



Adhesion of powders to metal surfaces during compaction

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Background and objectives	Materials					
Adhesion of powders to metal surfaces during compaction (referred to as "sticking") is a	Material	Formula	Туре	Sticking/ Not Sticking	Grade/Provider	Melting Point [°C]
catalysts etc. Sticking is difficult to predict in early stage of product formulation and process	Ibuprofen	CH ₁₃ H ₁₈ O ₂	API	Sticking	Ibuprofen 50- GMP Pharma Grade (BASF)	50-85
machines such as rotary tablet presses are used. The remedy consists of stopping	Acetylsalicylic Acid (Aspirin)	C ₉ H ₈ O ₄	API	Sticking	A5376 (Sigma Aldrich)	135
	Paracetamol	C ₈ H ₉ NO ₂	API	Not Sticking	A5000 (Sigma Aldrich)	159
builds up on the tooling surfaces again. In pharmaceuticals it is believed that 25% of solid	Mannitol (Pearlitol)	$C_6H_{14}O_6$	Excipient	Sticking	SD200 (Roquette)	166 - 170
dosage forms are affected by sticking.	NEOSORB (Sorbitol)	C ₆ H ₁₄ O ₆	Excipient	Sticking	P100C (Roquette)	97
	Maize Starch B	(C ₆ H ₁₀ O ₅) _n +(H ₂ O)	Excipient	Sticking	Maize starch	257



Objectives:

- Establish a test method to quantify material adhesion on compaction tooling over an industrially relevant range of process and environmental conditions.
- Identify the key factors affecting the amount and/or rate of powder adhesion on 2. compaction tooling including: powder properties (e.g. mechanical, thermal), powder tool interactions (friction, adhesion), tooling materials and finish, compaction parameters (stress, rate) and environmental conditions (temperature, relative humidity).
- 3. Establish a predictive criteria for the propensity of adhesion given a set of molecular/crystal properties and process/environmental conditions.

Sticking hypotheses:

1. Temperature. Sticking can be understood as a coupled thermo-mechanical problem with two sources of heat: 1) Powder compaction involves dissipative processes that generate heat and 2) At the tool interface heat is also generated due to friction. As sticking (gradual deposition of the material to surfaces) progresses the properties of the materials and surfaces evolve, e.g. phase transformations due to stress, strain rate, temperature. 2. Humidity. Moisture uptake by hygroscopic materials leads to water acting as a binder at

the interfaces, leading to sticking.

3. Particle breakage. Brittle materials or granules break during compaction, creating new surfaces. These new unlubricated surfaces give cohesion/strength to compact but also lead to sticking.

Heated die observations

Microcel	(C ₆ H ₁₀ O ₅) _n	Excipient	Not Sticking	MC102 (Roquette)	260 - 270
Lactose Granulated	C ₁₂ H ₂₂ O ₁₁	Excipient	Not Sticking	SuperTab 30GR (DFE Pharma)	202 - 222
Lactose Spray Dried	C ₁₂ H ₂₂ O ₁₁	Excipient	Not Sticking	SuperTab 11SD (DFE Pharma)	202 - 222
Glycolys	(C ₂ H ₄ O ₃) _n Na _n	Excipient	Sticking	Potato starch (Roquette)	-
Solutab A (Croscarmellose sodium)	C ₈ H ₁₆ NaO ₈	Excipient	-	Solutab A (Roquette)	Greater than 90
Magnesium Stearate	[CH ₃ (CH ₂) ₁₆ CO ₂] ₂ Mg	Lubricant	-	-	89

Kelvin probe AFM

Kelvin Probe Microscopy (KPM) is a type of scanning force microscopy in which the contactpotential-difference between two surfaces is measured.



relative to the surface is noticed on all three metal surfaces. These features are NOT powders deposited on the surface.

The potential map shows a significant variation in the potential only for the 5cycles punch. Therefore, the height variations in the 1-cycle punch are surface features and not residues of powder.

Room Temperature	Pressur e [MPa]	Thickness [mm]	Notes	Elevated Temperature	Pressure [MPa]	Thickness [mm]	Notes	NUL. B10 Material Test System
Ibuprofen	20	4.5	Hazing		20	4.5	Major sticking	
	50	4.3	Minor sticking		50	4.3	Major sticking	
	75	4.2	Hazing		75	4.0	Major sticking	
	100	4.1	Minor sticking	Ibuprofen	100	4.2	Major sticking	
	150	4.1	Minor sticking		100	4.1		
	200	4.1	Minor sticking		150	4.1	Major sticking	
	250	4.1	Minor sticking		200	4.1	Major sticking	
	20	-	Tablet not formed	Paracetam ol	75	4.1	Weak tablet	
	50	-	Tablet not formed		100	3.9	Minor sticking	
_	75	4.1	Weak tablet		150	3.9	Minor sticking	
Paracetamol	100	3.9	No sticking		200	-		
	150	3.9	No sticking		200	-		
	200	-	Lamination, electrostatic	- Mannitol -	5 00	-	Allached particles	
	250	-	Lamination, electrostatic		20	-	Major sticking	
	20	-	Hazing		50	-	Major sticking	
	50	-	Minor sticking		75	-	Major sticking	
	75	-	Minor sticking	 Leicester sticking classification No sticking: loose particles on surface Attached particles: removed with lab tissue Hazing: dull appearance, removed with lab tissue 			n	
Mannitol	100	-	Minor sticking				surface	
	150	-	Minor sticking				vith lab tissue	
	200	-	Minor sticking				oved with lab tissue	
Stick	ng o	bserva	ations for Ibupr	 Minor stick removal Major stick 	king: part	ks of mate	rial, solvent removal	
		20 MPa	50 MPa	-	75 MPa		100 MPa	150 MPa 200 MPa
24 °C						10		
50 °C		<i>S</i>						



Clean

1-Cycle

KPM is a potential characterization tool for powder depositions on the punch surfaces.

5-Cycles (the light features are powder depositions)

Particle size measurements



Conclusions and outlook

- A trilayer punch-tablet system was developed for systematic sticking studies. We observed:
- Ibuprofen: sticking at room and elevated temperature.
- Mannitol at room temperature: sticking is influenced by compaction pressure and punch roughness. Further studies needed.
- Mannitol at elevated temperature: sticking.
- Paracetamol: little or no sticking at room and elevated temperature.

- Sticking is present for all compaction pressures at room and elevated temperatures. There is a clear increase in the amount of powder sticking at 50 °C.
- At 50 °C large groups of particles (highlighted in red) are sticking. This feature is not observed at room temperature (assumed to be a consequence of extended plastic deformation).



The KP AFM method was developed to observe sticking. Strain rate was identified as a potential parameter responsible for sticking.

Future work:

- Heated die sticking experiments for: aspirin, maize starch, sorbitol, 2 lactose grades, microcrystalline cellulose.
- Measurement of trilayer interface strength.
- Effect of strain rate: compaction with drop impact tester at Leicester University.
- AFM with particle probe to test against different steel surfaces.

Infrared

Spinning dis

on surface of disk

- SEM/EDX of sticking.
- Rotary friction system.
- **PSD** measurements.
- Investigation of particle breakage hypothesis.
- Investigation of humidity hypothesis.





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