Crushing, Grinding, Milling, Sieving

Grinding

- **Disintegrating operation**
- Aim:
  - reduction of the particle size

Grinding

- **Grinding:** the mechanical process of ***reducing particle size of solids***, while the ***specific surface area increases*** parallel with the reduction of the particle size. It is commonly done by mills.

  - **Crushing:** the division of the original particle into some rough particles

    (work in the 'mm'-range)

  - **Milling:** the division of the rough particles and gain fine powders of the grinded materials (fine grinding)

    (work in the '100μm'-range)

  - **Micronization:** special grinding process where the particles can be isometric

    (work in the '1-20μm'-range)

Grinding

Grinding can be:

- ***Preparatory process***- preparing products with defined particle size to produce the appropriate dosage form

- ***Finishing process*** – applicable pharmaceutical product e.g. species (tea mixture)

Grinding

Advantages:

- *increasing of the special surface area*→ increase the dissolution rate → better-faster dissolution→ better bioavailability
- *better flowability* (shape and size of the particles are better)→ provide a greater uniformity (tablets, capsules)
- *equal sized particles* (homodisperse system) → well-controllable processes (drying)

Grinding

Disadvantages:

- *energy-consuming process*, the temperature of the system can be increased and this can lead to undesired changes (oxidation)
- *polymorphs* can be formed (the polymorphs may have different therapeutical effects)
- *air-adsorption* is an adverse process as well (tableting of aerophil substances)

The physics of grinding

- The solid particle can be crushed where the continuity of material structure is crushed („defect in crystals”):
  - discontinuity
  - holes
  - cracks
  - occlusions (inclusions)
  - edges and peaks (energy-rich areas of the surface)

Energy of grinding

<u>**Energy-requirement of grinding**</u>:

Energy-requirement of grinding means how much kWh energy is needed to reduce the surface of the particles of one ton sample.
[kWh/t]

Energy of grinding

***Rittinger „surface" theory*:**

The energy required for grinding is directly proportional with the increase of surface. (square relationship) (brittle materials or fine milling)

***Kick-Kirpichev „volume" theory*:**

The energy required for grinding is directly related to the decrease in particle volume (coarse milling)

***Bond combined theory*:**

The energy required for the size reduction is inversely proportional to the square root of the particle size

Grinding

(Rittinger µm /Bond µm-mm / Kick mm-cm)

C_1,C_2,C_3 constants of the materials

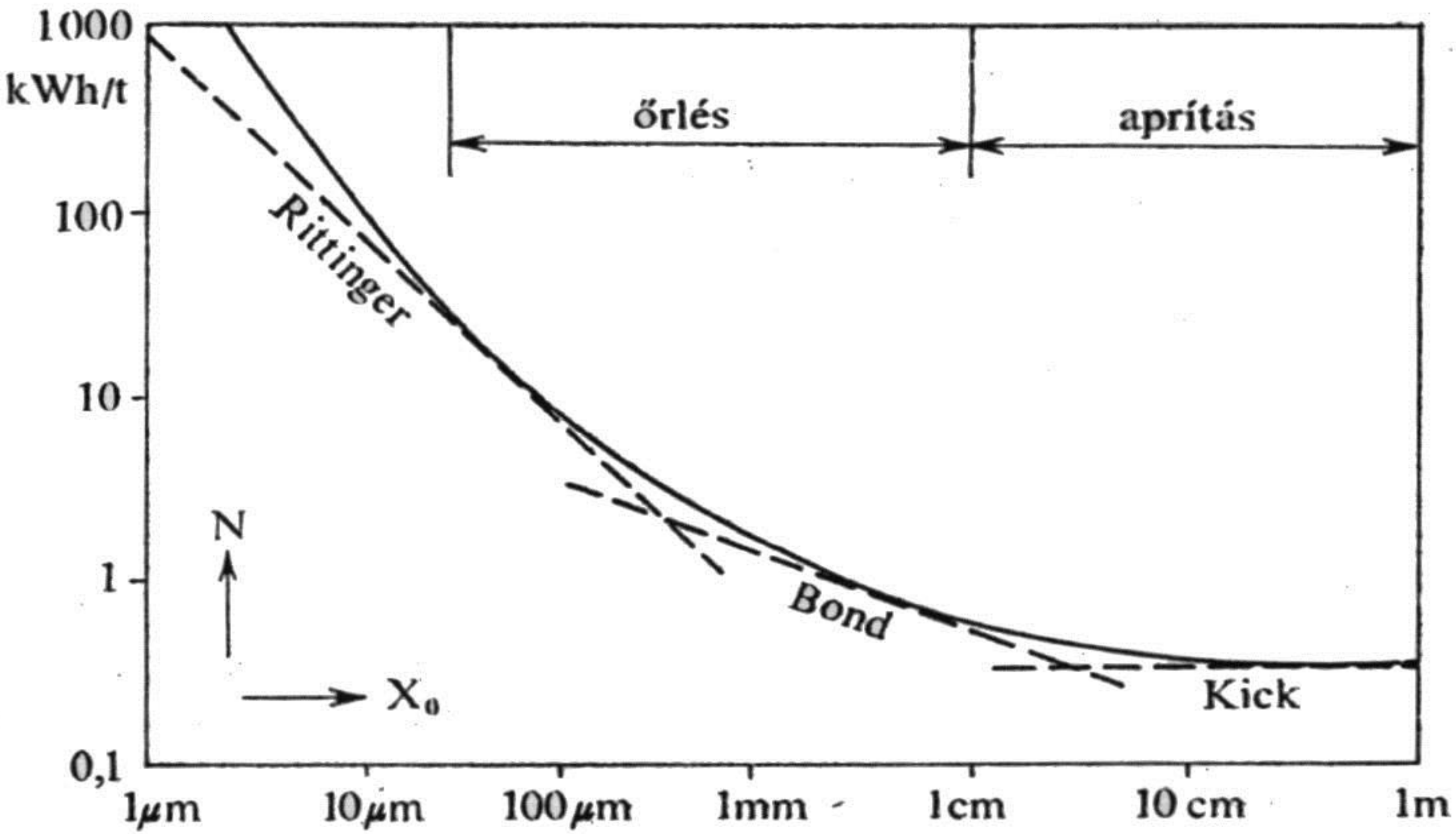
X_0 initial particle size

X desired particle size

	Rittinger	Bond	Kick
X- work needed for crushing of materials with this particle size	X^2	$X^{2,5}$	X^3
numbers of particles in the case of same volume	$1/x$	$1/\sqrt{x}$	$\lg 1/x$
N – grinding work per mass/volume unit (p. size goes from x_0 to x)	$N_R = C_1(1/x - 1/x_0)$	$N_B = C_3(1/\sqrt{x} - 1/\sqrt{x_0})$	$N_K = C_2(\lg 1/x - \lg 1/x0)$

Grinding

Theories can be used in different ranges of particle size



Grinding

Efficacy of grinding:

- ***Degree of grinding*** – this is the ratio of the initial and desired particle size

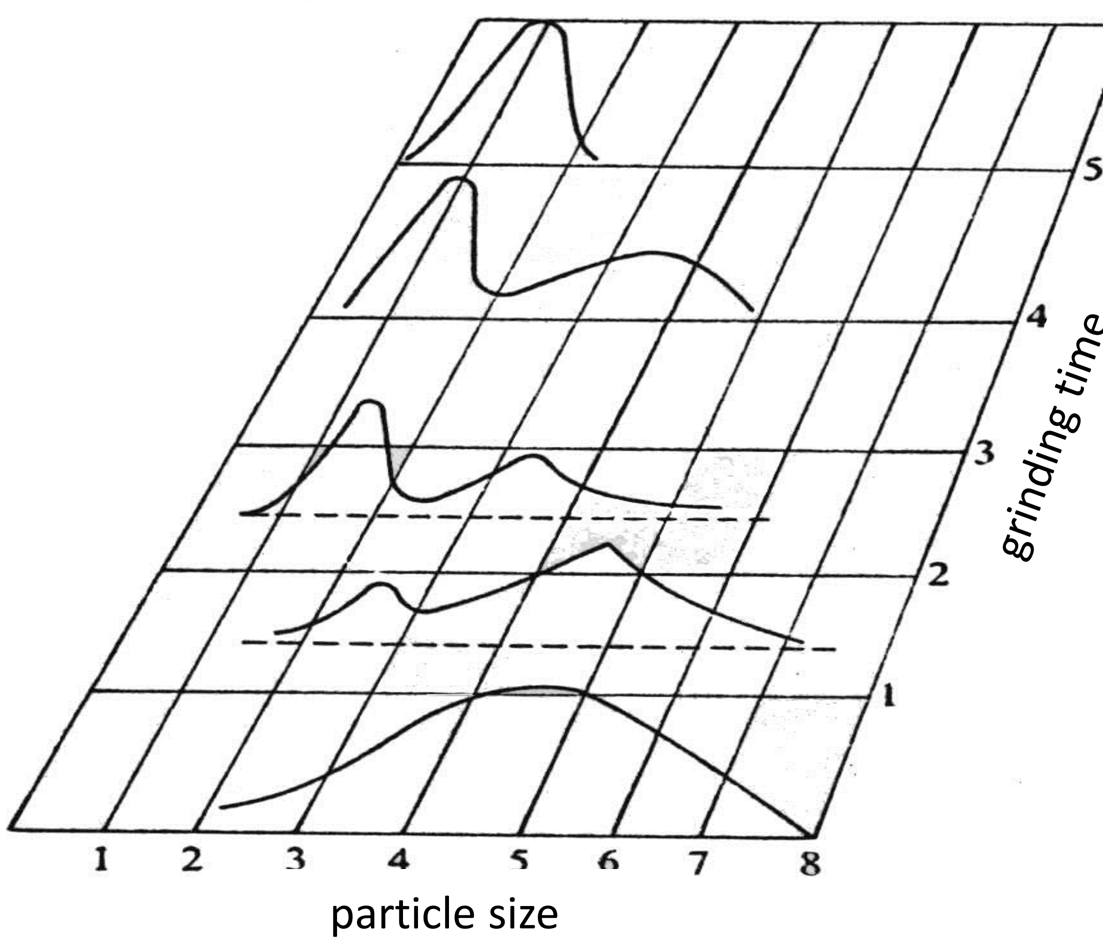
  (This is a specific parameter, and there are specific indicators:

  - max. size,
  - mean size,
  - 80% of the mean size)

- The reciprocal value : grinding ratio

Grinding

The particle size distribution is plotted against time



Grinding

- The grinding has a kinetic, it is changing in time: the grinding ratio (speed) is the amount of the material what is ground (to the desired particle size) in unit time. (that amount of newly grinded material what can fall through the chosen sieve.)

- The specific value of the grinding ratio (R) can be referred to the non-ground amount of material (at the same time, because it changes).

$$dR/dt = - c*R$$

$$R = R_o e^{-ct}$$

R_o = amount of material remaining on the sieve at t = 0

t = time

c = proportionality factor

- *The exponential decrease of the grinding ratio can not always proved in the practice*
- (*there is a linear ratio between time and sieve residue in logaritmic scale*)

Grinding

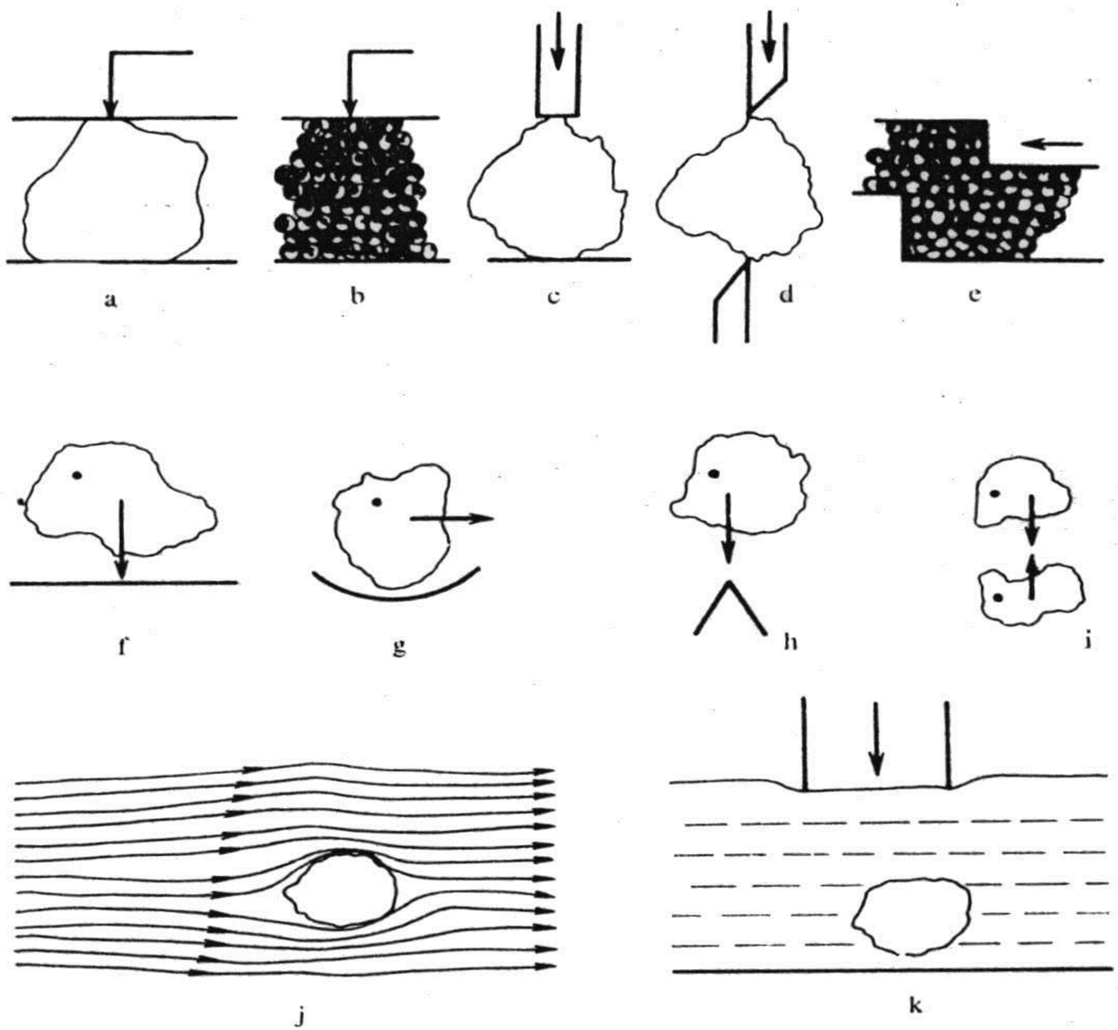
- Rosin - Rammler – equation is more practical

$$\mathbf{R=R_o e^{-ct^n}}$$

- n = empiric factor, (uniformity coefficient)
- The work is exponencionally increasing with the decrease of the particle size.
- The time needed for the grinding is exponencionally increasing with decrease of the particle size.

Grinding

- Stress mechanisms



a	compressing force
b	compressing force + frictional force
c	striking force
d	cutting force
e	Shearing force (stress)
f	impact force
g	impact force (indention)
h	impact force (edge)
i	impact force (particles)
j	wet grinding
k	wet grinding

Grinding

- ***Slower acting equipment*** **(<100 m/s)**

  compressing force (a)

  compressing force + frictional force (b)

  striking forces (c)

  cutting force (stationary and moving blades) (d)

  shear stress (e)

- ***Fast acting devices*** **(>100 m/s)**

  huge impact forces (f, g, h, i)

- **Wet grinding (j, k)**

  The viscosity of the applied liquid (what is more than the water has) compels to impaction and shear the particles with the wall of the device (colloid mill)

Grinding

- ***The needed type of force:***

impaction	compressing	striking
cutting	shearing	friction

- ***Different types of material***

hard, abrasive	medium hard	soft
rigid	flexible	hardy
plastic	fibrous	fused

Grinding

Methods of grinding

- **Open-circuit process –** the particles pass through the disintegrator (shredder) **just once**

  multi-stage - more shredding equipment (subsequently grind the particles)

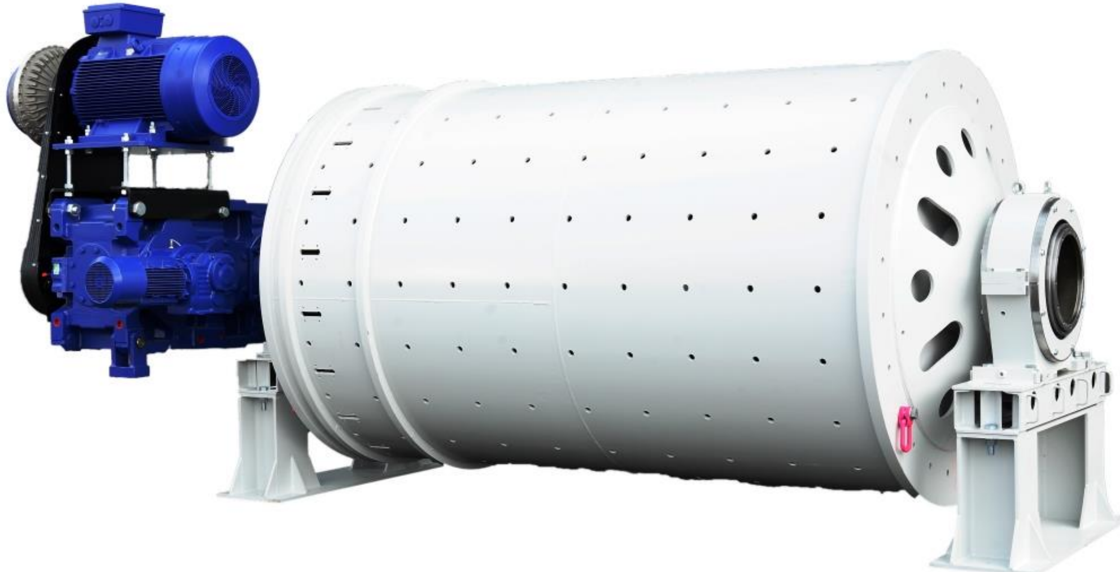
- **Closed-circuit process –** after one grinding cycle the particles pass through a size-separation device or classifier (sieve), and the oversize particles are returned to the grinding chamber for further milling.

DISINTEGRATORs

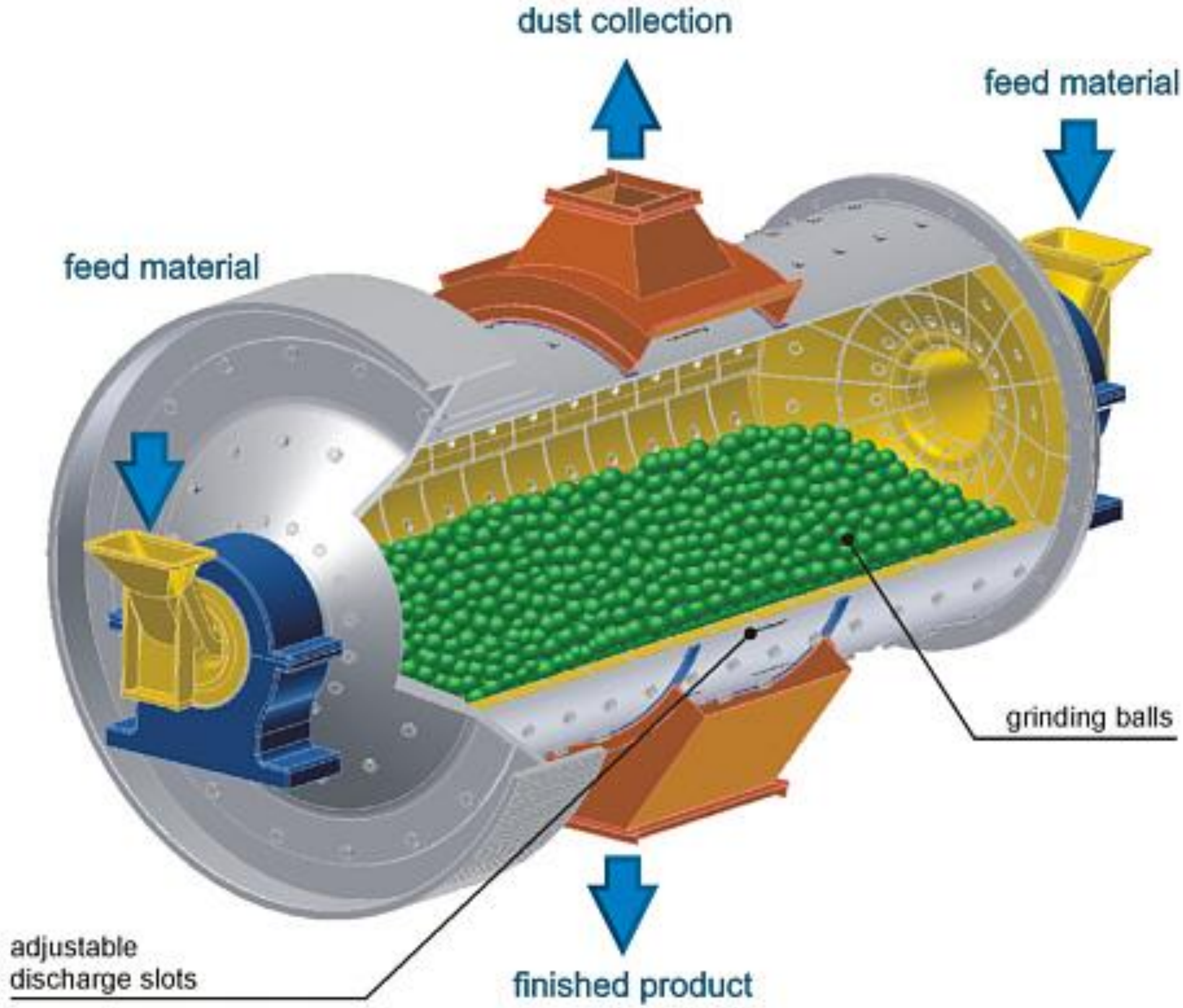
Types of disintegrators:

50 -5 mm	coarse mill (crusher)
5 -0,1 mm	fine mill
0,020 -0,001 mm	colloid mill
(1-20 μm)	micronizator

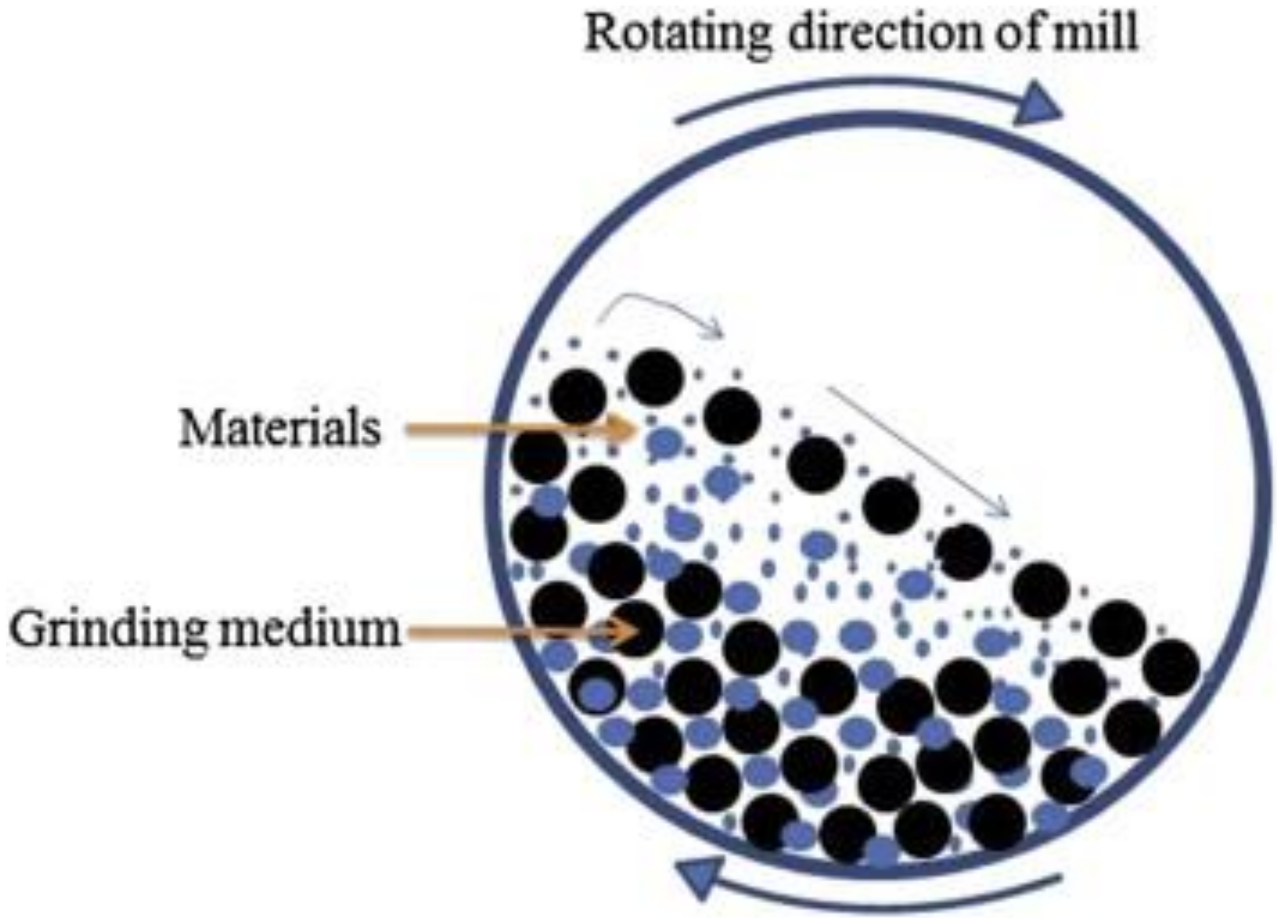
Disintegrators
Ball mill



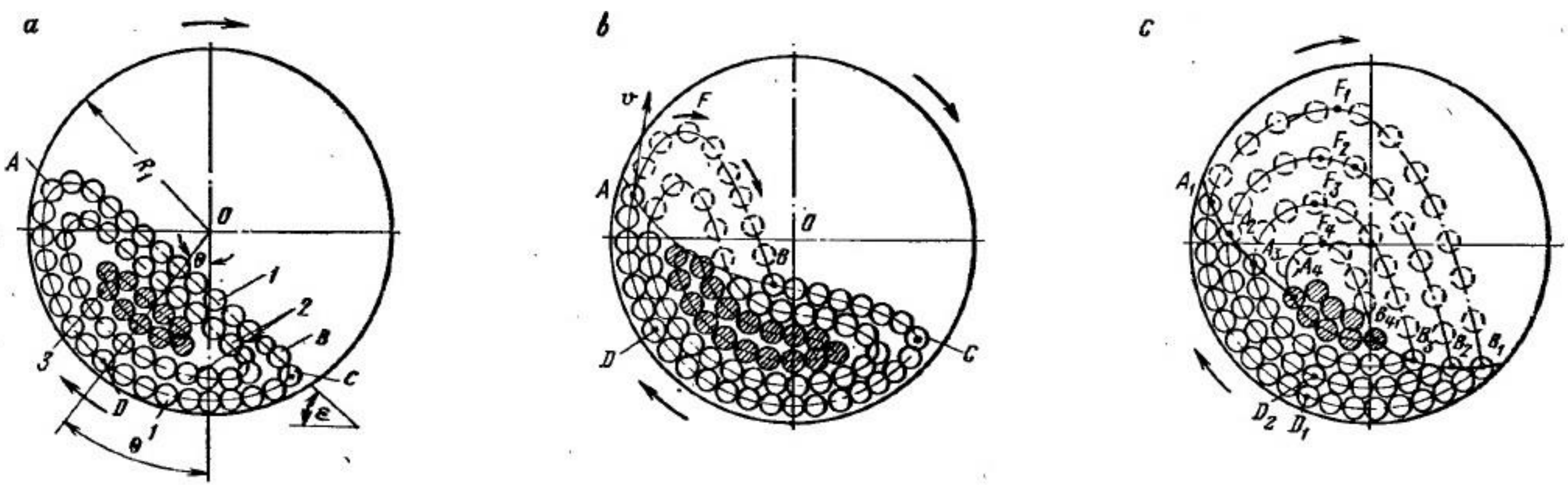
- Consists of a hollow cylinder, which rotates around its horizontal axis
- The cylinder contains balls, which are free to move (ceramic, quartz, stainless steel)
- Wet or dry milling


dust collection
feed material
feed material
grinding balls
adjustable
discharge slots
finished product

Ball mill



Ball Mill



DISINTEGRATORs
Ball mill

<u>EFFECT</u>: abrasive, impacting, shearing, frictioning

- ***cataract effect*** **– collision of particles moving on a defined orbit (fine particles)**
- ***cascade effect*** **- sliding, rolling, shearing, rubbing effects of grinding elements (balls) (rough particles)**

<u>SPEED OF ROTATION:</u>

- **If the rpm is too low: → *cascade effect***
- **In optimal range of rpm: → *cascade and cataract effect***
- **If the rpm is too high (more than the critical rotation speed) → *no any grinding effect***

DISINTEGRATORs
Ball mill

- **Critical (rotation) speed- the balls move together with the drum (equal speed (it sticks to the wall))→ no grinding**

$$n_{cr} = 42,3/\sqrt{D}$$

- *calculation:*

D = 2r = diameter of the drum (m)

n = rotation speed (min^{-1})

m = mass of the balls (kg)

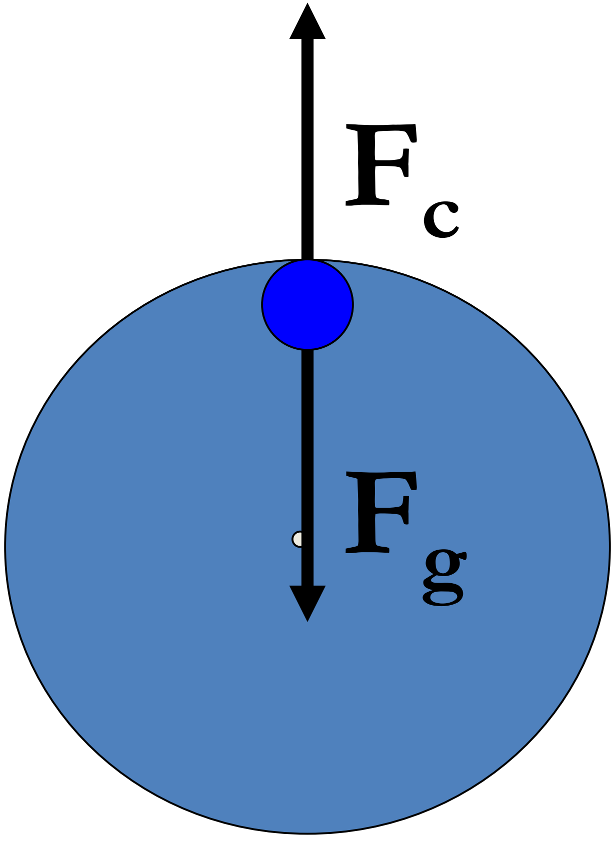
g = acceleration of gravity (9,81 ms^{-2})

F_c = centrifuge force (N)

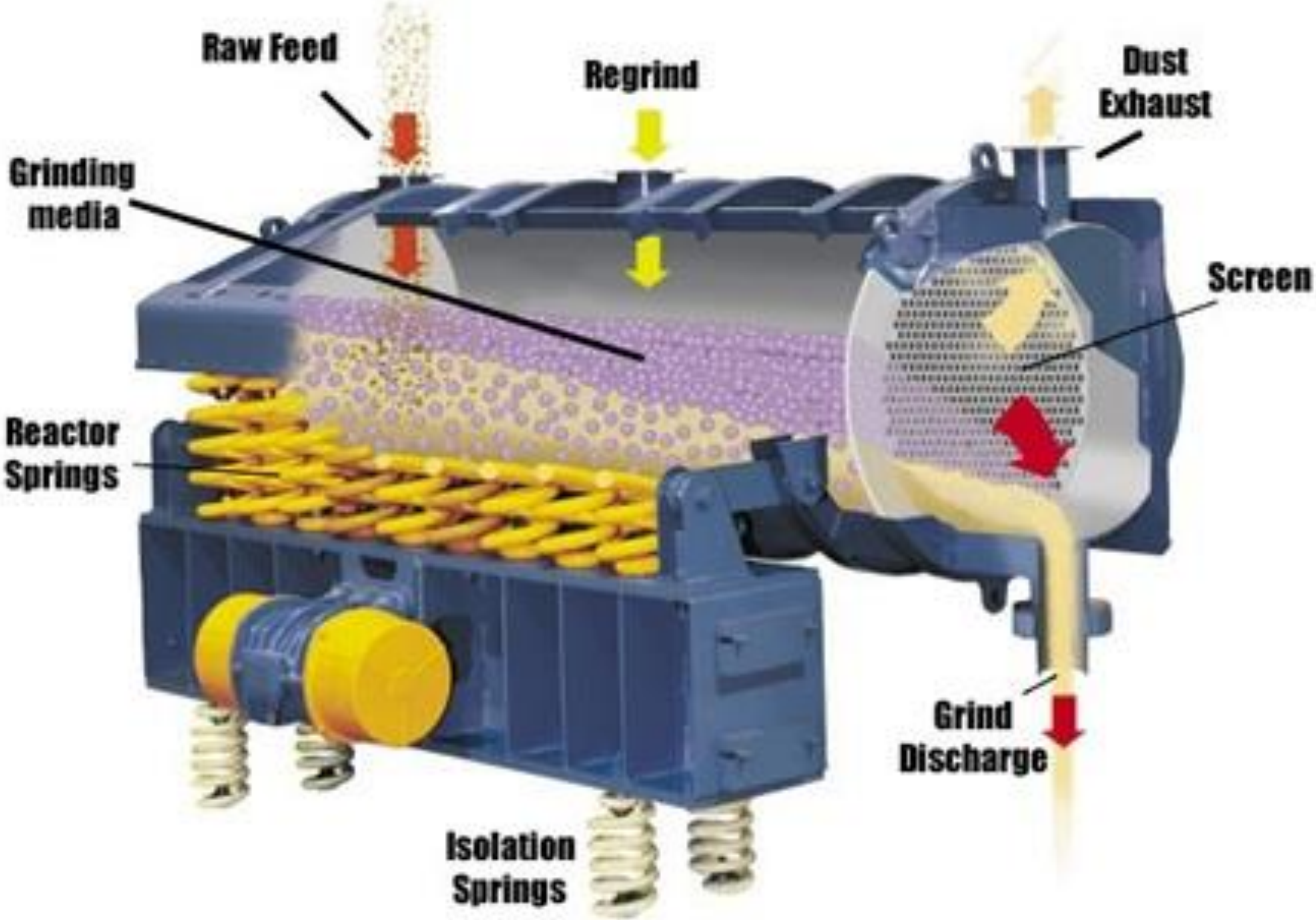
DISINTEGRATORs
Ball mill

- **Critical speed -**

$$F_g = F_c$$

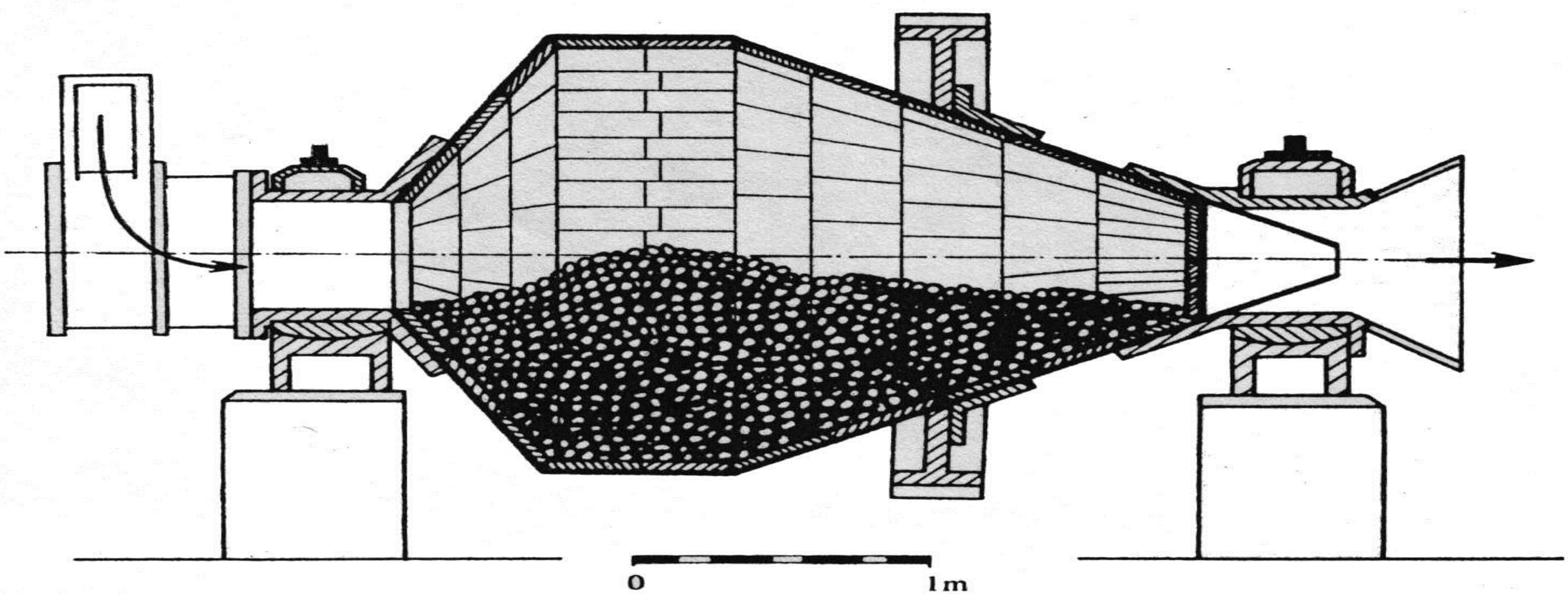


DISINTEGRATORs
ball mill



DISINTEGRATORs
Ball mill

- **Optimal speed range of the drum: 23-28/√D**
- Disadvantages: multi-step operation, poor efficiency

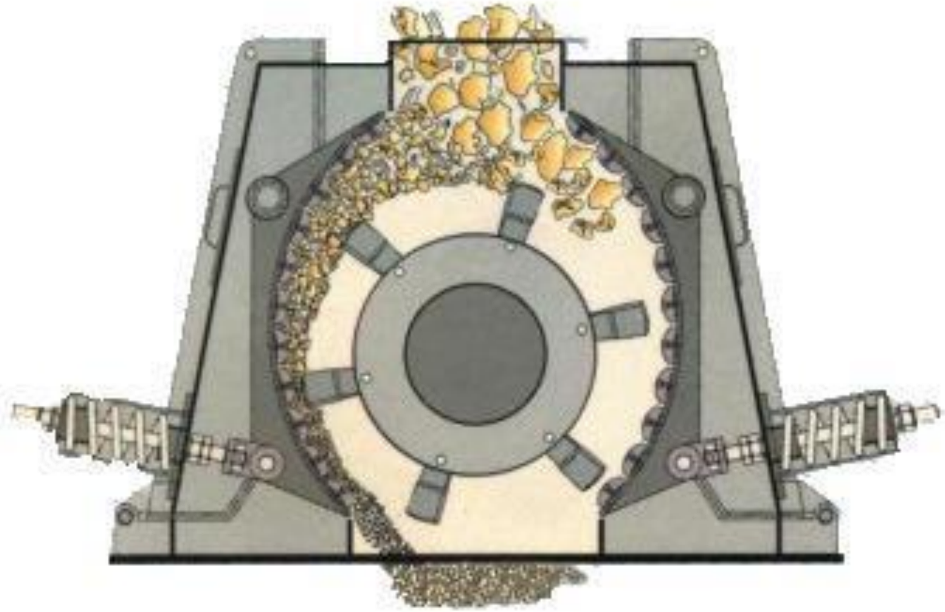


DISINTEGRATORs
Rod mill

- Similar to the ball mill, where the grinding effect is caused by rods instead of ball
- In some cases, the rod mill is combined with ball mill ➔ rod mill with applied balls

DISINTEGRATORs
Hammer mill

- The hammers rotate freely around a fixed point on the central rotor and when material is fed into the mill
- The rotor spins at a high speed so the hammers can shred the material
- The wall of the drum is ribbed.



Hammer mill

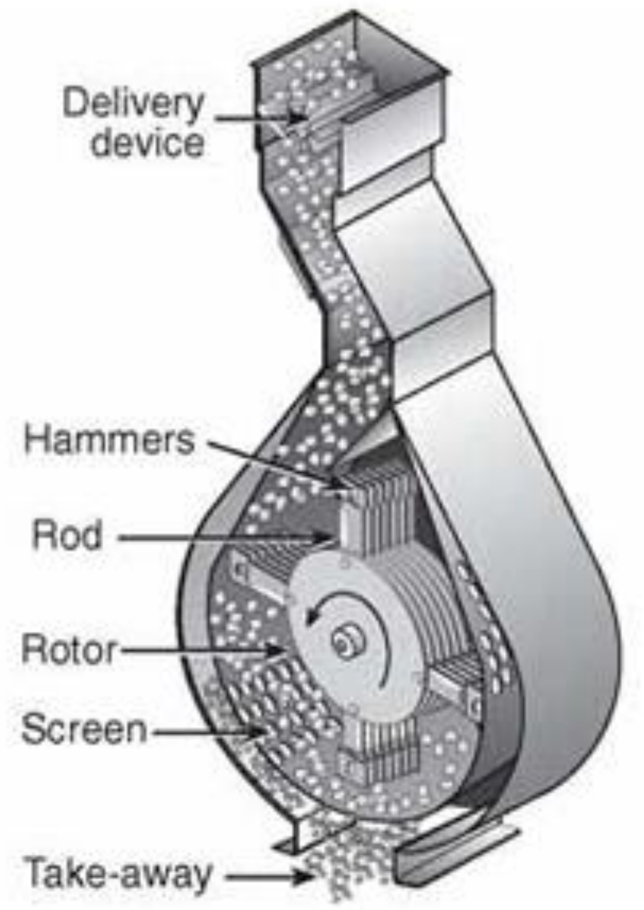
DISINTEGRATORs
Hammer mill

<u>The disintegration efficacy depends on:</u>

- **-rpm**
- **-structure of the ribbed wall**
- **-sieve (distance of the threads)**

Maximal efficiency: 80 kg/h

Minimal particle size: 70 μm



DISINTEGRATORs
Dispersion mills/Colloid mills

- They are capable to grind (micronize) the fine particles till to reach more smaller particles.

Examples:

- **<u>Disc mill: </u>**the rotor equipped buffle plates rotates in a proper hole of the stator -
- **<u>Jet mill:</u>** high-speed liquid or gas flow is used to break the particles (shear force)
- **<u>Vibration mill: </u>**the centrifugal force springs the mill's drum out of the center point, this imbalance is stopped by a spring if the motor is stopped. This cycle is sequential, eventually the motion is a vibration-rotation.

  Minimal particle size: 1 μm

DISINTEGRATORs
Colloid mills

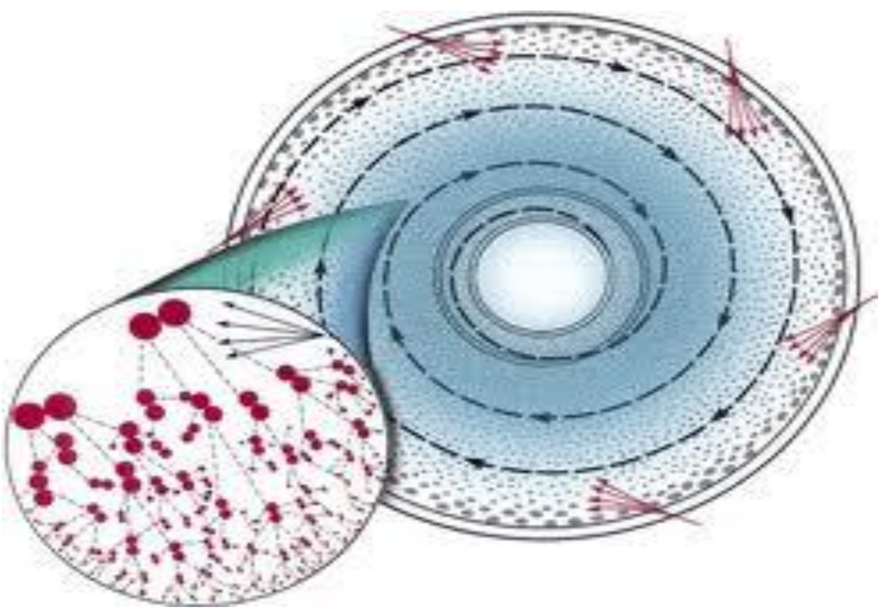
The colloid mills consist of stator and rotor

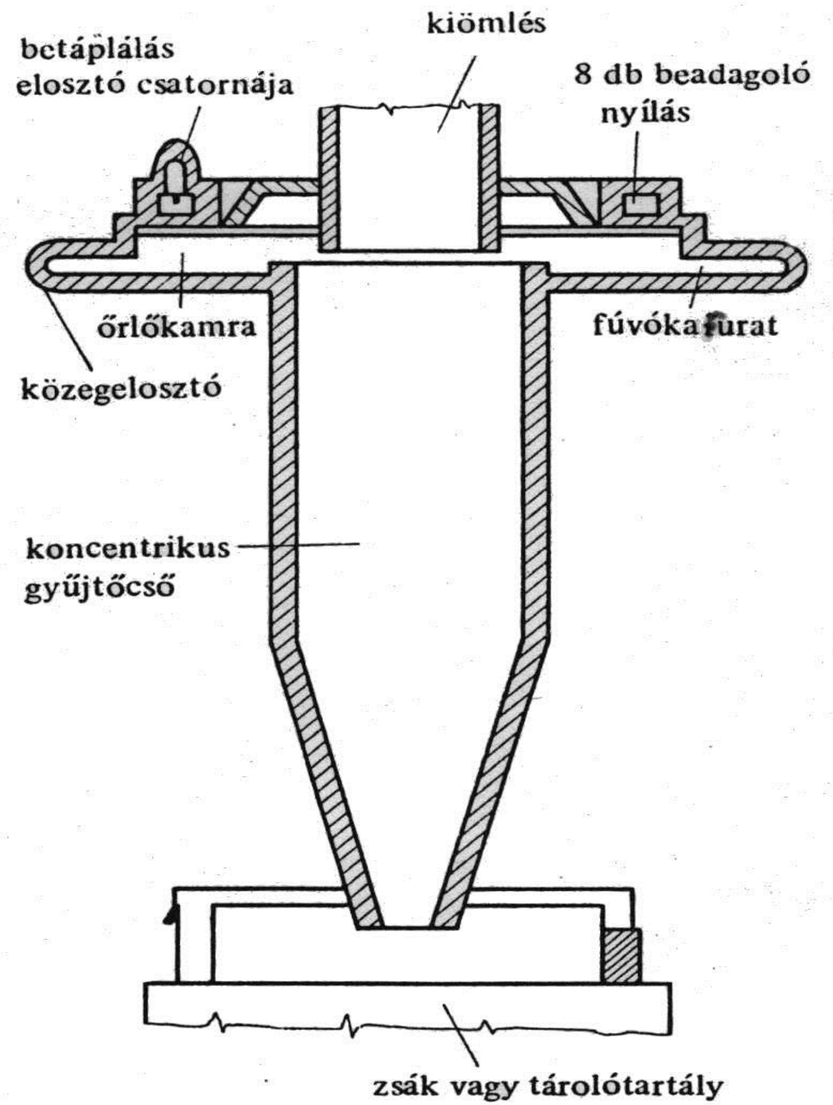
The minimal particle size what can be achieved is below 0.1 μm

Grinding force: shearing

DISINTEGRATORs - JET MILL
Micronizer

- The coarse powder gets by air on a tangential orbit.
- The particles impact with each other and with the wall, too.
- The smaller particles move to the middle hole of the reaction space.





DISINTEGRATORs - JET MILL
Micronizer

- The size of the isometric particles
  1 -20 μm

- (It is commonly applied in case of antibiotics, e.g. sulfonamide)

- 50-100 kg - 2-3 μm particles can be prepared in one hour

SIEVING

Ph.Eur:

- **Sieves are made of sieve cloth, which is an inert material.** The distance of the wires is a characteristic data of the sieve. (sieve number)

- The prescribed size of the particles is marked in brackets after the name of the material in µm. (sieve number)

SIEVES

Ph.Hg.VII.		Ph. Eur
Sieve No.	mm / μm	μm
I.	6,3 / 6300	8000 / 5600
II.	4,0 / 4000	4000
III.	2,0 / 2000	2000
IV.	1,2 / 1200	1400 / 1000
V.	0,80 / 800	1000 / 710
VI.	0,320 / 320	355 / 250
VII.	0,160 / 160	180 / 125
VIII.	0,063 / 63	63

SIEVES

Ph.Eur.:

Determination of particle size with sieve analysis:

Powder with sieve number VI. means that 95% of the powder passes through the No.VI. and not more than 40% of powder passes through the No. VII.

- Coarse powder: > 95 % 1400.– max. 40 % 355.
- Moderately fine powder: > 95 % 355.– max. 40 % 180 .
- Fine powder: > 95 % 180.– max. 40 % 125.
- Very fine powder: > 95 % 125 – max. 40 % 90.

Determination of particle size with microscope:

- 10 – 100 mg suspended sample → 10 μg sample of the suspension has to be examined.

SIEVING

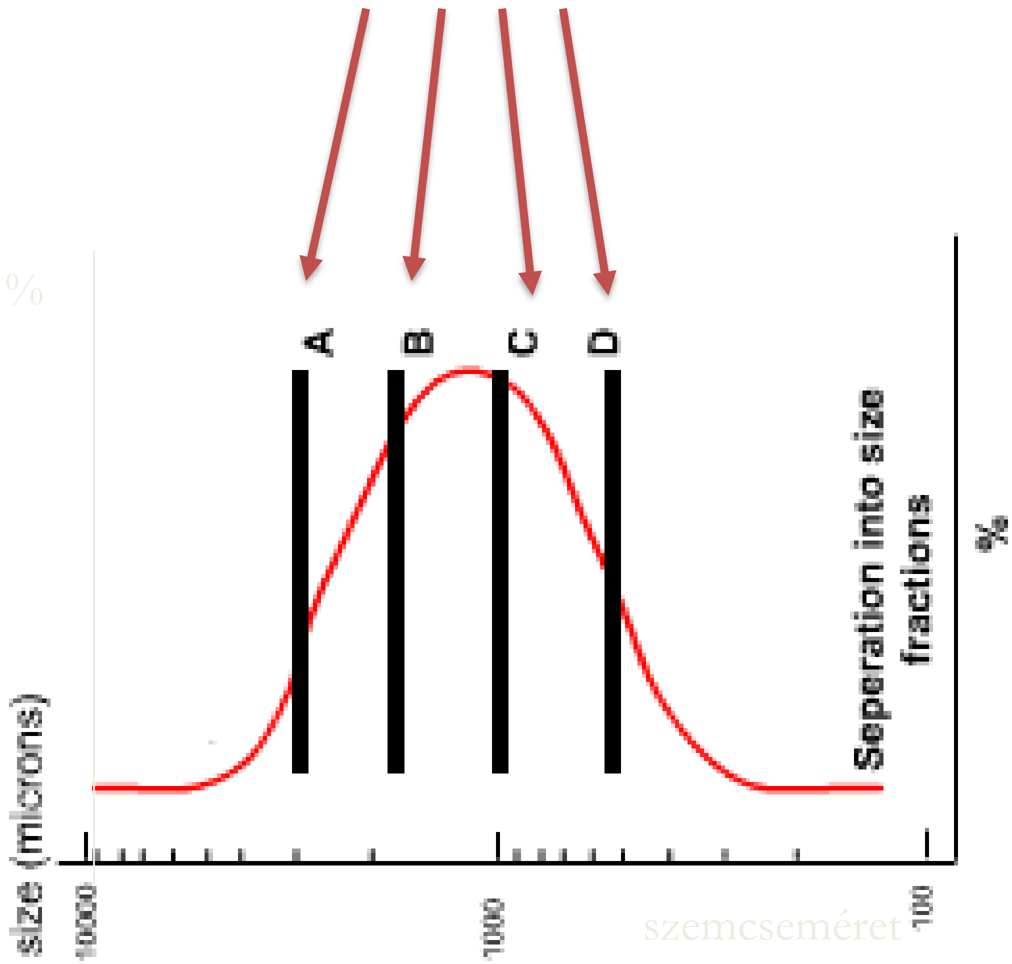
Ph.Eur. 6

No	Distence of wires (mm)	Thicknes of wires (mm)	Latin name of fractions of the grinded material if it is separated by sieving
I.	**6,3**	2,5	roughly grinded (scissus)
II	**4,0**	1,6	grinded (conscissus)
III.	**2,0**	1,0	moderately grinded(semiconscissus)
IV.	**1,2**	0,8	well grinded(minutim conscissus)
V.	**0,80**	0,5	rough powder (pulvis grossus)
VI.	**0,32**	0,2	moderate-fine powder (pulvis semisubtilis)
VII.	**0,16**	0,1	fine powder (pulvis subtilis)
VIII.	**0,063**	0,04	very fine powder (pulvis subtillisimus)

Sieve analysis

Particle size analysis

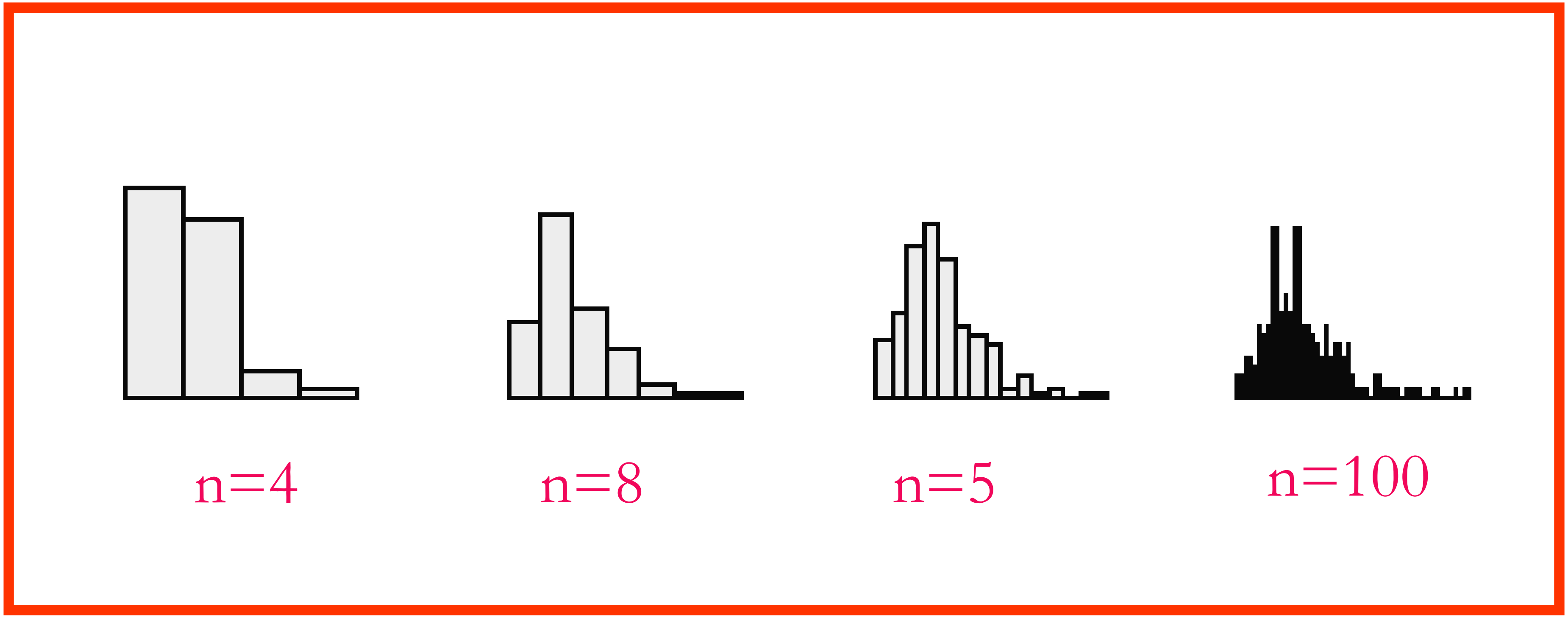
sieves



Particle size distribution

Illustration of distribution

the number of particle fraction



Sieve analysis for examinatation of particle size distribution

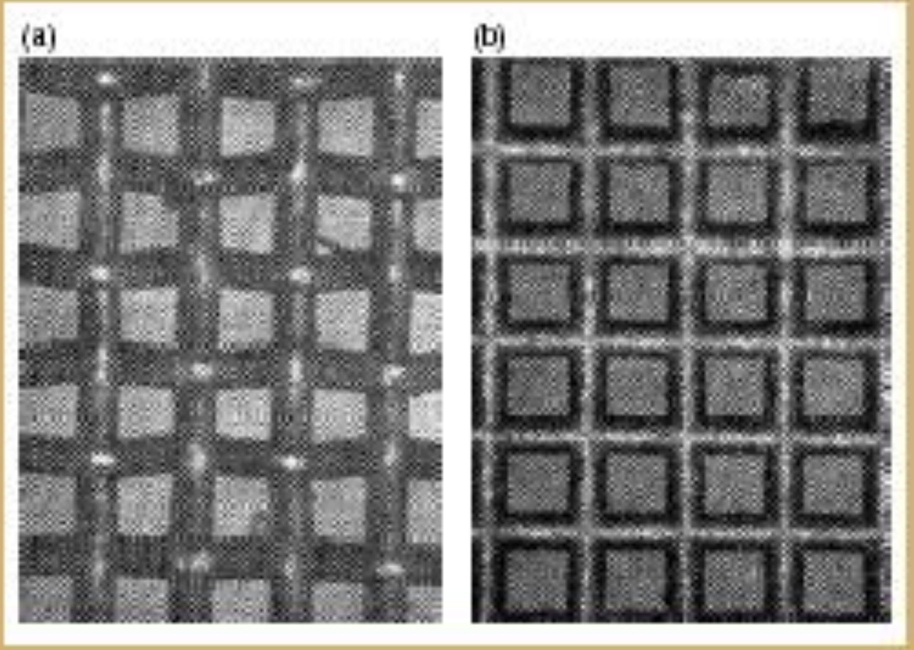
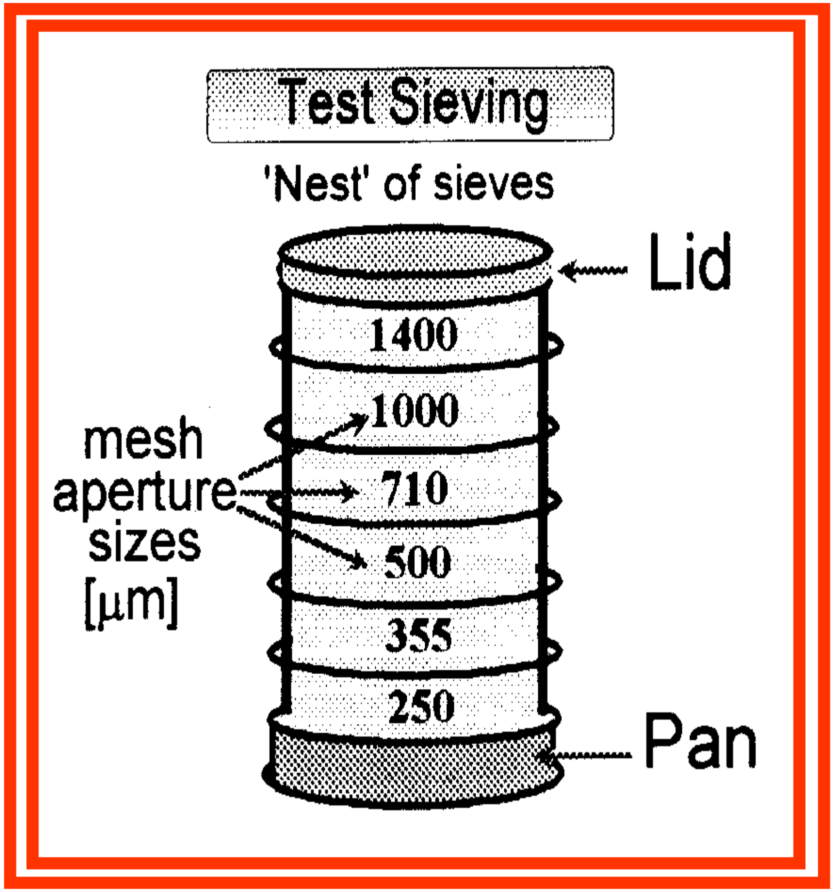
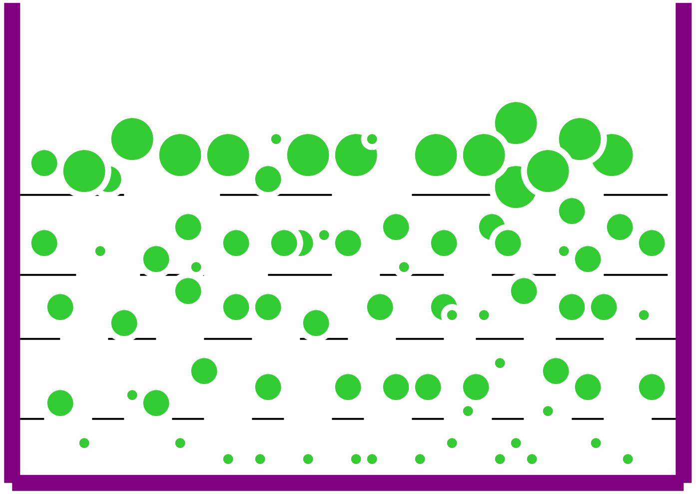


Figure 1: Photomicrographs of (a) woven-wire screen and (b) micromesh screen.

Sieve analysis





Sieve analysis for examinatation of particle size distribution

Sieve analysis

advantage

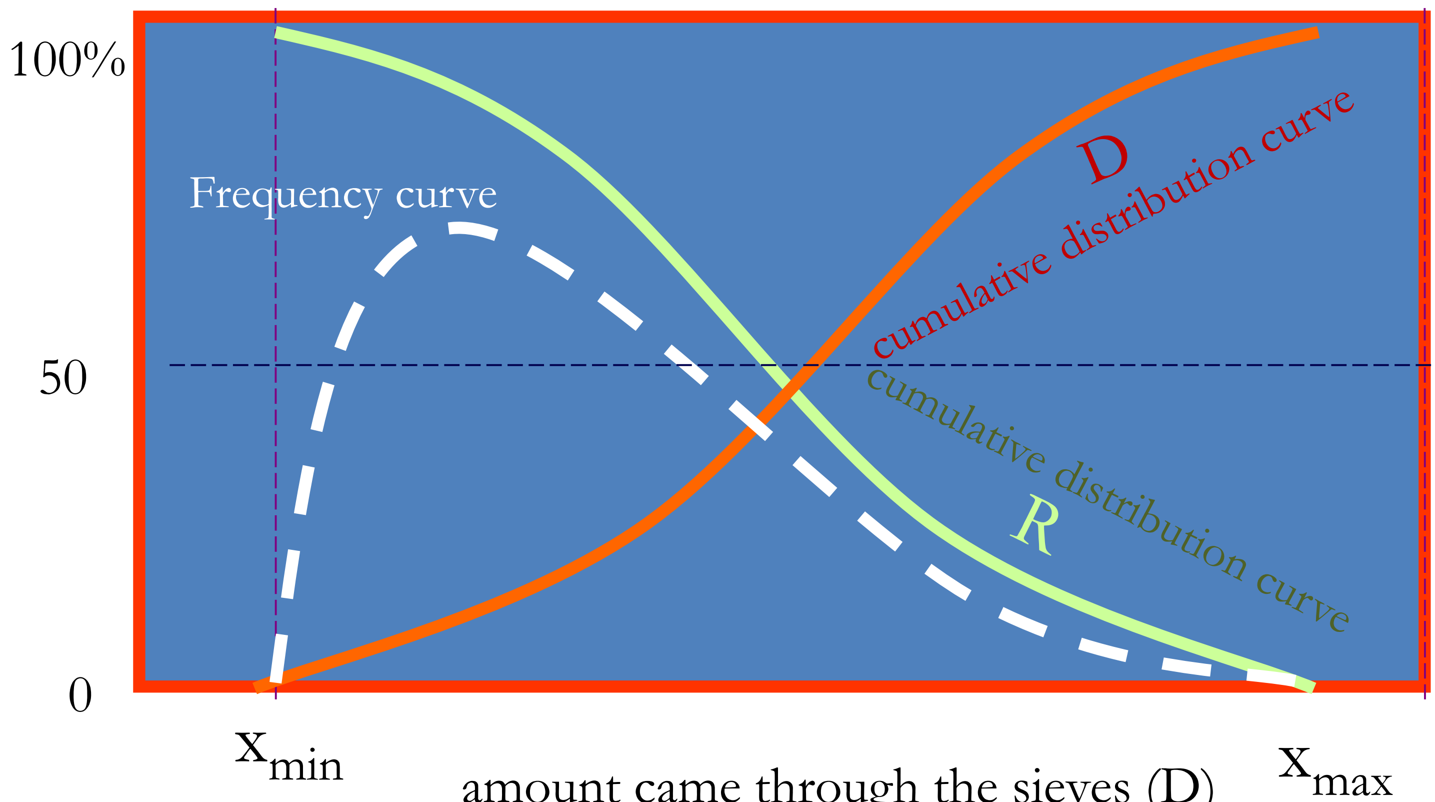
- possibility to examine wide particle size distribution
- simple examination
- sieves can be calibrated
- particle fractions can be separated

disadvantage

- large amount of material
- lowest particle size limit $d > 20\ \mu m$
- not too fast

Particle size

Dispersion and frequency curves



amount came through the sieves (D)
amount remained on sieves (R).

Thank you for your attention!