Post-Harvest Operations and Processing of Fruits, Vegetables, Spices and Plantation Crop Products Professor H. N. Mishra Agriculture and Food Engineering Department Indian Institute of Technology, Kharagpur



Hello, everybody, namaskar. In the second part of lecture on powders and premixes today we will discuss some of the major issues related to food powders, the phenomena of agglomeration and granulation in the powders, powder mixing, then properties of the food powder, physical, chemical, functional properties, handling of food powders and premixes. Finally, we will also discuss some important aspects of packaging and storage of food powders.

ſerminology	Particle size (µm)	Characteristics	Examples
Powder	Fine 50–200	More cohesive, high bulk density	Coffee powder, spice powders, colorants
Granules	Coarse 200-4000	Free flowing, low bulk density	Instant coffee
Flour	100-5000	Irregular particle shape, larger size, low fluidity	Nut flours
Dust	5-100	Finer than the desired size, normally fly during processing and handling	Any powder, tea dust

So, in the first part of the lecture, I told you that depending upon the particle size the grounded materials are given different terminologies. Like, the powders have the fine particle size in the range of 50 to 200 micron. Their characteristics is more cohesive. They are high in bulk density and the examples of such powders may include coffee powder, spice powder, colorants and so on.

The granules are coarse in the particle size and their size may be 200 to 4000 micron. They are generally free flowing and have low bulk density. The example of this may be instant coffee. Flour have a particle size in the range of 100 to 5000 microns. They have irregular shape, larger size and low fluidity. And the nut flours are the examples of this.

And then the another category is the dust. They are very fine powders. Size may be up to 5 to 100 micron. They are finer than the desired size, normally fly during processing and handling. And any powder, normally the by-product of the process like in the tea industry, is a finer and dustings. They are tea dust, very fine powders.

Major issues related to food powders

- Most food powders are not directly consumed; they are usually mixed with water and other liquids or solids to produce wet or dry formulations (premixes).
- Prevention of contamination with microorganisms and undesirable chemical components is a huge issue for food powder manufacturers.
- Other issues while delivering the food powders include the ability to handle and transport ingredient powders, dust problems, dust fire and explosion hazards, allergy problems, creation of desirable powder particle properties, and the ability to dissolve these powders when required.

So, the major issue related to the food powders include, as you know, most of the food powders are not directly consumed. They are usually mixed with water and other liquids or solids to produce wet or dry formulation like premixes, So, the prevention of contamination with microorganisms and undesirable chemical compounds is a huge issue for food powder manufacturers.

Other issues while delivering the food powders include the ability to handle and transport ingredient powders. If many powders are used as ingredients in various food process operations, so how to handle and transport this. Dust problems. Dust fire and explosion hazards, allergy problems, creation of desirable powder particle properties, and more importantly, the ability to dissolve these powders when required. So, these are some of the issues that need proper consideration during manufacturing of these food powders, particularly, fruits and vegetable powder.



Agglomeration. It is a very important phenomena. It is an unit operation during which native particles are assembled to form bigger agglomerates in which the original particle can still be distinguished. Agglomeration is basically a physical phenomena and can be described as the sticking of particular solids which is caused by short range physical or chemical forces among the particles themselves as a result of physical or chemical modification of the surface of the solid.

The main purpose of agglomeration is to improve certain physical properties of food powder such as bulk density, flowability, dispersibility and stability. Agglomerated products are easy to use by the consumers and hence are preferred over the traditional non-agglomerated products that are usually non-flowable in nature.



Industrial agglomeration occurs in three zones in fluidized bed, spray dryer, and you can see here in the figure, a schematic of a spray drying. So, normally for production of the agglomerates, spray

drying, fluidized spread irons are more commonly used. So, you can see here in the Zone 1, weak agglomerates are formed by the droplet coalescence and contact between the wet droplets and fine particles.

Then, the particle further comes down in the Zone 2. Here, fine dry particles recirculate before exit so that sticky and dry particles can collide and form agglomerates. Finally, in the Zone 3, there is a formation and stabilization of final agglomerate. So, air temperature and velocity can be manipulated to affect the agglomerate structure through further granulation.



The particles size or the appearance of agglomerates are generally classified in the three forms, depending upon their particle size and how they are formed, how they are assembled together. And they may be onion, raspberry and grapes. Grapes may be compact grape or lose grape, as you can see here in the figure.

The onions are formed when small droplets of high moisture content with fine particles is spread over the surface. Raspberry is formed when the large droplets of high moisture collide with large amount of fines. The fines adhere to the surface but do not penetrate the droplet, you can see here, the raspberry. So, they are adhering on the surface.

The grapes are formed by the collision of similar quantities of droplets and fine particles. So, normally, that if you see that onions has maximum bulk density whereas the loose grapes has the minimum bulk density. Onions has the highest mechanical stability whereas the loose grapes has the lowest mechanical stability, and raspberry and compact grapes are in between. So, higher moisture content droplets result in compact structure and high mechanical strength. The optimum dissolvable powder generally obtained between compact and loose grape regions.

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Now let us see powder mixing. This is very important phenomena. It is one of the most common operation in the industries which work on the powders. Mixing ensures production of dye powders with constant quality as well as for the technological performance of a wide range of powders like soup powders, spice mixes, juice powders etc. These powders are commonly mixed individually or in combination in powder mixers. The main objective of mixing is, in the food industry, to generate product homogeneity.



The mixing quality is mainly affected by mixer type, design of the mixer, that is, size, shape, paddle geometry rotational speed, mixing time, and finally, the type of powders which are being mixed, their particle size, their other characteristic, their properties, which we will discuss a little later. So, mixing requires the relative motion of the different particles making up the powder in order to reach the desired uniform and non-homogeneous distribution.



The mixing mechanism may be, that is, mixing may be either by diffusion or by the convection. Mixing by convection involves clumps of particles moving across the mixer. They are driven by a blade or by centrifugal forces in a drum. Convection can quickly decrease the scale and the intensity of segregation in the mixer.

Mixing by diffusion occurs when a blade moves a clump of particles if those situated at the frontier of the clumps are rearranged under the effect of the gravity. Percolation of some fine particles may, between larger ones may also be occurred there, which allows some particles to leave their native clump to join another. And they are, in the process, mixed together. Shear mixing is induced by the momentum exchange of powders particles having different velocities. And it is developed by the formation of slipping planes in the bulk material.

Some mixing	g indices re	ported in th	e literature			
Author(s)	Lacey	Kramer	Ashton	Poole	Rose	
Reference	Lacey (1954)	Lacey (1954)	Ashton and Valentin (1966)	Poole <i>et al.</i> (1964)	Rose (1959)	
Mixing index	$\frac{\sigma_0^2-\sigma^2}{\sigma_0^2-\sigma_r^2}$	$\left(\frac{\sigma_0-\sigma}{\sigma_0-\sigma_r}\right)$	$\frac{\log[\sigma_0^2 / \sigma^2]}{\log[\sigma_0^2 / \sigma_r^2]}$	$\frac{\sigma}{\sigma_r}$	$1-\frac{\sigma}{\sigma_0}$	
σ²: Varianc σ _r ²: Varianc	e of sample, o ce of random	50 ² : Variance of mixtures	a completely seg	regated mixtur	e;	S S S S S S S S S S S S S S S S S S S

In the literature, various mixing indices are reported. Some of them, I have given here. Like, one can conclude by these models the mixing index, and then finally decide the level of mixing of different powders. These are the reference, the model and the researcher who developed these models. So, there are similar, many such models available in the literature, and you can use to find out the mixing indices.



Now let us discuss the powder properties. The properties of the powder may be classified broadly into three groups, that is, fundamental properties, functional properties and there are some characteristics which are considered as a defective property of the powder. So, the fundamental properties include moisture, shape, surface composition, surface morphology, size and distribution. Even internal molecular and macro structure, bulk density, angle of repose and glass transition and melting. The functional properties include flowability, compressibility, reconstitution properties and so on. Whereas dustiness, stickiness, caking, specks, etc. come under the defective properties category.

Properties of powders (contd...) Moisture Plays a vital role in all aspects of general handling of any type of powder. Associated with increased cohesiveness, mainly due to inter-particle fiquid bridges. Inter-particle films or bridges are responsible for spontaneous agglomeration of particles. The formation of liquid films or bridges at the surface of food powders may cause flow difficulties and severe caking problems.

So, the moisture, obviously it plays a very vital role in all aspects of the general handling of any type of powder. It is associated with increased cohesiveness, mainly due to the inter-particle liquid bridges. Inter-particle films or bridges are responsible for spontaneous agglomeration of particle. The formation of liquid films or bridges at the surface of food powders may cause flow difficulties and severe caking problems.

Shape name	Shape description	
Acicular	Needle shape	
Angular	Roughly polyhedral shape	
Crystalline	Freely developed geometric shape in a fluid medium	
Dentritic	Branched crystalline shape	
Fibrous	Regularly or irregular thread-like	
Flaky	Plate-like	
Granular	Approximately equidimensional irregular shape	
Irregular	Lacking any symmetry	
Modular	Rounded irregular shape	NE.
Spherical	Global shape	

In this table, I have given some common particle shapes, the name of the shape as well as description. Like acicular, there may be needle shape, or flaky, plate-like. Irregular shaped particles lack any symmetry. Modular may be rounded, irregular shaped or spherical, which is generally global shape. So, there are various names of the shape given, and their description, it is provided and taken from the literature.



Then another important property of powder is the bulk density. Powders have lose bulk density, that is, a measured density after a powder is freely poured into a container. And compact density is known as the density after it is allowed to compress by mechanical pressure, vibration and impacts.

So, when a, when powders are poured into a container, the particles of a non-cohesive powder tend to occupy most of the available volume, and thus they have high density. Whereas, a cohesive powder tend to produce an open structure supported by the inter particle forces. And thus, they have generally low density. And you can see here, in the non-cohesive powder, there is a random void but structural void in the cohesive powders.

True density can be measured by fluid displacement method like pycnometry, which are in common use in the industry. And in this, the displacement can be carried out using either a liquid

or a gas, and with the gas implied normally being air. The two common techniques to determine true or apparent density when applicable are liquid pycnometry and air pycnometry. The bulk density, compressibility and flowability of a food powder are highly dependent on particle size, and its distribution. Segregation will occur in a free-flowing powder mixture because of the differences in the particle size.

Hausner ratio. This is one of the standard method of evaluating the flowability of a particular system, is to calculate the Hausner ratio. And that is done after tapping, so Hausner ratio is defined as the ratio of a powder systems, that is, the initial lose bulk density to its tapped density. Or in other words, you can say ratio of loose volume to tapped volume.

And Hausner ratio HR, if it is in between 1 and 1.1, then it is a indicated that powder is a free flowing powders. If the Hausner ratio is found to be in between 1.1 and 1.25, the powder is a medium flowing powder. 1.25 to 1.4 is for the difficulty flowing powder. And Hausner ratio, it is more than 1.4 means the powder is a very difficulty flowing.

So, the compression tests are useful in characterizing the flowability of powders because the interparticle forces enabling non-flowing open structures strand is still in a powder beds, and they are crushed under relatively low-pressure. Adding a small amount of fine powders such as anti-caking agents improve the flowability of the powdered materials. So, the higher the compressibility, the poorer will be the flowability of the powder.

Also, angle of repose, it is defined as the angle at which a material will rest on a stationary heap. It is the angle theta formed by the heap slope and the horizontal plane when the powder is dropped on a platform. So, according to the literature reports, if angles of repose is up to 35 degree, it indicates a free flowability of the powder.

Angle of repose between 35 and 45, there will be some cohesiveness. Between 45 and 55, the cohesiveness will be there, means there is a loss of free flowability. And if the angle of repose is 55 degree and above, means there is a very high cohesiveness, and thus the powder is almost, it has very limited flowability or non-flowable at all. So, the bulk solids when transported, treated or stocked, can flow like liquids, but they can also form a stable heap or pile due to internal forces.

Then, particle size. The particle size distribution measurement is a common method in any physical, mechanical or chemical process because it is directly related to material behavior and not

the physical properties of the products. Size distribution is also one of the factors affecting the flowability of food powder.

As you can see here in this figure, there are four different particle size distribution for a given particulate materials depending upon the quantity measured. Either it is measured by number or measured by length or by surface or measured by mass or volume. And the relationship between the k and fL(x)

 $fL(x) = k_1 \cdot x \cdot fN(x)$

 $fS(x) = k_2.x^2.fN(x)$

 $fM(x) = k_3. x^3. fN(x)$

where the constant k_1 , k_2 , k_3 contain a shape factor which may often be particle size dependent, and x is the particle size.

Technique	Approximate size range (µm)	Type of size distribution	
Sieving			
Woven wire	37-4,000	By mass	
Electro formed	5-120	By mass	
Microscopy			
Optical microscopy	3-1,000	By number	
Transmission electron microscopy	0.002-1		
Scanning electron microscopy	0.02-1.000		
Gravity sedimentation			
Incremental	2-100	By mass	-
Cumulative	2-100	By mass	
Centrifugal sedimentation			
Two layer-incremental	0.01-10	By mass	
Cumulative			
Homogeneous-incremental			
Flow classification			
Gravity elutriation (dry)	5-100	By mass	
Centrifugal elutriation (dry)	2-50	By mass	A CONCEPT
Impact separation (dry)	0.3-50	By mass or number	12-1
Cyclonic separation (wet or dry)	5-50	By mass	a ter
Particle counters			
Coulter principle (wet)	0.8-200	By number	and here
Laser refraction			State 1
Low angle laser light scattering	0.1-3,000	By number	and the second s

There are different analytical techniques for measuring the particle size. Like, it may be sieving or microscopy, like optical microscopy, transmission electron microscopy, scanning electron microscopy, or even flow classification, gravity methods or centrifugal methods or even practical counters or linear refraction methods.

So, there various methods which are used for measuring the particle size. The approximate size ranges which are measured by these methods, and whether they are measured by mass, measured by number, all these details are given here in the table. One can refer, and they indicate the particle size of the material.

Then the morphology. It is, again, when you dry this powder, when we prepare this powder, depending upon the method of the preparation, the morphology of the material may get changed. And you can see here, there are four pictures. Number 1a is the microstructure of a raw fruit, raw strawberry food. And then second one, b, is the microstructure as found from the scanning electron microscope, is of freeze-dried strawberry powder. c is the conventional dried and finally, the last one, d, is the spray dried strawberry powder. And you can see the difference in the structure.

The powders obtained from freeze dried and spray dried, conventionally dried, their original porous structure of the fruit was largely destroyed. And this is mainly as a result of grinding process because after the freeze drying it is found in the form of flake, or conventionally dried, it is in the form of flake or seed. Then it is, during grinding, the original structure is destroyed.

Freeze-dried powders, they are, along with the destruction of the originally spongy structure, the valuable properties of water absorption also, is affected. Spray-dried powder, here, the water was quickly, in the preparation of the powder during spray drying, water was quickly evaporated at a high temperature with a high rotary atomizer speed, which resulted in the formation of round and oval shapes with some concavity on the surface and multiple creases and other deformation.

Now, let us see the rehydration or reconstitution property. This is another very important property for the food powder because all the powders or rehydrated fruits and vegetables, before consumption they need to be rehydrated. And the rehydration characteristics, this varies according to the method used for the preparation and drying methods.

So, the reconstitutability is the rate at which dried food pick up the moisture or absorbs water. It reverts to a condition which resembles the undried material when put in contact with an excessive amount of this liquid. So, when water is added, then by the capillary forces, this water penetrates into the narrow spaces between the fine particles, and the particle gets wet.

And further increase in the water causes the diffusion of the particles, that is, particles swells. It will, and finally, it will start dissolving and it will form a thick gel like mass that resist further water penetration. And finally, a dispersion is formed. The dissolution rate depends upon the surface area of the particle.

Lumps containing dry particles in the middle will be formed requiring strong mechanical stirring to be homogeneously dispersed or dissolve in the liquid. So, the particle will be glassy state or rubbery state. In the glassy state, surface porosity is responsible for the powder hygroscopicity.

So, high shearing, during reconstitution, improves water entrance through particle surface porosity. Whereas in the case of rubbery state, various layers are resulting from the water uptake during storage. In this case, in the rubbery state, it does not improve the water entrance at diffusion because the diffusion is the limiting step. So, in this case, that is, the limiting step is accordingly improved, the diffusion, by properly selecting, either the drying process parameter or after dehydration, there are various treatment which can be given like some time ionizing radiations et cetera, which break the larger polymers, larger polysaccharide into a smaller one.

And it improves the reconstitution behavior. So, there are certain methods. So, this should be followed to improve. But the important thing is that all these dehydrated materials or these powders, they should have a good reconstitution property and that should be ensured by having proper method.

Then comes, finally, handling of food powders, and many food powders contain amorphous glassy component such as amorphous sugars. Particularly, this is in the case of fruits and vegetable powders. So, they contain monosaccharides powders and they have low melting properties, low glass transition temperature, and they are very hygroscopic.

So, above the glass transition temperature for a given water content, as you can see here in this figure, this is a glass transition line and above that, is the sticky zone. And then finally, comes the non-sticky or lower viscosity zone. So, above the glass transition temperature, the powder particles become sticky, and that is called the sticky point temperature.

So, as I discussed in the earlier when we were discussing the spray drying or even drum drying for preparation of fruit juice powder, care has to be taken so that this material, particles temperature does not go above its sticky point temperature or above its glass transition temperature.

So, sticky point is typically about 10 to 20 degree Celsius above the glass transition onset temperature for low molecular weight carbohydrates. Although, it depends upon the composition of the material. So, on moving through the sticky zone, with increasing water content or temperature, the stickiness will first increase to a maximum, after which it will then decrease due to the continuous reduction in the viscosity.

Then packaging and storage of food powders, very important aspect. The packaging has to be done and it is normally done for the protection from moisture, light, oxygen and temperatures. And absorption or desoprtion of moisture can significantly affect the shelf life of dry powder, and also its usability.

So, when they gain moisture, the powder become lumpy or converted into the form of cake which may lead to the deleterious changes such as structural transformation, enzymatic reactions, browning reactions, oxidation depending upon the temperature as well as availability of the oxygen.

So, the effectiveness of a package can be determined during shelf life testing or by combining information from break point testing, and knowledge about the characteristics of the moisture permeability of the packaging material. So, generally, water activity less than 0.6 is considered sufficient to prevent the growth of microorganism, chemical reactions and enzymatic changes.

Packaging of food powders in different materials Metal cans Metal cans are used for their excellent physical strength, durability, absolute barrier properties to moisture, O₂, and light, absence of flavor or odor, and rigidity. Because bare steel is susceptible to corrosion, it is commonly electrolytically coated with a very thin layer of tin; in addition, an organic lacquer is applied to further protect the metal from corrosion and avoid metal-food contact. Powders with a higher fat content are more susceptible to oxidation, and most powders are susceptible to deteriorative effects such as lumping and caking from moisture ingress. With adequately constructed cans, a shelf life in excess of 5 years is realistic, when powdered products have been gas flushed with N₂ to minimize the amount of available O₂.

So, the packaging material which are normally used for powder packing are metal cans. They have the excellent physical strength, durability, absolute barrier properties to moisture, oxygen, light. They are inert, they do not have any special flavor, odor, and also, they are rigid. So, because of these properties, the bare steel is not used because bare steel is susceptible to corrosion.

So, it is commonly electrolytically coated with a very thin layer of tin. So, tin coated metal cans are used. And in addition to it, an organic lacquer is applied to further protect the metal from the corrosion and avoid metal food contact. Powders with a high fat content are more susceptible to oxidation, and most powders are susceptible to deteriorative effects such as lumping and caking from the moisture ingress.

So, it is very important that these materials, when they are packed, they should be impermeable, fully impermeable to oxygen, they should be fully impermeable to moisture content. So, with adequately constructed cans, in the metal cans, if it is properly made and if it is properly packaged, there is inside no oxygen, it is flushed with the liquid nitrogen or such other inert gas, then one can expect the shelf life of food powders as high as 5 years.

Multilayer pouches

- Aluminum foil/plastic film laminates have been introduced as a replacement for the tin can.
- The laminates can be formed, filled, gas-flushed, and sealed on a single machine from reel stock.
- Flexible packages reduce the volume of traditional packaging such as metal cans, reduce transport costs, reduce the cost of the packaging, and require less material, thus minimizing post-consumer waste.
- Aluminum foil is capable of providing such a close-to-absolute barrier when built into a flexible material.
- A sandwich construction with two plastic layers i.e. one on the inside (LDPE) so that the pouch can be sealed; and the other on the outside (BOPP or PET) to provide mechanical protection and also carry information.

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Then multilayer pouches where aluminum foil, plastic film laminates has been introduced as a replacement of the tin can. In the earlier lectures when we were discussing about packaging of fresh produce or cut fruits and vegetables, there we discussed these things, how these laminates are made.

So, these flexible packages reduce the volume of traditional packaging. Aluminum foil is capable of providing a, such a close to absolute barrier with built into a flexible material. And in this laminates, multilayer laminates, a sandwich construction with two plastic layers, one on the inside which may be LDPE, so that the pouch can be sealed, and the other on the outside may be BOPP or PET to provide mechanical protection and also to carry the information and for labeling.

For pouches of shorter shelf life, the aluminum foil layer may be replaced with a high barrier plastic film such as copolymer of ethyl vinyl alcohol commonly known as EVOH or

polyvinylidene chloride, with the addition of a thin layer of metal or silica oxide deposition to enhance its oxygen barrier characteristics. So, sachets with larger capacity, that is, in excess of 250-gram, comprising a high barrier plastic layer sandwiched between LDPE and BOPP or PET would be able to achieve a shelf life to a similar shelf life to an aluminum foil or sandwich portion pack pouch.

So, finally food powders are very important fruits and vegetable powders. Many times, they are important commodities, they are used as ingredients or they are used in the preparation of various mixes, the flours or spices, so properly because this particle size and other properties are very important.

Almost all the properties, free flowability and other things, particle size and its moisture absorption, hygroscopicity so all this should be properly taken care of, and the powders or particles, when they are made, there is many time their agglomeration is done like in the case of coffee powder. That is to improve its reconstitution behavior or to improve its stability. Agglomeration technology is used for such other things.

But important thing is that this powder should have a good free flow nature, they should have a good reconstitution properties, they should be properly able to be mixed into one another and homogeneously or uniformly. So, accordingly the proper conditions of packaging, manufacturing technology processes should be used and then its packaging also, is an important step.

So, proper care should be, because most of these powders, they have that high hygroscopic nature. So, if they absorb moisture, they will lose their free flow nature and their other characteristics will change. Their mixing and other properties will be adversely affected. So, it is important that they are properly packaged in a moisture proof material. Water vapor, air, oxygen and other gases should completely impermeable to this.

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So these are the references which are used in this lecture. Thank you very much for your patient hearing. Thank you.