

INFLUENCE OF POWDER PROPERTIES ON PREMIX PRODUCTION

A premix is a uniform mixture of micro ingredients along with a carrier or diluents. It is an integral part of poultry feed. It allows small inclusion nutrients to be more uniformly or safely distributed throughout the feed.

The homogeneity, ease of handling and stability of a premix is highly influenced by the powder properties of its various components.

The characterization of powder will give way for opportunities to improve the powder processes while ensuring product quality.

Importance of Powder:¹

- Enhancement of shelf life of ingredients due to absence of water content.
- Simple transport economics.
- Maintains the stability of the ingredient functionality until utilized.

Most ingredients are supplied in powder form & therefore Powder technology is an increasingly important issue both to premix manufacturers and feed millers.

Characterization of powder properties is the measurement and determination of defined powder properties.

An understanding of the properties and processing characteristics of the powders is an essential requirement in

process design, performance improvement and troubleshooting.

Research challenges and opportunities concerned to powders can be broadly divided into two categories:

Processes that give powder its properties namely,

- Production processes
- Mixing
- Agglomeration/Granulation.

Handling and Transport Processes like

- Storage
- Transport
- Packaging Equipment

Powder characterization is a necessity because of its application in quality assurance, process design, trouble shooting and research. Reliable and accurate determination of powder properties is essential to all aspects of premix production.

Various factors that influence powder properties/application are:

- a. Bulk Density
- b. Compressibility
- c. Electrostatic charge
- d. Flowability
- e. Particle size Distribution
- f. Lumpiness
- g. Moisture
- h. Hygroscopicity

Bulk & Tap Density

Is a measure of packing or conversely the amount of space between the particles in the powder. It is dependent upon packing condition, level of aeration, compaction, moisture adsorption and attrition effects. Bulk density is determined by placing a sample of powder of known weight in a graduated

Common Variables Influencing powder behaviour

Particle variables	External factors
Particle size and its Distribution	Flow rate
Shape	Compaction Condition
Cohesivity	Vibration
Particle Interaction	Humidity
Propensity to electrostatic charge	Aeration
Attrition Characteristics	Transportation
Hardness	Container surface effects
Stiffness	Storage time

cylinder. Tap density is determined by tapping the powder in the graduated cylinder until it no longer settles.⁴ True density of a powder is determined using helium pycnometer.

Dividing true density by bulk/tap density yields a number, which is related to the number of free spaces in the powder.

Sphere particles have a value of 0.53. Irregular shaped particles have a value of 0.74 or more.

Application

The particles with high bulk density increases in throughput in the powder plant, bags faster and stack the pallets easier. The flowability of the product will also get increased. Adding suitable carriers like limestone up to 30-40% of the powder mix can increase its bulk density.

Compressibility

It is an index of the stacking behavior or a pressure resistance of a material during storage and transport. Products in powder form tend to cake when bags are stored in stacks. It determines the tendency to

form lumps or cakes when the powder is stacked in a pallet.

When bags containing powders are stacked, pressure of the top bags will be generated on the bottom bags leading to lumping or caking. Certain particle structures and a sticky surface of the particles will contribute to hardening. The liberation of moisture will aggravate the problem by forming bridges between individual particles.

It is tested in the laboratory by selecting a testing arrangement having weight that develops a similar pressure during storage.

The percentage compressibility is calculated as follows:

$$\% \text{ Compressibility} = (P - A) * 100 / P$$

Where P= packed bulk density

A= aerated bulk density

The more compressible a material the less flowable it will be. The borderline between free flowing and non-free flowing is about 20% to 21% compressibility. Percentage compressibility is an excellent indication of uniformity in size and shape, deformability, surface area, cohesion and moisture content.

Lumping or caking due to compression can be reduced by using forms with lesser tendency to lump and by maintaining

hygroscopic, spray dried and starch containing additives separately until adding them directly to the mix.

Electrostatic Charge

It is a measure of the static charges generated in the powder. Bipolar charging, where particles have both positive and negative charges, may lead to opposite charge attraction leading to greater cohesion. On the other hand, unipolar charging will lead to repulsion between the particles, which may increase bulk density and may lead to flooding upon discharge. The smaller the particles size higher the chargeability.

It is observed using a beaker-rod method. In this the powder to be tested is rubbed energetically in a beaker with a glass or hard rubber rod in a circular movement inside the beaker glass wall.

Powders, which become charged electro statically, form a firmly adhesive coating on the beaker wall and on the glass rod. In contrast, powders, which do not develop a charge, do not form a coating.

Compressibility Scale

SCALE	INDICATION
0	Sample collapses completely on removing the cylinder
1	Tablet only collapses when touched
2	Tablet only collapses in lumps when touched
3	Tablet is formed and need gentle force
4	Tablet is formed and needs force to break
5	Formed tablet can be broken only by strong force.

ELECTROSTATIC CHARGE SCALE

Volts/ 10g	Chargeability
<10	Very low
10-100	Low
101-350	Medium
351-600	High
>601	Very High

Influence on mixing

When powders with high chargeability are mixed they have a tendency to cling to all pieces of equipment when they come in contact with bins, augers and mixers. They will demix with excessive mixing since they accumulate very high charges. Particles with the opposite charge will tightly adhere to the mixer. Particles with same charge forcibly separate from each other. Both positive and negatively charged particles lead to a poorly mixed batch with high coefficient of variation.

Influence on agglomeration

Particles with opposite charges attract each other when mixed and leads to the development of Agglomerates. These agglomerates also result in the formation of lumps if untreated.

Chargeability in the raw material can be reduced by granulation. In premixes, adding a liquid binder such as mineral oil at 1-3% can reduce the chargeability.

Flowability

The flowability of a powder is an important quality attribute for ease of handling, processing, and final application. The chemical and physical state of the components in the powder will influence the cohesive nature, stickiness & caking characteristics of the powder, which will influence its flow characteristics. The feed industry demands feed additives and premixes with good

flowability since it reduces labor, improves accuracy and throughput. Flowability is becoming a critical element of quality in a premix.

Flowability is tested using a flowability funnel method and also by angle of repose method.

IMPORTANCE OF FLOWABILITY

- Influence on powder blending
- Influence on finish product quality
- Influence on filling of containers
- Influence on cleaning

Influence on powder blending

Good flowability is indispensable for an accurate metering of ingredients from bins and augers, micro ingredient metering machines. It is also vital for accurate mixing of different ingredients of a powder blend. The uniform distribution of micro ingredients in premixes and feeds is mainly dependent on its flowability. The mixing efficiency of a powder blend is enhanced with good flowable ingredients.

Influence on finished product quality

Powder with poor flowability leads to caking in bins, augers and mixers resulting in poor finish product quality in terms of low assay and presence of mixer scales that separated from the mixer thereby contaminating the ultimate product.

Influence on filling of containers

The unloading of the final product from the mixers to the

ultimate containers is very much influenced by its flowability. Accurate metering of the contents into the representative containers is achievable with good flowable powder.

Influence on maintenance of mixer

Such powders have a tendency to cake and adhere to the mixer thereby increasing the downtime to clean the mixer involving more labor. It increases the cost of production and if not cleaned properly serves as a source of contamination.

EVALUATION OF FLOWABILITY OF POWDER BY:

- a) Angle of Repose
- b) Angle of Spatula
- c) Compressibility

a) Angle of repose

Tan α	Angle of Repose °
0.50	27
0.60	31
0.70	35
0.80	39
0.90	42
1.00	45
1.10	48
1.20	50
1.30	52
1.40	54
1.50	56
1.60	58
1.70	60
1.80	61
1.90	62
2.00	63

Angle of Repose	Flowability
31 and below	Excellent
32 - 45	Good
46 - 56	Medium
57 and above	Poor

It is one of the flowability index. Smaller the angle of repose, greater the flowability. It is measured using the cylinder cone method. Free flowing powders exhibit a high flow rate and a smaller angle of repose.

In order to have a powder of good flowability, it should exhibit lesser angle of repose between 25° and 45°. This can be obtained by controlling the particle size and its moisture content.

b) Angle of spatula⁵

This is a readily determined property that gives a relative angle of internal friction or angle of rupture for a dry material. Any spatula with a 5x1/3" blade, can be introduced into the bottom of a mass of dry material. The spatula is then lifted straight up and out of the material.

It is an indirect measure of cohesion, surface area, size, shape, uniformity, fluidity, porosity and deformity.

The higher the angle of spatula of a material, the less flowable it will be. A material is considered free flowing if its angle of spatula is lower than 40°.

c) Compressibility

The more compressible the powder, the less flowable it will be. The powder with compressibility less than 20% is considered to have good flowability.

Effect of relative Humidity and temperature on powder flowability:²

As the relative humidity of the

Free flow is enhanced by:

Low surface energy	Large particle size
More or less spherical shape	Non hygroscopicity
Minimum electrostatic charge	High Density

surrounding air is increased, powder tends to absorb water, which may form liquid bridges between powder particles & result in greater powder cohesion and reduced flowability.

Increase in the temperature of the powder increases dissolution of particles and this may facilitate changes in crystalline form that result in caking & flow problems. All this process is time dependent.

Effect of Consolidation time on Flowability³

It is observed that increased consolidation time decreases the flowability of powder. The bulk density and cohesion of powder increases during the consolidation time resulting in a more compact and cohesive powder with reduced flowability.

In order to obtain uniform and homogenized powder mix it is necessary to select additives and active ingredients with excellent flowability characteristics.

Particle Size Distribution:⁷

Particle size distribution of a given powder indicates the distribution of particles of varying sizes in microns. It is determined by sieving the powder through a series of standard test sieves.

Particle size distribution indicates how readily the material

segregates in the various components of the system or simply it is the distribution of particles in the powder sample. Effective communication of particle size data will help to define the best powder and provide information for process control.

IMPORTANCE OF PARTICLE SIZE

- ✓ Influence On Flowability
- ✓ Influence On Agglomeration And Caking
- ✓ Influence On Bulk Density

Influence on flowability

Particle size is very important for all the raw materials as it directly affects the flowability of the finished product. In general, smaller particle size leads to poorer flowability and bigger particle size leads poorer dispersibility.

In addition to the particle size, particles should be more or less of spherical shape, smooth and uniform surface to have free flow of the powder.

Influence on agglomeration and caking

If the raw material is more hygroscopic, the smaller particles present in the raw material will absorb the moisture as it has more surface area per unit weight. This will increase the interaction between the particles,

leading to agglomeration and further caking. The smaller particles have tendency to fill the voids of the bigger particles making it more cohesive.

Influence on bulk density

Bulk density varies with changes in particle size distribution and particle shape. When large differences exist between loose and packed values, this indicates the material is more compressible or pressure sensitive.

So, it is a need to fix the particle size that should be large enough, having uniformity and spherical in shape to produce a powder with good flowability and low dustiness and small enough to give good dispersibility and uniformity.

Moisture

Moisture is a very important factor of all raw materials and finished goods. It affects an ingredients nutritional content and its performance during handling, storage and processing. It directly indicates the tendency of particle to agglomerate, to charge or to lump.

The finished product moisture mainly depends on the moisture of the raw materials used. If it is on the higher range, then it brings about the agglomeration, electrostatic charge and lumpiness. The premix ingredient should have a moisture $\leq 5\%$ to reduce the potential of oxidation-reduction reaction.

The moisture content is

determined indirectly using a moisture meter or directly by an oven method. The temperature is usually selected based on its sensitivity to temperature.

Hygroscopicity

It is the potential of a material to absorb water. Particle size and structural appearance of samples play important role because of the surface/weight ratio. The smaller the particle size the more absorptive the product is because there is more surface area and more interaction between particles.

To test the hygroscopicity, the material is exposed to an environment of 90% relative humidity over a period of time (minimum a day). If the material is hygroscopic, the sample weight increases from the moisture taken up in the course of time. This can induce changes in physical appearance due to chemical interactions, crystallization as well as dissolution.

Influence on flowability

The hygroscopic raw material absorbs the moisture and will

increase the interaction between the particles. This in turn lowers the flowability and leads to caking. So, proper storage conditions are necessary; which if fails dampens the product.

Influence on stability

Absorption of water by the premix results in significant chemical interactions like redox reactions between the different components of the mix leading to chemical incompatibility. Mixing of hygroscopic materials also bring about crystallization, recrystallization and dissolution ultimately affecting the stability of final product.

Hygroscopicity problems can be solved by using less hygroscopic forms, i.e. preferring spray-dried forms to crystalline forms. Carriers having $<5\%$ moisture and use flow agents to remove moisture from the medium.

Lumpiness

It is a measure of the tendency of the materials to solidify on exposure to heat with limited exposure to external moisture.

In premixes and blends some

HYGROSCOPICITY SCALE:⁴

SCALE	INDICATION
0	Unchanged.
1	Sample is dry and firm.
2	Surface of sample is moist and sticky.
3	Sample is moist and sticky; surface forms a crust.
4	Sample is moist and lumpy.
5	Sample is moist and forms a solid lump.
6	Sample solidifies to form a cake
7	Sample is partly liquefied.

Lumpiness scale:⁴

SCALE	INDICATION
0	Product flows as freely as starting material.
1	Product flows freely when tapped.
2	Product flows freely when tapped; a few agglomerated particles are observed.
3	Product barely flows when tapped, lumps are observed.
4	Product does not flow, when tapped and removed from the vials the sample falls apart in soft lumps.
5	Product does not flow at all; sample has formed one solid cake.

feed additives become chemically reactive in the presence of heat and moisture. In a closed container elevation of temperature such as during transport or storage in hot weather will release bound moisture to carrier or other feed additives, which in turn causes caking of the powder.

It is observed using a simple vial filled with the product and maintained at 50°C for 24 hours.

Lumps and cakes reduce finish product quality due to poor appearance and assay below claim. Using forms with least tendency to lump can reduce lumping in premix and maintaining all feed additives with tendency to lump separate and added directly to the final feed. It is also necessary to

maintain optimum temperature and humidity to prevent lumping.

Conclusion

In the world of powders there is a multitude of property definitions and measuring techniques, which may vary according to its applications. There is a need to standardize the definition and measurement of these properties. This will help to streamline the quality of powder and obtain greater reproducibility.

The recognition of the importance of powder science and technology is essential for the efficient handling of powders. This aids in solving most of the problems encountered in various powder processes.

References

1. Powder Research to promote competitive Manufacture of added value food ingredients. Strategic Document for Research in Food Powders, March 2003. P-5.
2. Effect of relative humidity and temperature on food powder flowability. E. Teunou, J.J Fitzpatrick. Journal of food engineering 42 (1999) P 109-116.
3. Effect of storage time and consolidation on food powder flowability. E. Teunou, J.J Fitzpatrick. Journal of food Engineering 43 (2000) P 97-101.
4. BASF, KC 9617, Physico-chemical properties of vitamins, trace minerals and carriers.
5. Quantitative evaluation of feeding different physical forms of stabilizers. Ivan Saenz et.al. Polymer Additives.
6. Ralph L. Carr, Classifying flow properties of solids, chemical Engineering, Jan 18, 1965.
7. Remington: The science and pharmacy 19th Edition, vol II Pg 1602.



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