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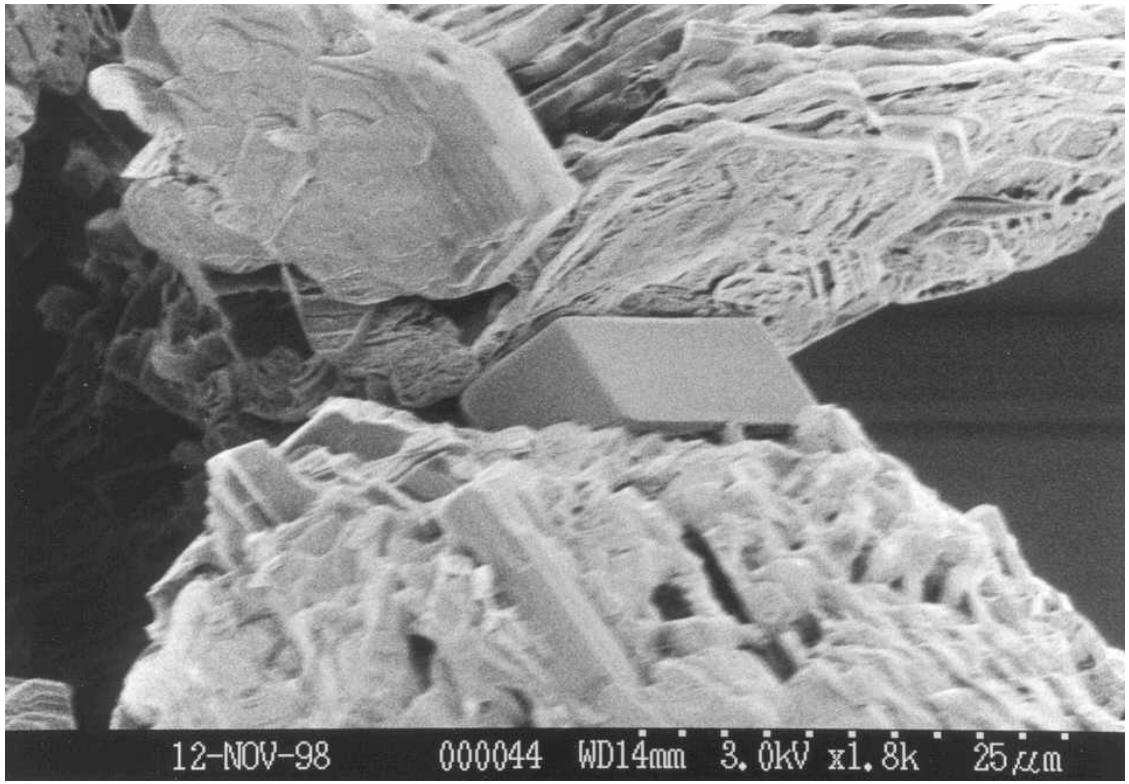
Powder Caking - a review



Jamie Cleaver

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Introduction

Presentation gives structure and outline contents for review

Title 1 Rapid stability test methodologies for granular and tabletted products

Title 2 Powder caking

Identification of issues

- Degradation of granular powder products during storage
- Degradation = loss of functionality
  - Poor flow
  - Poor dispersion/dissolution
  - Poor compaction
  - Loss flavour/texture
  - Microbial growth

Identification of issues

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- Long time scales/slow kinetics
- Simultaneous mechanisms

- Predictive capability very desirable
- Requires
  - Understanding fundamental mechanisms
  - Establishing current theoretical and experimental advances

Objectives of review

- To identify fundamental mechanisms, plus current experimental and theoretical advances
- To review the developments in rapid test methodologies
- To identify potential pathways towards development of rapid stability prediction

Intrinsic material stability

- Chemical transitions
  - hydration, oxidation, decomposition
- Physical transitions
  - dissolution/crystallisation,
  - solid-solid phase transitions
    - Amorphous-crystalline

- Kinetics of transitions and their dependence on temperature and water activity
- Enthalpies of transitions

Particulate characteristics

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- Particle contacts
  - Contact area (elastic, plastic, creep)
  - Sintering
  - Liquid bridges (adhesion/dissolution/crystallisation)
- Microstructure
  - Voidage, coordination number, size distribution
  - Vapour diffusion

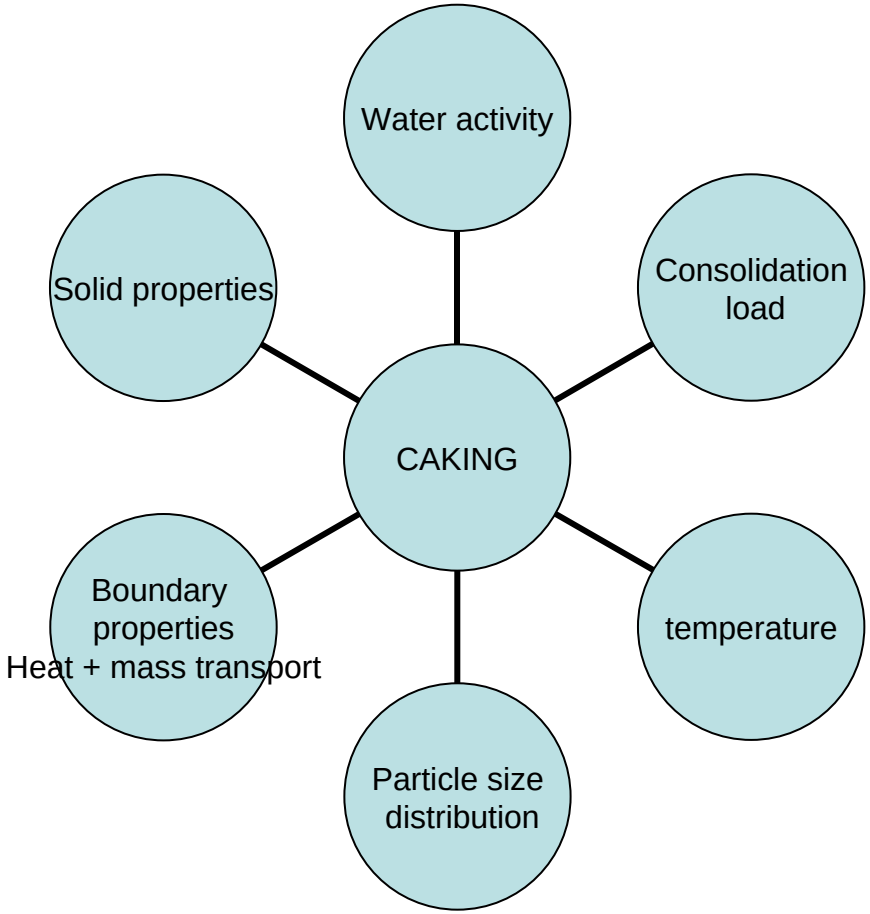
Particulate characteristics

- External consolidation stress
- Electrical effects
  - Electrostatic charging (tribo-charging)
  - Piezo-electric crystals
  - Pyro-electric crystals
  - Ferro-electric crystals

Boundary characteristics

- Sealed or open (mass transfer)
- Thermal insulation (heat transfer)

Caking parameter summary



The caking literature

Systems commonly studied

- Fertilizers (ammonium nitrate, NPK)
- Dairy powders
- Sugar
- Detergents
- Salt

The caking literature

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Cake test methods

- Shear cell tests
- Tensile tests
- Compression tests
- Penetration tests
- Centrifuge tests
- Blow tests
- Creep tests

The caking literature

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Acceleration techniques

Technique	comment
Adding liquid water	Poor distribution, localised dissolution
Elevated temperature	Increase kinetics BUT may change phase, solubility, glass transition etc
Percolation of conditioned air	Good for open systems, no good for closed systems
Temperature/humidity cycling	Limited use due to short times and slow kinetics
Compaction	Useful for ductile or creeping systems

Accelerated tests may not be representative

Related literature

- Tabletting/compaction
- Granulation/Agglomeration
- Filtration

The way forward?

Need to develop predictive capability based on:

- Material phase properties
- Material-water characteristics
- Material mechanical properties
- Multi-component composition
- Particulate properties (PSD, voidage)
- External load and internal stress distribution
- Nature of the boundary

Cool techniques

- Magnetic resonance profiling for water diffusion
- AFM – single particle mechanical properties as a function of RH
- Gravimetric analyser for water sorption
- Dynamic mechanical thermal analysis
- Acoustic probe technique?

Conclusion

- Intrinsic material stability
- Particulate characteristics
- The caking literature
- The way forward

Please give me input!