Pressure-Temperature-Humidity-Time

Powders and granular materials are unique in terms of industrial materials in that they can remember their stress and environmental history. In other words, a powder can change depending on how it is handled and stored. For example, if a powder is stored in an industrial tote containing a 1 ton mass, the gas in the powder will be removed (compressibility) and the powder particles may form large particles (agglomerates) due to the pressure acting on the particles. If stored long enough in this way, the powder may actually become a solid (caking). When the pressure is removed, the powder may or may not go back to its original condition before storage.

Typical Storage Tote

Powders with severe memory effects

Temperature and humidity also cause history effects in powders and granular materials. For this reason it is important to study the how pressure, temperature, and humidity effect a material's flow properties over time.

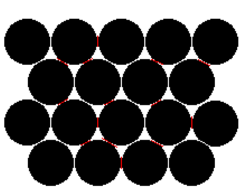
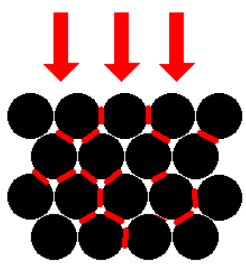
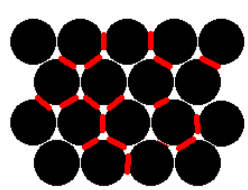
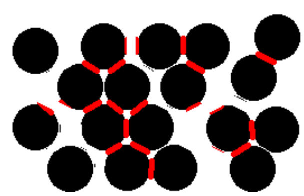
Instantaneous and Stability Tests

Instantaneous powder tests measure powder properties as they currently are in the test powder. Stability tests apply stresses to the samples to determine if powders change with storage, shear forces, aeration, etc. Instantaneous tests are also used to test powder stability when powders are subjected to external stresses before testing (temperature, pressure, humidity, shear).

A large percentage of powder flow problems are caused by instabilities in powders after storage and processing. For this reason, it is very important to know and simulate the storage and processing conditions a powder will be subjected to as it is stored and used. The main causes of powder instability are pressure, humidity, temperature, shear, and impact.

Pressure acts on a powder by pressing powder particles together. This pressure can come in many forms. The most prevalent source of pressure is the powder mass itself. In a storage container, pressure is created by the weight of material over an area of powder. This pressure is a gradient that increases with the depth of the powder bed. For most applications, the maximum pressure is used for testing purposes. For example, for a typical industrial tote, the bed pressure will be from 5-10 kPa depending on the density of the powder. This pressure is calculated by multiplying the mass of material in the tote times the acceleration of gravity and dividing by the bottom area of the tote. In some storage facilities or during shipping, totes are double stacked meaning the pressure will by also doubled.

For some powders this pressure causes irreversible changes in the powder particles. These changes include increases in the effective particle size due to agglomeration and caking, decreases in particle size due to fracturing of particles, and shape changes to to non-elastic compression. Time, temperature, and humidity play very important roles in the degree to which pressure will change a powder. Typically the longer a powder is under pressure, the higher the temperature, and the higher the humidity, the more the powder will be affected by the pressure.

			
Unconsolidated loose bonds between particles	**Consolidated** pressure creates strong bonds between particles	**After Consolidation** bonds must be broken for powder to flow	**On Flow** material breaks along weakest bonds- strong agglomerates remain

Caking due to Temperature, Humidity, Pressure and Time

Mercury Scientific offers two instruments to measure caking in powders and granular materials over time. The Volution Powder Flow Tester and the Evolution Powder tester. Both instruments measure unconfined yield strength but use different methods. The data presented was collected using our Evolution Powder Tester. Similar data can be obtained with the Volution tester.

The Evolution Powder Tester uses uni-axial compression to assess the flowability of powders. The operator begins the flowability test by filling the analysis cell with either 25 cc or 75cc of sample. The cell is then placed in the Evolution and the material is compressed to a predefined pressure. This pressure is referred to as the major consolidation stress. This stress can also be applied using weights over a long period of time to measure the effects of time, humidity, and temperature.

Instantaneous compression on the Evolution Powder Tester

Time Compression in an oven

After compression, the Evolution removes the sample from the cell and applies force to the material until it breaks. The pressure required to break the sample is the unconfined yield strength. The unconfined yield strength represents the force required to make the material flow.

Sample breaking on Evolution

Sample break step with normally installed break cap

A flow factor can be calculated by dividing the major consolidation strength by the unconfined yield strength. The greater the value of the flow factor the better the material will flow at any given pressure. A plot of the unconfined yield strength versus the major consolidation stress is referred to as a flow function and represents the material's flowability under a wide range of pressures.

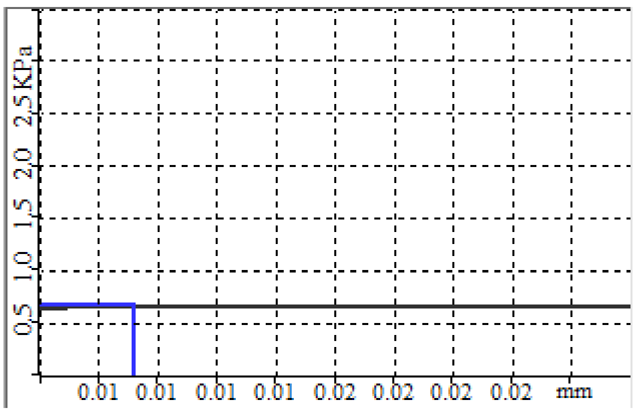
Table 1 presents generally accepted classification of powders and granular materials with different flow factor

Flow Factor	Classification
< 1	Not flowing
$1 < ff < 2$	Very Cohesive
$2 < ff < 4$	Cohesive
$4 < ff < 10$	Easy Flowing
> 10	Free Flowing

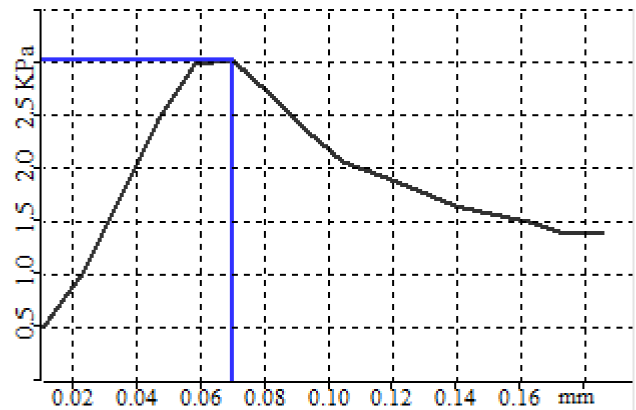
Table 1

Experimental Data- Granular material

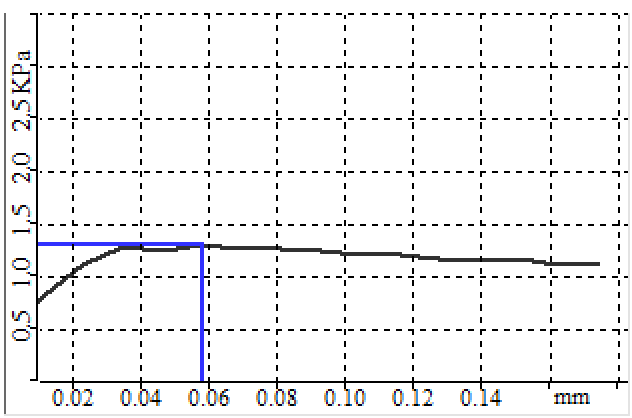
A granular material with 1 millimeter particles was tested for temperature and time effects. Test pressure was 10 kPa to simulate storage in a tote. Break graphs are below.



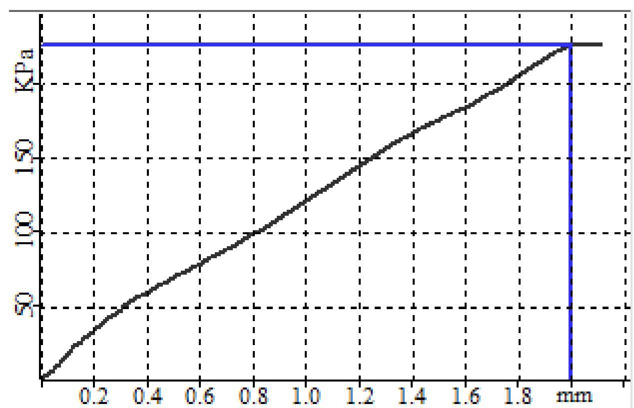
Test 1: Instantaneous Compression
Room Temperature



Test 3: 1 Hr Compression
40 C Dry Compression Then Cooled to Ambient



Test 2 :1 Hr Compression
40 C Dry Tested Hot



Test 4 : 15 Hr Compression
40 C Humid

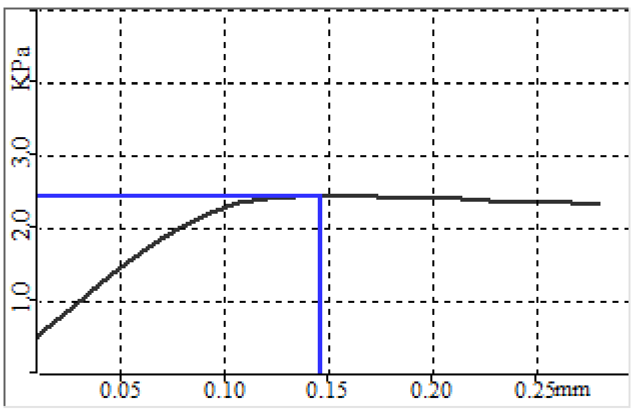
Name	Condition	Strength	Flow Factor	Classification
Test 1	Instantaneous Compression Room Temp	0.66 kPa	15.1	Free Flowing
Test 2	1 Hr Compression 40 C Dry Sample Tested Hot	1.29 kPa	7.6	Easy Flowing
Test 3	1 Hr Compression 40 C Dry Sample Tested After Cooling	3.01 kPa	3.3	Cohesive
Test 4	15 Hr Compression 40 C Humid	226 kPa	0.1	Not Flowing

The granular material starts out as free flowing in Test 1 under ambient conditions and 1 ton tote level pressure. This is typical for granular materials which generally flow very well. After 1 hour of compression under tote level pressure at 40 degrees Celsius in Test 2, the sample moves to the easy flowing category which still represents reasonably good flow behavior. This is important because many materials are used hot. Problems develop however after the sample is allowed to cool in Test 3 after compression at 40 C. The sample moves into the cohesive flow classification which means that bonds have formed between the particles in the material as the sample cooled. This is important because many if not most materials are produced at elevated temperatures and then cool in storage. The sample becomes solid with humidity and temperature in Test 4 where it is classified as not flowing.

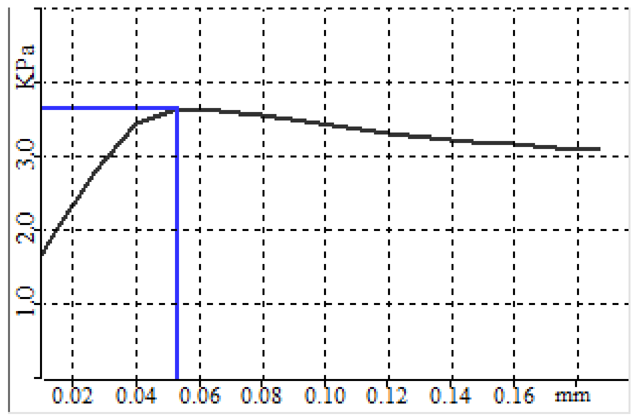
The data for the tested material illustrates how a sample that flows very well can change into a poorly flowing material if handled under the wrong conditions.

Experimental Data- Powder

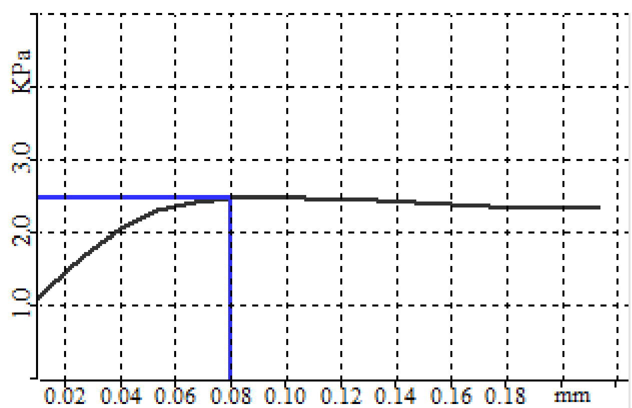
Two similar powders from different manufacturers were tested for pressure over time effects. Test pressure was 5 kPa to simulate storage on single stacked pallets. Break graphs are below.



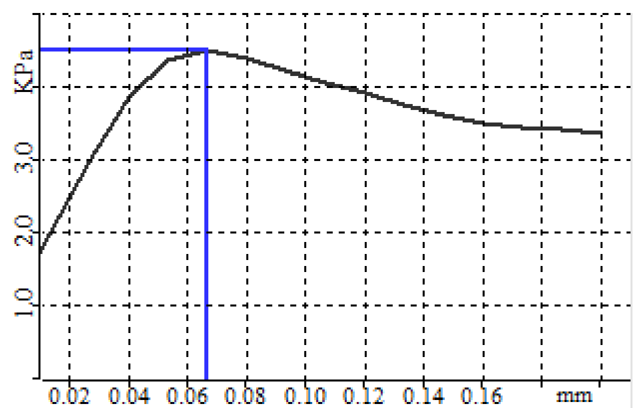
Sample A: Instantaneous Compression
Room Temperature



Sample A: 24 Hr Compression
Room Temperature



Sample B: Instantaneous Compression
Room Temperature



Sample B: 24 Hr Compression
Room Temperature

Name	Condition	Strength	Flow Factor	Classification
Sample A	Instantaneous Compression Room Temperature	2.43 kPa	2.1	Cohesive
Sample A	24 Hr Compression Room Temperature	3.63 kPa	1.4	Very Cohesive
Sample B	Instantaneous Compression Room Temperature	2.48 kPa	2.0	Cohesive
Sample B	24 Hr Compression Room Temperature	4.48 kPa	1.1	Very Cohesive

Under instantaneous compression Sample A and Sample B had similar strengths with sample A having a slightly lower yield strength. After 24 hours of compression, both samples moved from the cohesive to very cohesive classification. In addition, the difference between the two sample expanded to 23%.

The data for the tested materials illustrates how samples that are similar in the short term may preform much differently when stored for long periods. In this case powders with similar specifications had different responses to pressure over time.

Agglomeration due to Temperature, Humidity, Pressure and Time

The Revolution Powder Analyzer is used to study particle agglomeration. The Revolution uses a rotating drum with borosilicate glass sides to measure the flowability potential of powders. The operator begins the flowability test by filling the rotating drum with 100 cc of powder. A motor rotates two high precision silicone rollers that in turn rotate the drum. A digital camera with the assistance of cold cathode back-light illumination takes digital images of the powder during the rotation process. From the images collected, the software measures the behavior of the powder due to the drum rotation and how this behavior changes over time.

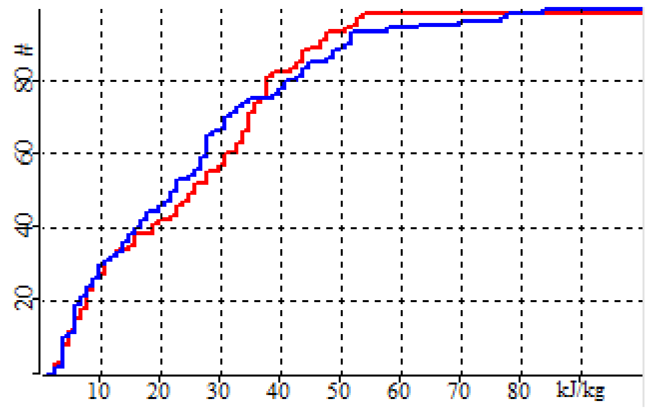
The Revolution measures many flow parameters. Agglomeration is studied by comparing the avalanche energy and dynamic density of samples before and after they have been subjected to storage conditions. The Avalanche Energy is the amount of energy released by an avalanche in the sample powder as the sample drum turns. The dynamic density is the density of the sample as it flows in the sample drum. Storage conditions include temperature, humidity, and pressure exposure.

Filling the sample drum

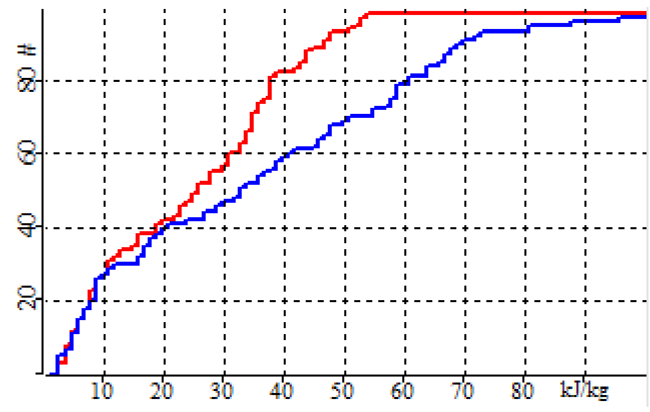
Sample Drum on Rollers

Experimental Data- Powder

Two samples of similar materials from different manufacturers were tested for agglomeration after compression with 15kPa of pressure for 30 minutes. Cumulative Avalanche Energy graphs are below.



Sample C
Blue = Initial Test
Red = After Pressure



Sample D
Blue = Initial Test
Red = After Pressure

Name	Initial Av Energy	After Pressure	Change	Initial Dynamic Density	After Pressure	Change
Sample C	23.9 mJ/kg	23.4 mJ/kg	-2.2%	0.466 g/cc	0.478 g/cc	2.6%
Sample D	32.5 mJ/kg	28.2 mJ/kg	-13.3%	0.412 g/cc	0.445 g/cc	8.0%

Sample C changed very little after exposure pressure. Sample D showed flowability and density changes indicating the formation of agglomerated particles.

The data for the tested materials illustrates that changes in the local structure of granular materials and powders can effect powder flow performance.