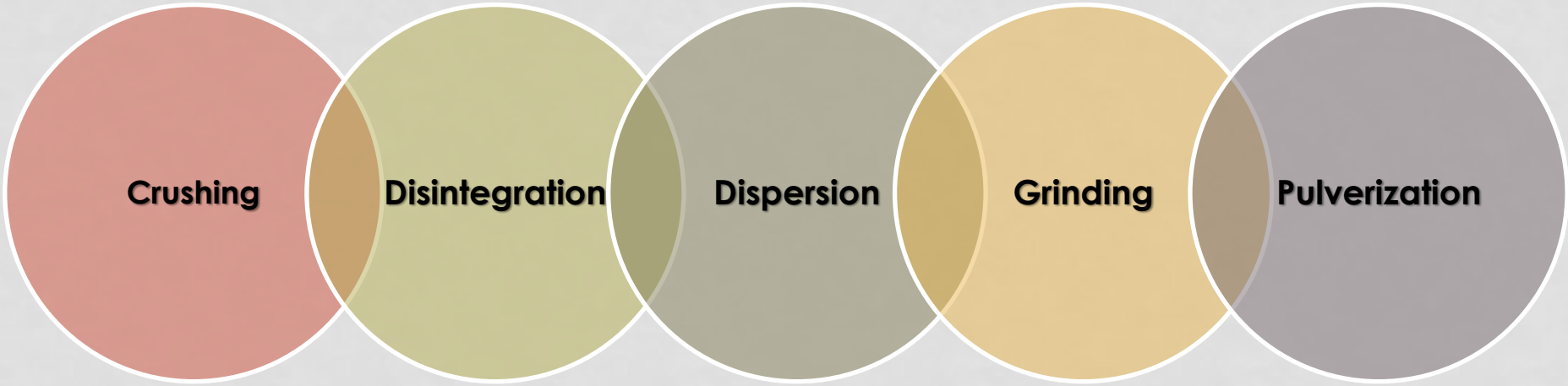
MILLING

LAB-6-

INTRODUCTION

Milling: Is a mechanical process of reducing particle size of solids.
Milling also termed synonymously as **comminution** which represent:



All of these depend on product, equipment and process.

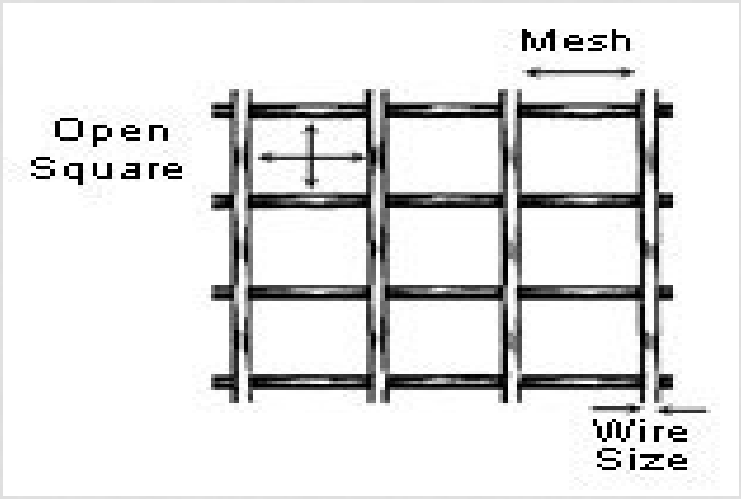
https://www.youtube.com/watch?v=cA7gG8gbj8I

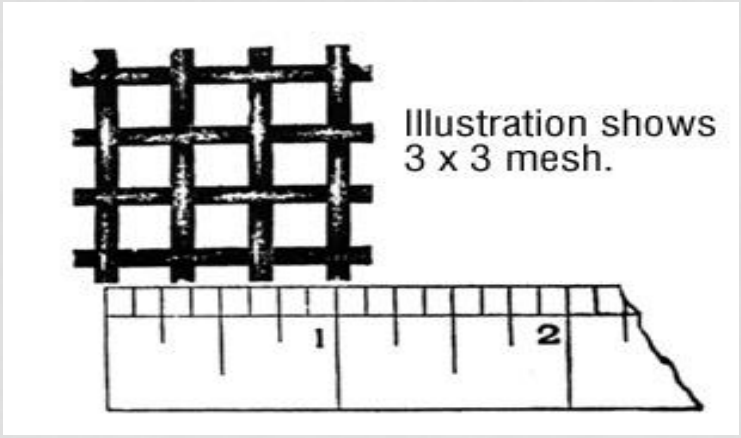
INTRODUCTION- MILLING EQUIPMENT

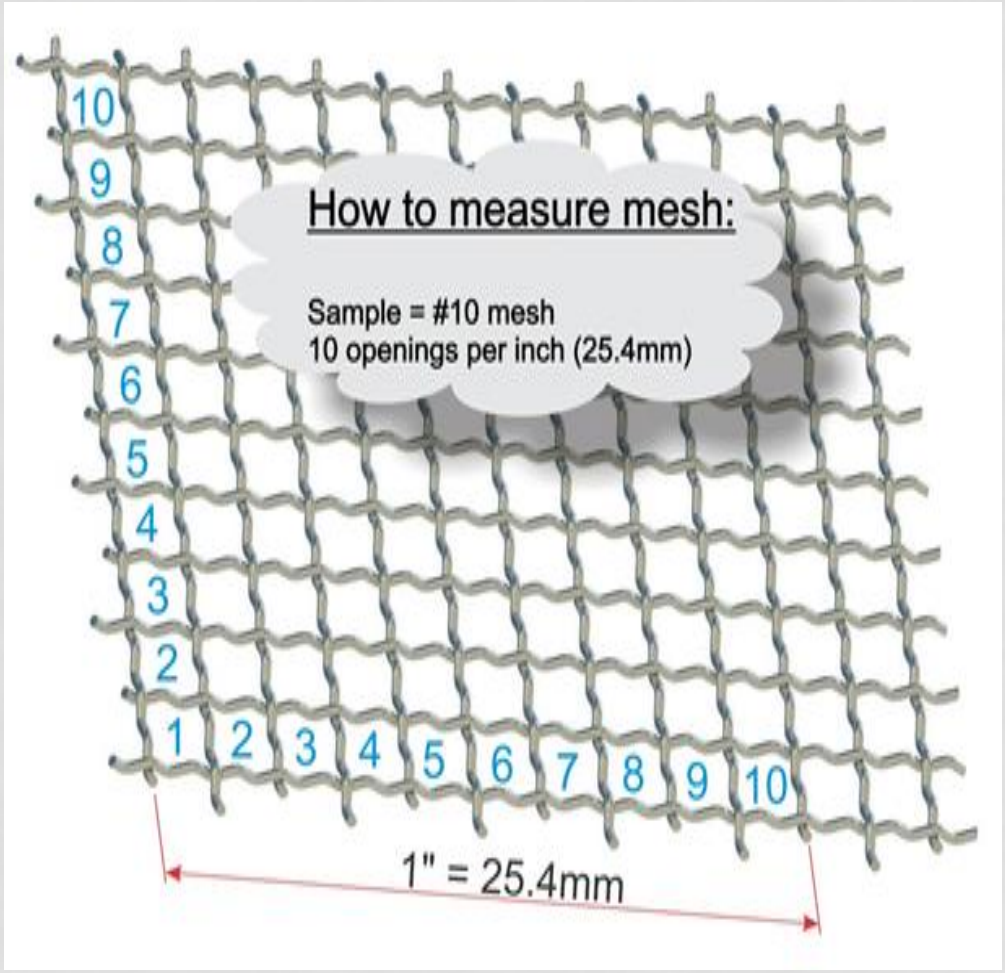
Milling equipment classified according to the size of the milled product into:

- Coarse milling (particles > 20-mesh)
- Intermediate (particles 200-20 mesh [74-840 micron])
- Fine (particles < 200 mesh)

NOTE: SIZE EXPRESSED IN TERM OF MESH (NUMBER OF OPENINGS PER LINEAR INCH OF A SCREEN).







PHARMACEUTICAL APPLICATIONS

1. **Increasing therapeutic efficacy of low solubility drugs** due to increasing specific surface area (S.A per unit wt.) thus increasing area of contact with dissolving fluid, e.g. griseofulvin.

2. **Facilitate drying of wet masses** due to increase surface area and reduce the distance (⇩ thickness) the moisture travel within particle to reach outer surface, e.g. granulation of wet mass in tablet preparation.

3. **Facilitate easier and uniform mixing or blending**

   The ingredients are approximately of same size.

PHARMACEUTICAL APPLICATIONS

4. Solid dosage form that is artificially colored are often milled (to ensure that the mixture is not mottled and is uniform from batch to batch).

5. Lubricants should be milled to fine powder (to ensure their ability to coat surface of powder or granules).

6. Milling in ointments, creams and pastes provide smooth texture, better appearance and improve physical stability.

https://www.youtube.com/watch?v=i35IC8aCjXE

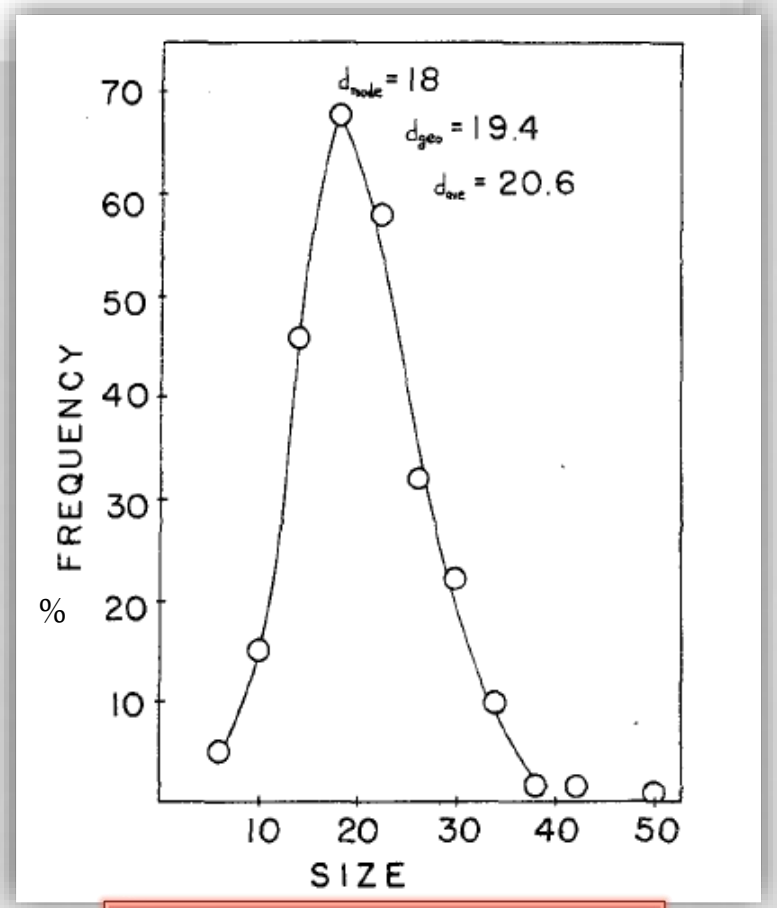
SIZE DISTRIBUTION AND MEASUREMENT

In naturally occurring particulate solids and milled solids.

The shape of particle is irregular, and size varies from largest to smallest size

Size distribution used instead of particle size

Which represents % frequency of each particle size (i.e what size present in what proportion)

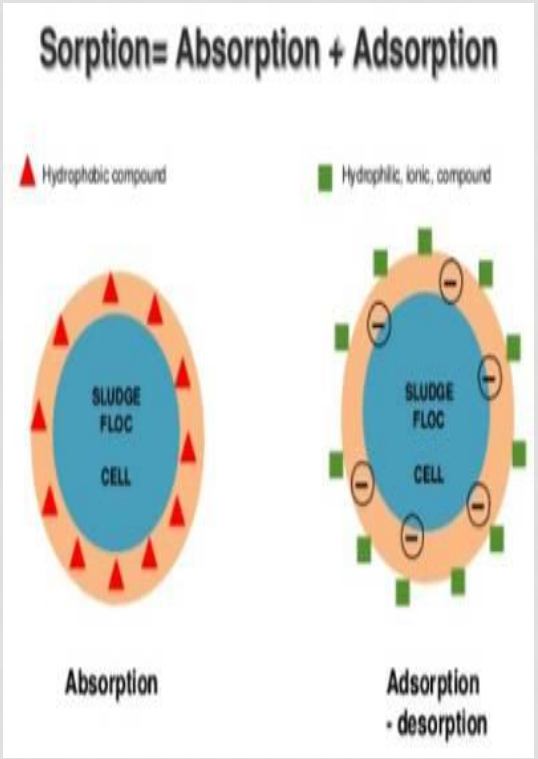


Bell shape structure

Normal or Gaussian distribution if not then (Skewed distribution)

METHODS OF MEASUREMENT OF SIZE DISTRIBUTION

1. Microscopy
2. Sieving
3. Sedimentation
4. Other methods (sorption, electrical conductivity, light and x-ray scattering, permeametry, and particle trajectory).



https://www.youtube.com/watch?v=QQTb5JMQ4-s

https://www.youtube.com/watch?v=Gmhc7hsY7P4

1- MICROSCOPY

- **Direct Method for measuring P.S. distribution.**

Disadvantages: can't resolve particles if it's size is close to the wave length of the light source.

https://www.youtube.com/watch?v=_CkcYrns-6I

https://www.youtube.com/watch?v=JuRyaDxERF4

2- SIEVING

- It is pan with bottom of wire cloth with square openings.
- Most widely method for measuring P.S. distribution.
- **Advantages**: Inexpensive, simple, rapid, limited variation between operators.

https://www.youtube.com/watch?v=3Xqq1cxhD-s
https://www.youtube.com/watch?v=XYC5YM_AxoM

U.S. Standard		*Tyler Standard*	
Micron	*Mesh*	*Micron*	*Mesh*
5660	3½	5613	3½
4760	4	4699	4
4000	5	3965	5
3360	6	3327	6
2830	7	2794	7
2380	8	2362	8
2000	10	1651	10
1680	12	1397	12
1410	14	1168	14
1190	16	991	16
1000	18	883	20
840	20	701	24
710	25	589	28
590	30	495	32
500	35	417	35
420	40	351	42
350	45	295	48
297	50	246	60
250	60	208	65
210	70	175	80
177	80	147	100
149	100	124	115
125	120	104	150
105	140	88	170
88	170	74	200
74	200		
62	230		
53	270		
44	325		
37	400		

2- SIEVING

Measuring diameter of powder bypass series of sieves: 30-mesh and retained on 45-mesh (diameter= 590 + 350)/2 or 470 microns.

Size of distribution effected by

- **1- No. of Sieves**
  (by passing powder through series of smaller sieves and weighing portion retained on each sieve).
- **2- Motion of sieve (vibratory, side-tap, bottom-tap, rotary-tab, rotary).**
- **3- Time of sieving.**
- **4- Load or thickness of powder (proportional to time).**

MILLING OPERATIONS

A- open-circuit milling:

Materials is reduced to the desired size by passing it through the mill.

B- closed-circuit milling:

Materials discharge from mill pass through classifier or size-separation device, and the oversize are returned to the grinding chamber for further reduction in size.

https://www.youtube.com/watch?v=XYC5YM_AxoM

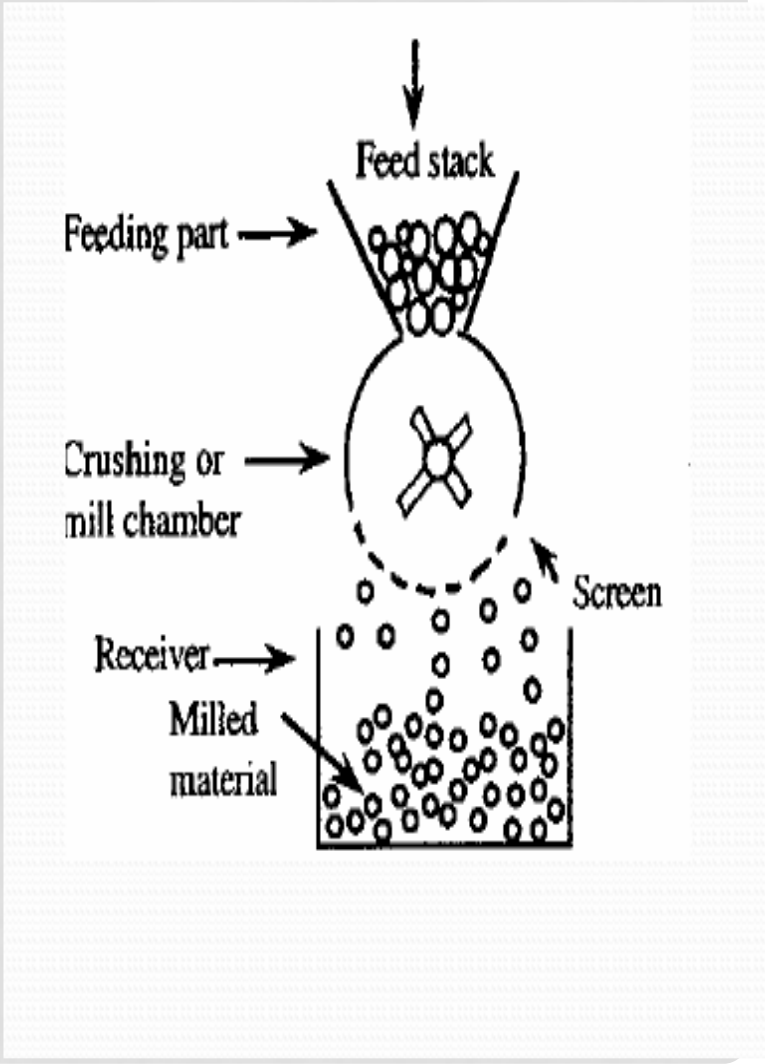
https://www.youtube.com/watch?v=f25qZT7ZZkM

PARTS OF MILLS

1- **Feed part**

2- **Grinding part (milling chamber)**

3- **Discharge part (Receiver)**



IMPORTANT NOTE

The rate of discharge should be equal to the rate of feed.

A- If rate of feed is slow

- the product discharge readily and the amount of undersize or fines is minimized.

B- If rate of feed is fast

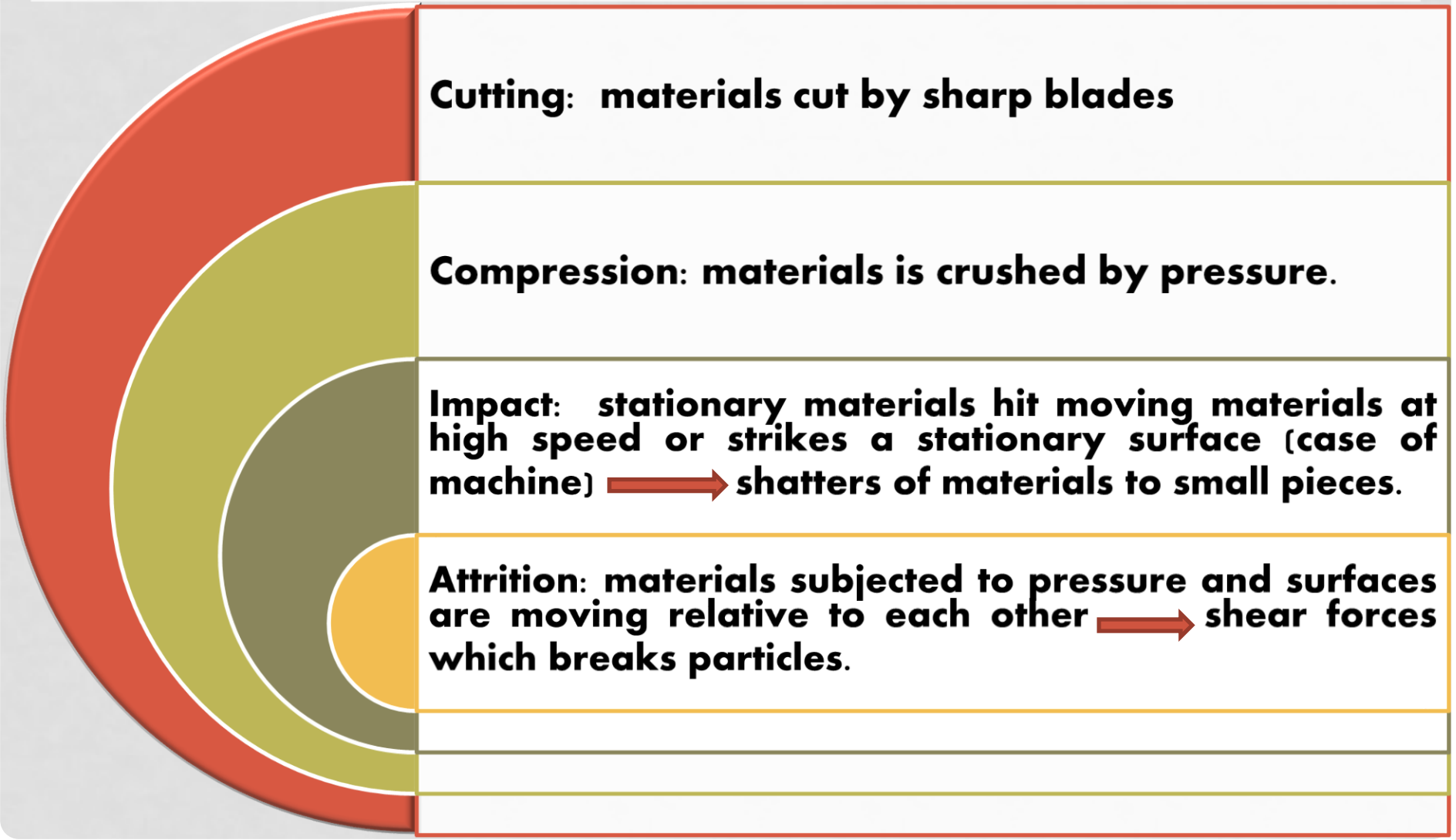
- the material remain in the milling chamber for long time because its discharge is impeded by large amount of material leads to greater size reduction and lower mill capacity.

TYPES OF MILLS

General Characteristics of Various Types of Mills

Type of Mill	*Action*	*Product Size*	*Used For*	*Not Used For*
Cutter	cutting	20- to 80-mesh	fibrous, crude animal and vegetable drugs	friable material
Revolving	attrition and impact	20- to 200-mesh	fine grinding of abrasive material	soft material
Hammer	impact	4- to 325-mesh	almost all drugs	abrasive material
Roller	pressure	20- to 200-mesh	soft material	abrasive material
Attrition	attrition	20- to 200-mesh	soft and fibrous material	abrasive material
Fluid-energy	attrition and impact	1 to 30 μm	moderately hard and friable material	soft and sticky material

MECHANISM OF SIZE REDUCTION



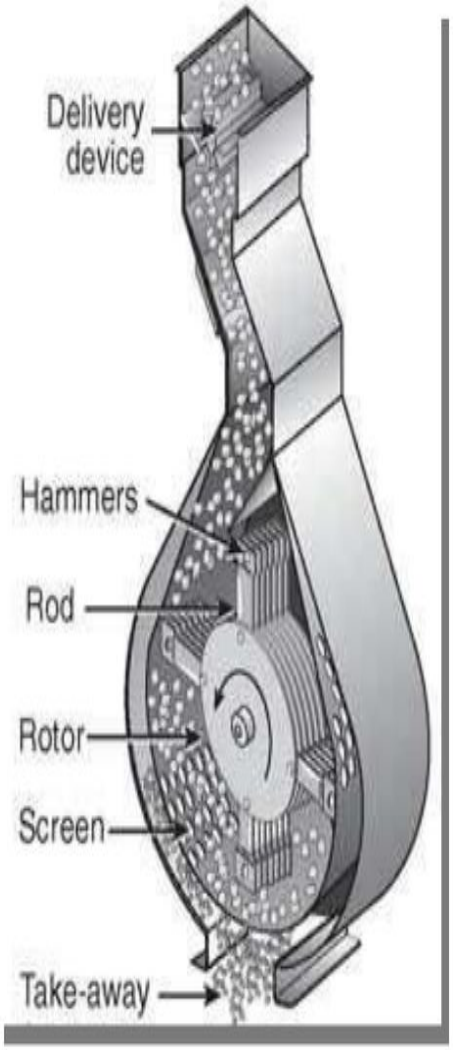
HAMMER MILL

Principle:

Operates as an impact between rapidly moving hammers mounted on the rotor and the powder material.

Used for almost any type of size reduction (dry material, wet filter-press cakes, ointment, slurries).

It is popular in pharmaceutical industry because of versatility



https://www.youtube.com/watch?v=fSTvbE77ucE

https://www.youtube.com/watch?v=odd9Mv18z0

BALL MILL

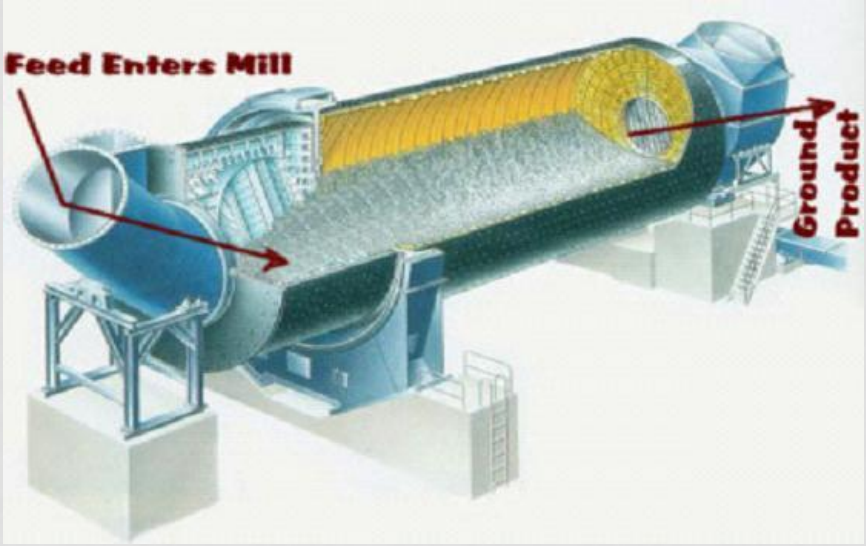
Principle:
Combination of impact and attrition.

A horizontal rotating hollow vessel of cylindrical shape filled with balls of steel or pebbles (grinding medium).

It includes

A- Pebble mill
B- Rods or bars mill

https://www.youtube.com/watch?v=L6sgGXXYdEU



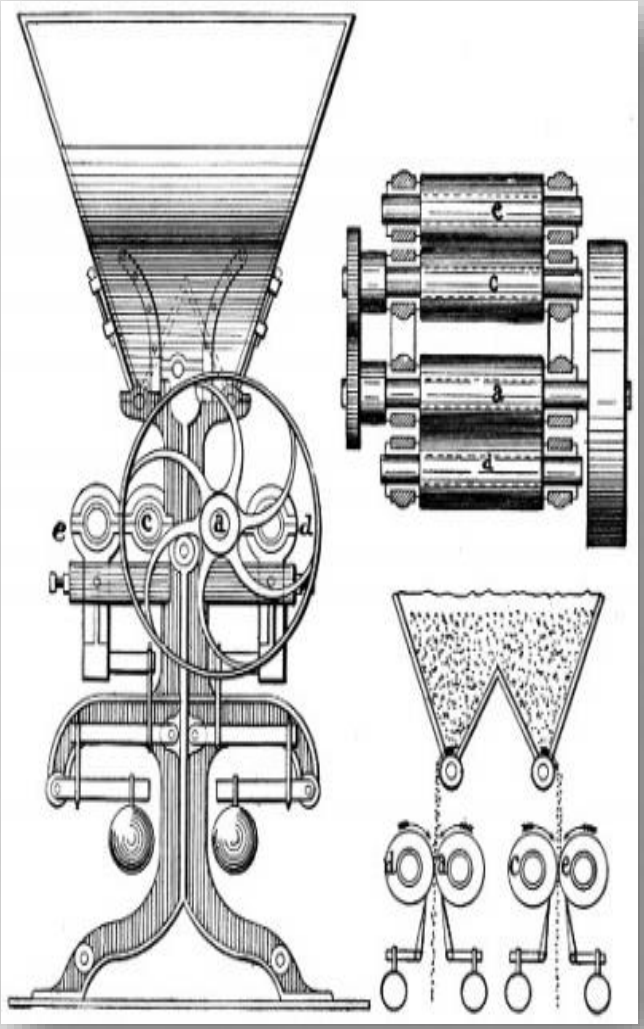
ROLLER MILL

PRINCIPLE:

combination of compression and shearing action.

MECHANISM OF ACTION:

2-5 smooth rollers operating at different speed.



https://www.youtube.com/watch?v=yffPcrHcy3c

CUTTING MILL

Principle: cutting and shearing action

Uses: for fibrous and tough material.

Types: single and double runner disc mills.

https://www.youtube.com/watch?v=1FVHv3SVxUk

PROCEDURE

1. weigh the beaker (200 g).
2. weigh the brown sugar granules (150 g) in a beaker and subtract from the weigh of the beaker.

350 g (weight of beaker + brown sugar)

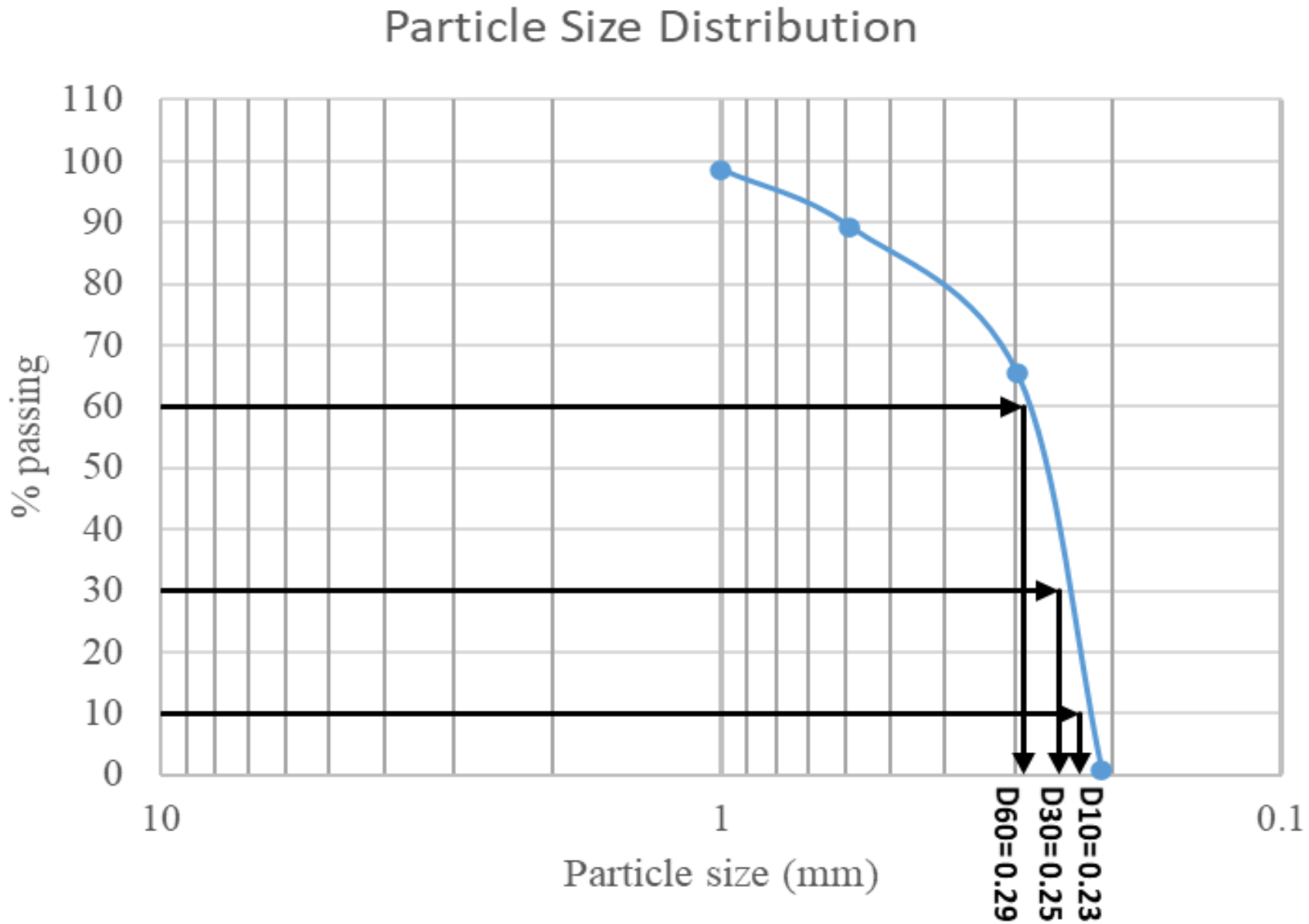
3. Mill the brown sugar in the hammer mill for 1 min to allow the powder to pass through the sieve [18 mesh] then weigh the remaining.
4. Sieve the remaining powder through series of sieves (0.59 mm [30 mesh], 0.297 mm [50 mesh], 0.210 mm [70 mesh] and weigh the powder remaining.

WHOLE WEIGHT OF BROWN SUGAR = 150 G

Particle size	Wt. of sugar retained	Cumulative wt. retained	% cumulative retained	% pass (100 - % cumulative)
1 mm	2 g	2 g	2/150 x 100 = 1.33 %	100 – 1.33 = 98.67%
0.59 mm	14 g	16 g	16/150 x 100 = 10.66%	100 - 10.66 = 89.34%
0.297 mm	36 g	52 g	52/150 x 100 = 34.66%	100 – 34.66 = 65.34%
0.21 mm	97 g**	149 g	149/150 x 100 = 99.3%	100 – 99.3 = 0.7%

* This means that most of sugar can not pass through the sieve

https://www.youtube.com/watch?v=JcLtXblqqGg



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Thank You