COMPACTION AND COMPRESSION

Definitions

Compression

Compression means a reduction in the bulk volume of a material as a result of the removal of the gaseous phase (air) by applied pressure.

Consolidation

Consolidation is an increase in the mechanical strength of a material resulting from particle-particle interactions.

Compaction

Compaction of powders is the general term used to describe the situation in which these materials are subjected to some level of mechanical force.

The physics of compaction may be simply stated as "the compression and consolidation of a two-phase (particulate solid-gas) system due to the applied force."

Derived Properties of Powders or Granules

Some derived properties which help in quantification of important variables are

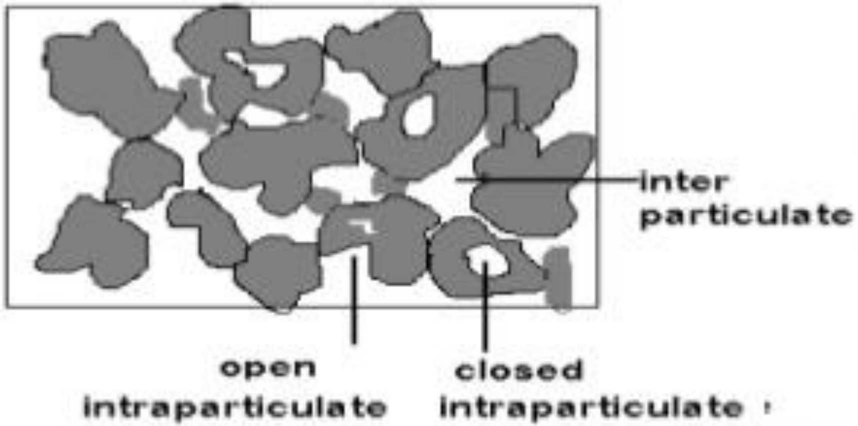
1. Volume
2. Density
3. Porosity
4. Flow properties.

Volume: Measurement of volume of powder is not easy as the measurement of mass of powders, because in powders there will be inter and intra- particular voids. Hence three types of volume can be considered for a powdered mass, they are,

1. True volume
2. Granular volume
3. Bulk volume

Mass-Volume relationships

1. **Open intraparticulate voids-those** within a single particle but open to the external environment.
2. **Closed interparticulate voids-those** within a single particle but closed to the external environment.
3. **Interparticulate voids-the** air spaces between the individual particles.



Therefore, at least three interpretations of "powder volume" may be proposed

True Volume of the powder (Vt): True volume is the total volume of the solid particles. It is a volume of the particles excluding the inter and intra particulate spaces in a powder. Or it is volume of powder itself .

Granular Volume of the powder (Vg): It is the cumulative volume occupied by the particles, including all intraparticulate (but not interparticulate) voids. Or it is the volume of powder itself and volume of intraparticulate spaces.

Bulk Volume of the powder (Vb): It is the total volume occupied by the entire powder mass under the particular packing achieved during the measurement.

It comprises the true volume and inter and intra particulate voids.

Relative volume (VR): It is the ratio of the the volume, V of the sample under specific experimental conditions, to the true volume Vt.

$VR = V / Vt$

VR tends to become unity as all air is eliminated from the mass during the compression process.

Density

The ratio of mass to volume is known as the density (ρ) of the material

By considering the three types of volume of powders, we can define the respective densities as,

- **True density (ρt):** Mass of the powder/ True volume of the powder .

True density, $\rho t = M / Vt$

- **Granular density (ρg):** Mass of the powder/ granule volume of the powder.

Granular density, $\rho g = M / Vg$

- **Bulk density (ρb):** It is the ratio of total mass of the powder to the bulk Volume of the powder. It is measured by pouring the weighed powder into a measuring cylinder and the volume is noted.

It is expressed in gm/ml and is given by: $\rho b = M / Vb$

Where , M is the total mass of the powder

Vb is the Bulk Volume of the powder

Porosity

The spaces between the particles in a powder are known to be voids. The volume occupied by such voids is known to be void volume.

Void volume (Vv) = Bulk volume –True volume

The porosity of the powders is defined as ratio of the void volume to the bulk volume of the packing. Or, the ratios of the total volume of void spaces (Vv) to the bulk volume of the material.

Porosity (E) = void volume /bulk volume.

$$= Vv/Vb$$

$$= [Vb - Vt / Vb] \text{ (As, } Vv = Vb - Vt)$$

$$= 1-(Vt/bt)$$

Porosity is frequently expressed in percent

$$E = [1- Vt / Vb] \times 100$$

The relation between porosity and compression is important because porosity determines the rate of disintegration, dissolution and drug absorption.

Flow Properties

To get uniformity of the weight of the tablet, the powder should possess a good flow property. Flow properties of the powders depend on the-

1. Particle size,
2. Shape,
3. Porosity and density,
4. Moisture of the powder.

Particle size

The rate of flow of powder is directly proportional to the diameter to the particles.

Beyond particular point, flow properties decreases as the particle size in increases. Because in small particle (10µ) the vanderwaal's, electrostatic and surface tension forces causes cohesion of the particles resulting poor flow.

As the particle size increases, influence of gravitational force on the diameter increases the flow property. But appropriate blends of fines & coarse improves flow characteristic, as the fines get absorbed and coarse particle reduce friction.

Particle shape

Spherical, smooth particles improves flow properties, surface roughness leads to poor flow due to friction and cohesiveness , flat and elongated particles tend to pack loosely, obstructing the flow

Density & porosity

Particles having high density and low internal porosity tend to posses good flow properties.

Moisture

The higher moisture content, the flow property will be poor owing to cohesion and adhesion.

Angle of repose

The flow characteristic are measured by angle of repose . Angle of repose is defined as the maximum angle possible between the surface of a pile of powder and the horizontal plane.

tanØ= h/r

Ø = tan-1(h/r)

Where, h = height of pile

r =Radius of the base of pile.

Ø = Angle of repose .

The angle of repose is calculated by measuring the height and radius of heap of powder formed.

The frictional forces in a loose powder can be measured by Angle of repose.

The lower the angle of repose, better will be flow property.

The values of angle of repose are given below

Angle of repose (in degrees)	Type of Flow
<25	Excellent
25-30	Good
30-40	Passable
>40	Very poor

Carr's Consolidation (Compressibility) Index (CI)

It indicates powder flow properties. It is expressed in percentage.

It is defined as:

Consolidation Index = I = Tapped density-Poured density /Tapped density

Therefore = (Dt- Db/Dt) × 100

Where, Dt is the tapped density of the powder

Db is the Poured density of the powder

Determination of Tapped density & Poured density.

It is determined by passing a fixed quantity of powder into a measuring cylinder and the volume is noted .CI can be calculated by founding out by tapped density and Poured density of powder.

Grading of the powder for their flow properties according to Carr's index:

Carr's index(%)	Type of flow
5-15	Excellent
12-18	Good
18-21	Fair to passable
23-35	Poor
33-38	Very poor
>40	Very very poor

Consolidation

An increase in the mechanical strength of the material resulting from particle or particle interaction. (Increasing in mechanical strength of the mass)

Consolidation Process

Cold welding: When the surface of two particles approach each other closely enough, (e.g. at separation of less than 50nm) their free surface energies result in strong attractive force, this process known as cold welding.

Fusion bonding: Contacts of particles at multiple points upon application of load, produces heat which causes fusion or melting. If this heat is not dissipated, the local rise in temperature could be sufficient to cause melting of the contact area of the particles.

Upon removal of load it gets solidified giving rise to *fusion bonding* & increase the mechanical strength of massIn.

Factors affecting consolidation

Both “cold" and "fusion" welding, the process is influenced by several factors, including:

1. The chemical nature of the materials
2. The extent of the available surface
3. The presence of *surface contaminants*
4. The inter-surface distances

Strength of the Tablets

The tablet should be sufficiently strong to withstand the mechanical shocks during the subsequent handling and transport. The mechanical strength of tablet is described by the following parameters.

- Crushing Strength
- Friability
- Hardness
- Bonding Strength
- Fracture resistance.

Crushing Strength

The most popular estimate of tablet strength has been crushing strength, Sc, which may be defined as "that compressional force (Fc) which, when applied diametrically to a tablet, just fractures it."

It may then be described by the equation= $S_T = \frac{2F_c}{D \cdot H}$

Where S_T is the tensile strength, Fc is the compressional force and D & H are the diameter and thickness of the tablet, respectively.

Friability

The crushing strength test may not be the best measure of potential tablet behaviour during handling and packaging. The resistance to surface abrasion may be a more relevant parameter. For example by the friability test.

These test measure the weight loss on subjecting the tablets to a standardized agitation procedure. The most popular (commercially available) version is the Roche Friabilator, in which approximately 6 g (W_o) of de-dusted tablets are subjected to 100 free falls of 6 inches in a rotating drum and are then reweighed (w).

The friability, f, is given by: $f = 100 \cdot \left(1 - \frac{w_o}{w}\right)$

Factors Affecting Strength of the Tablets

The following factors affect the strength of tablet

1. Particle size
2. Particle shape & surface roughness
3. Compaction pressure
4. Binders
5. Lubricants
6. Entrapped air
7. Moisture content
8. Porosity

Particle size

Smaller particles have larger surface area & when these are exposed to atmosphere may be prone to oxidation and moisture absorption takes place which affects the strength of tablet.

Extensive fragmentation during compaction of a brittle material may result in a large number of interparticulate contact points, which in turn provide a large number of possible bonding zones. The tablets made of these materials can have a high mechanical strength.

The increase in mechanical strength is attributed to an increase in the surface area available for interparticulate attractions, as the particles become smaller.

Particle shape & surface roughness

The mechanical strength of tablets of materials with a high fragmentation tendency are less affected by particle shape and surface texture. Particle shape affects the inter particulate friction & flow properties of the powder. Spherical particles are considered to be ideal.

General particle shapes and their effect on powder flow are as follows:

Spherical particles - Good
Oblong shaped particles - Poor
Cubical shaped particles - Poor
Irregular shaped particles - Medium

Compaction pressure

The compaction pressure and speed affects the strength of the resulting tablet. A fragmenting material has been shown to be less affected by variations in compression speed. The behaviour of granules during compaction, the extent to which they bond together & the strength of the inter granule bonds relative to the strength of the granules determine tablet hardness.

Binders

A binder is a material that is added to a formulation in order to improve the mechanical strength of a tablet. In direct compression, it is generally considered that a binder should have a high compactibility to ensure the mechanical strength of the tablet mixture. Addition of a binder which increases elasticity can decrease tablet strength because of the breakage of bonds as the compaction pressure is released.

Lubricants

Lubricants are used to improve granule flow, minimize die wall friction & prevent adhesion of the granules to the punch faces. Lubricant decreases the strength of the tablets. When lubricants are added as dry powder to granules, they adhere & form a coat or a film around the host particles during the mixing process. The Lubricant film interferes with the bonding properties of host particle by acting as a physical barrier. When the tablet is blended lightly, the lubricant is present as a free fraction. Prolonged mixing time will produce a surface film of lubricants over the drug particles due to which inter particulate bonding is reduced.

Entrapped air

When the air does not freely escape from the granules in the die cavity, the force created by the expansion of the entrapped air may be sufficient to disrupt the bonds.

The presence of entrapped air will produce a tablet which can be broken easily & it lowers the tablet strength.

Moisture content

A small proportion of moisture content is desirable for the formation of a coherent tablet. At low moisture content there will be increase in die wall friction due to increased stress, hence the tablet hardness will be poor. At high moisture level the die wall friction is reduced owing to lubricating effect of moisture. At further increase in moisture content there will be decrease in compact strength due to reduction in inter particulate bond.

Optimum moisture content is in the range of 0.5 – 4%.

Porosity

When particles of large size are subjected to light compression the tablet will be highly porous–low tablet strength. Reduction in porosity is due to granule fragmentation giving smaller particles which may be more closely packed & plastic deformation which allows the granules to flow into the void spaces.

Parts of a Tablet Press

Tablet presses are designed with following basic components:

1. ***Hopper*** for holding and feeding granulation
2. ***Dies*** that define the size and shape of the tablet.
3. ***Punches*** for compressing the granulation within the dies.
4. ***Cam tracks*** for guiding the movement of the punches.
5. A ***feeding mechanism*** for moving granulation from hopper into the dies.

Auxiliary Equipment's

1. Granulation Feeding Device: In many cases, speed of die table is such that the time of die under feed frame is too short to allow adequate or consistent gravity filling of die with granules, resulting in weight variation and content uniformity. These also seen with poorly flowing granules. To avoid these problems, mechanized feeder can employ to force granules into die cavity.

2. Tablet weight monitoring devices:- High rate of tablet output with modern press requires continuous tablet weight monitoring with electronic monitoring devices.

3. Tablet Deduster: In almost all cases, tablets coming out of a tablet machine bear excess powder on its surface and are run through the tablet deduster to remove that excess powder.

4. Fette machine: Fette machine is device that chills the compression components to allow the compression of low melting point substance such as waxes and thereby making it possible to compress product with low meting points.

Instrumented tablet machines and tooling

The tablet press is a high-speed mechanical device. It compresses the ingredients into the required tablet shape with extreme precision. It can make the tablet in many shapes, although they are usually round or oval. Also, it can press the name of the manufacturer or the product into the top of the tablet.

Tablet punching machines work on the principle of compression.

A tablet is formed by the combined pressing action of two punches and a die.

Punches & Dies

Tooling Station: - The upper punch, the lower punch and the die which accommodate one station in a tablet press.

Tooling Set: A complete set of punches and dies to accommodate all stations in a tablet press.

Different Shapes of Dies and Punches

- Round shape punch die set
- Oval shape punch die set
- Capsule shape punch die set
- Geometric shape punch die set
- Irregular shape punch die set
- Core rod tooling punch die set

Round shape Punch Die Set

Used by pharmaceutical and veterinary industry.

Can manufacture following type of tablets:

- ✓Shallow Concave Ball Shape
- ✓ Deep Concave Flat Faced
- ✓Concave with Edges Flat with Bevel Edges
- ✓Normal Concave

Oval Shape Punch Die Set

Applicable to pharmaceutical and ayurvedic industries.

Can manufacture following types of tablets:

- ✓Flat Faced Flat with bevel edges
- ✓Concave/Deep/Deep Concave with bevel edges.

Capsule shape punch die set

Applicable to pharmaceutical and ayurvedic industries.

Can manufacture following types of tablets:

- ✓Concave with Edges
- ✓Deep Concave Flat Faced
- ✓Normal concave Flat with Bevel Edges.

Geometric Shape Punch Die Set

Applicable to pharmaceutical, confectionery, chemical, industrial powder metallurgy industries. Can manufacture following types of tablet:

- ✓Triangular
- ✓ Benzene
- ✓ Rhombus
- ✓ Rectangular Square

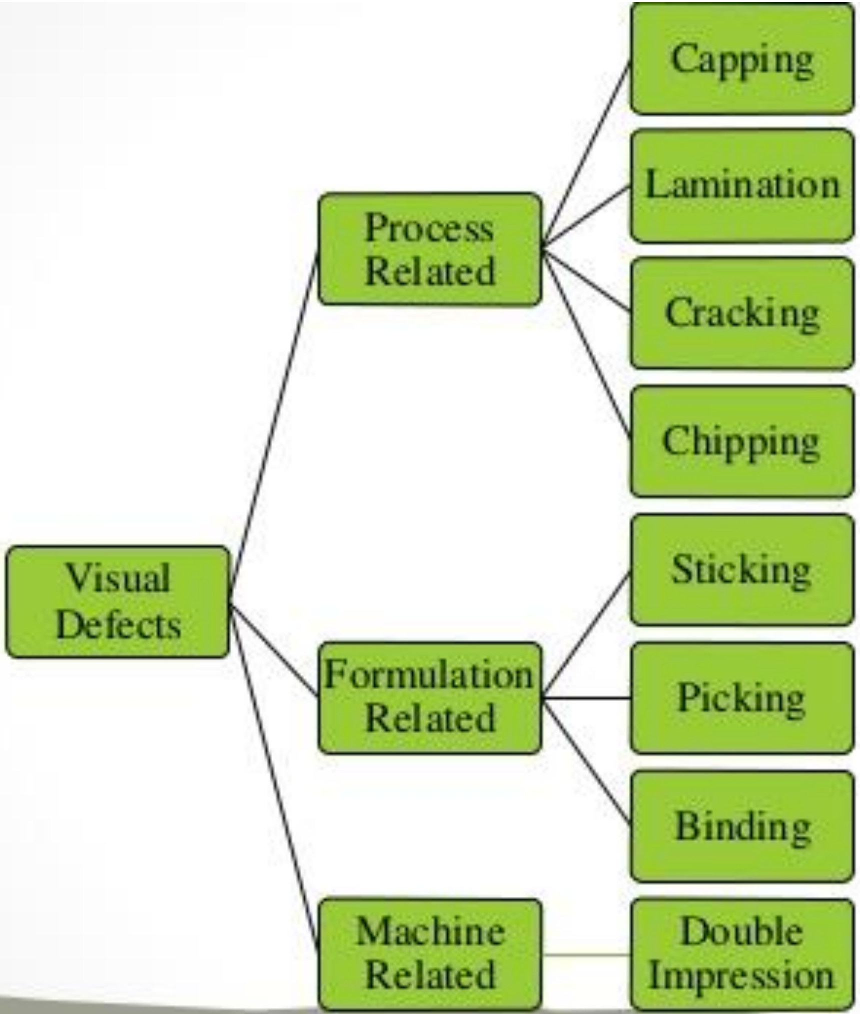
Irregular Shape Punch Die Set

Are applicable to confectionery industries.

Available with different size, concavity, and flat in plain or engraved break line.

Problems associated with large scale manufacturing of tablets.

- In olden days tablets were initially punched on small scale with hand operated machines, which suffered the problem of varied strength and integrity.
- But now the tablet punching machines are all mechanized, the mechanical feeding of feed from the hopper into the die, electronic monitoring of the press, but tablet process problem still persist.
- The Imperfections known as: 'VISUAL DEFECTS' are either related to Imperfections in any one or more of the following factors[1]:
  I. Formulation design
  II. Tableting process
  III. Machine


Visual Defects
Process Related
Formulation Related
Machine Related
Capping
Lamination
Cracking
Chipping
Sticking
Picking
Binding
Double Impression

The defects related to Tableting Process..

CAPPING:

Partial or complete separation of the top or bottom of tablet due to air-entrapment in the granular material.

LAMINATION:

Separation of tablet into two or more layers due to air-entrapment in the granular material.

CRACKING:

Small, fine cracks observed on the upper and lower central surface of tablets, or very rarely on the sidewall.

CHIPPING:

Breaking of tablet edges.

The defects related to Formulation.

STICKING:

The adhesion of granulation material to the die wall·

PICKING:

The removal of material from the surface of tablet and its adherence to the face of punch·

BINDING:

Sticking of the tablet to the die and does not eject properly out of the die·

The defect related to Machine.

DOUBLE IMPRESSION:

Due to free rotation of the punches, having some engraving on their faces·

The defect related to more than one factor.

MOTTLING:

Unequal distribution of color on a tablet with light or dark areas.

THANKS