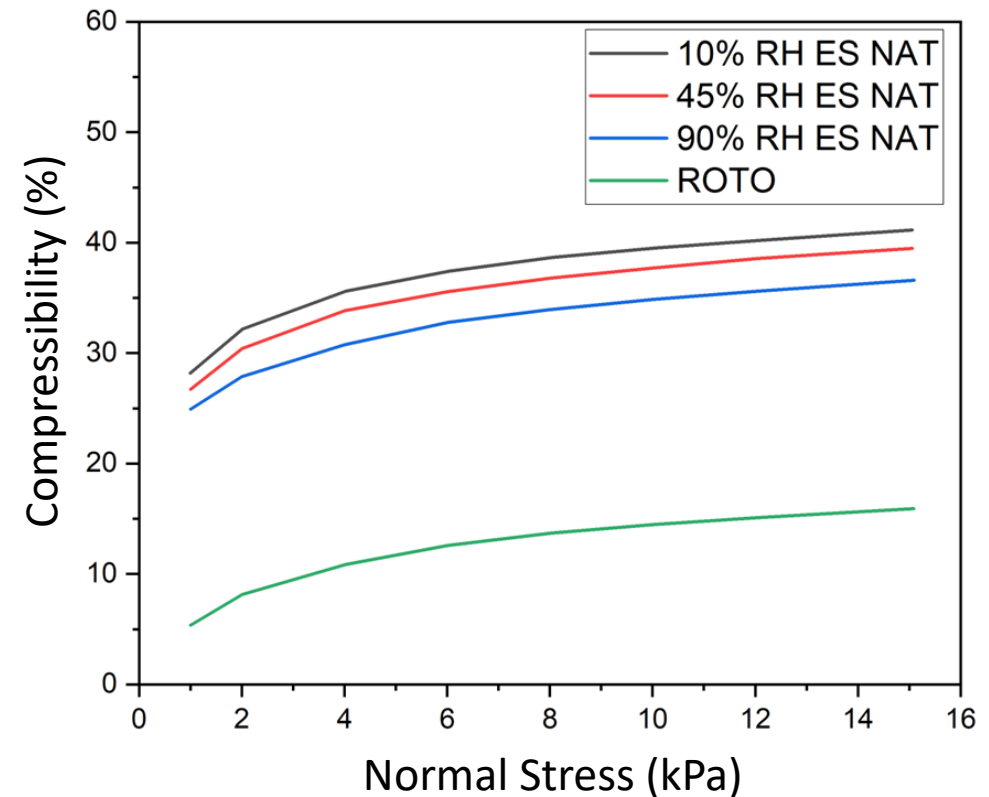
Appex. A2 – Temperature influence (Hygro)



- Higher temperature → faster kinetics
- No sorption behaviour difference at different temperature, despite 45 °C closer to ES NAT T_g

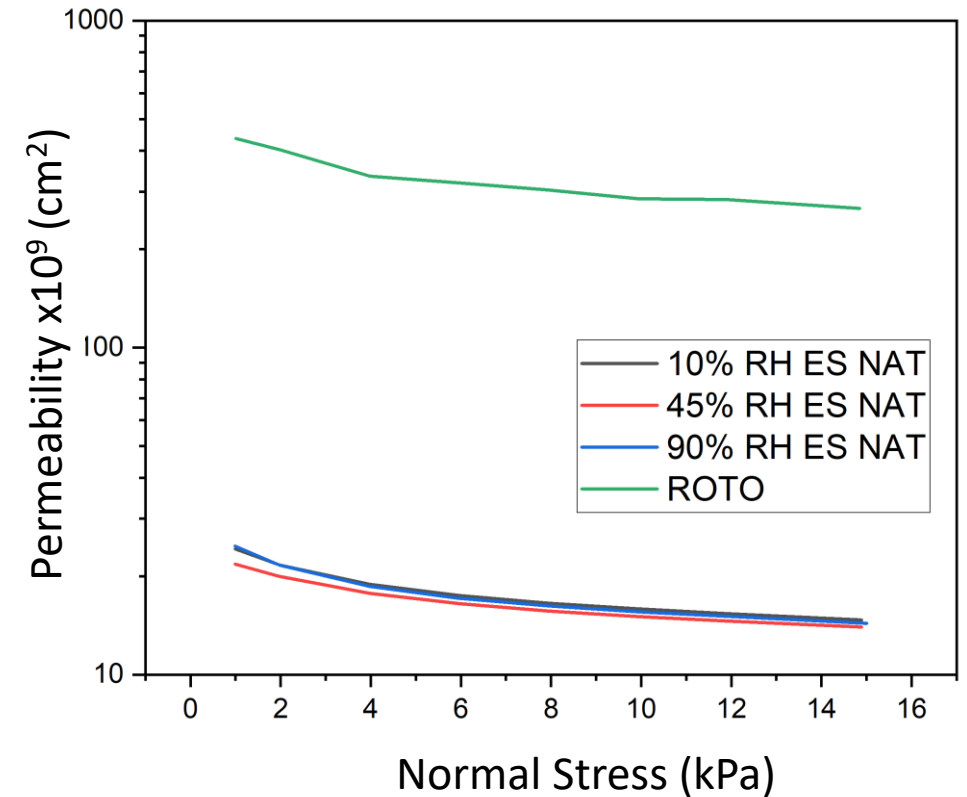
Appex. B1 – bulk property (compressibility)

- Note: Bulk properties **does not directly measure flowability**, but indicates particle cohesive force, particle rearrangement, and pore structure
- **High compressibility → strong cohesion**
 - ES NAT higher compressibility suggests prone to densification → irregular



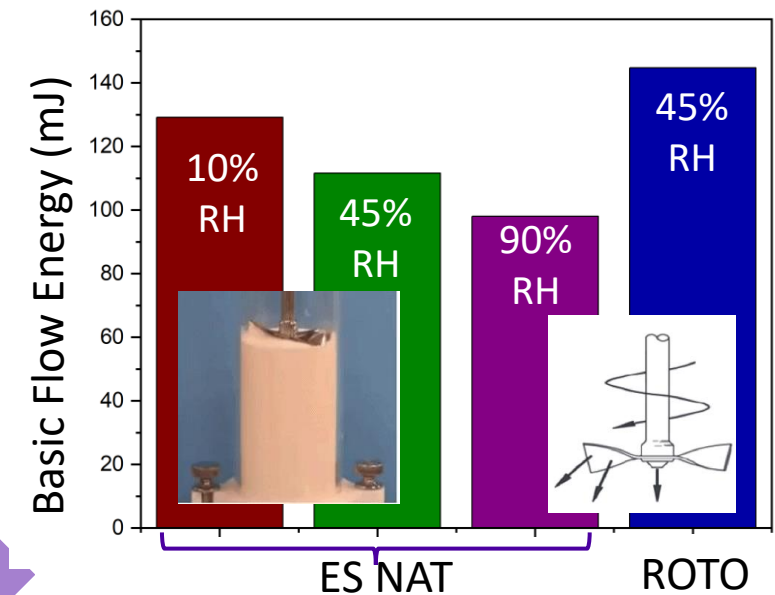
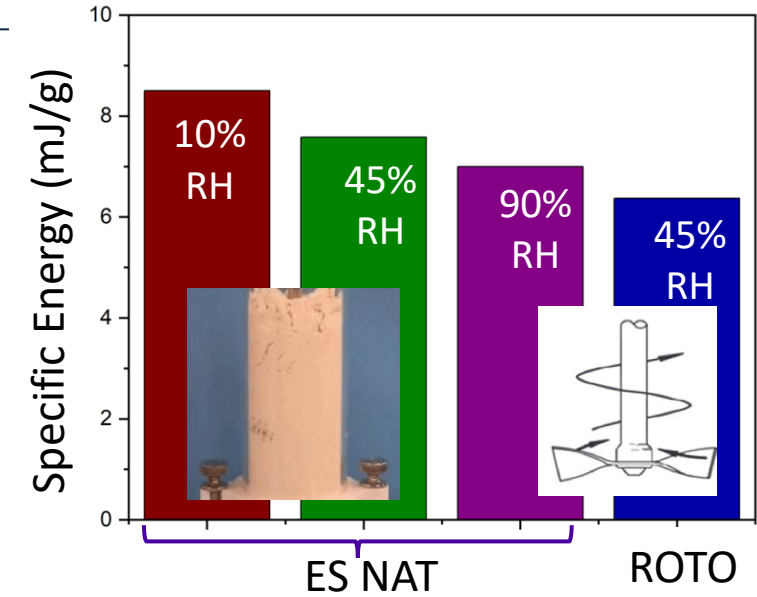
Appex. B2 – bulk property (permeability)

- **High permeability** influences air transmission through bulk → **encourages fluidisation** and venturi transport
 - ROTO higher permeability suggest easier fluidisation
- **Minimal influence of humidity**
- Both properties are in line with classical particle **size expectations**
 - In isolation. ROTO implies easier transp



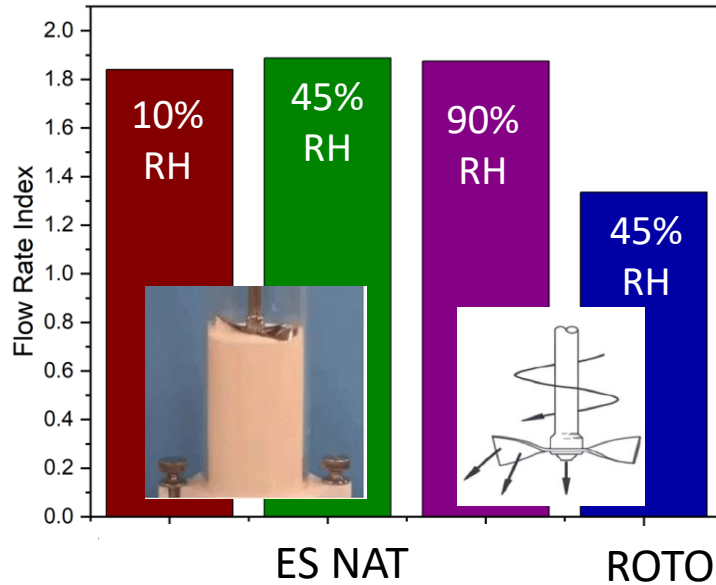
Appex. B3 - Powder rheology – dynamic properties

- Specific Energy
 - Low stress state flow
 - Weakly confined flow condition (upward blade movement)
 - Governed by **particle rearrangement**
 - In line with classical **size-based theory**
 - **Low humidity** has more **static influence**
 - **High humidity** acts as **lubricant**
- Basic Flow Energy
 - High stress state flow
 - Confined flow condition (downward blade movement)
 - Governed by **viscoelasticity damping**



Appex. B4 – Additional Dynamic Properties

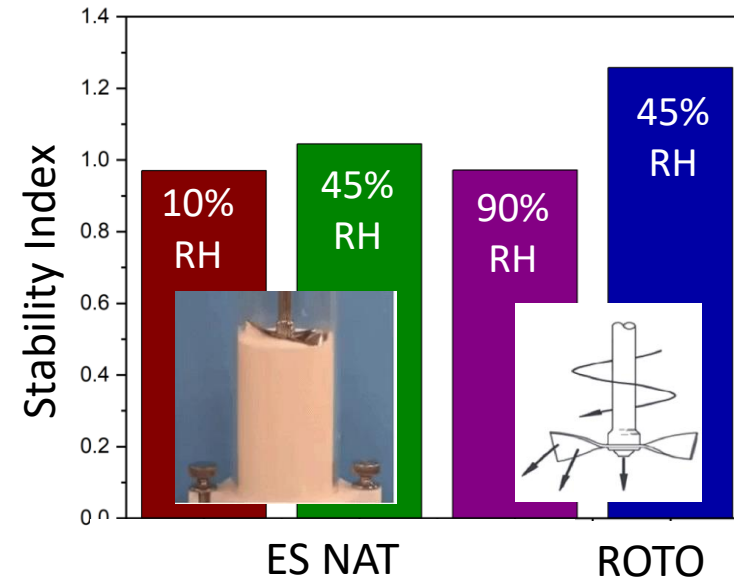
Flow Rate Index



$$FRI = \frac{E_{low}}{E_{high}}$$

- High stress state flow
- Flow rate dependent
- Define sensitivity to cohesion
- Governed by viscoelasticity damping

Stability Index Energy



- High stress state flow
- Sensitivity to repeated test cycle
- Governed by **particle segregation**
- **Across humidity, repeated motion agitates powder**