



NOVA SCHOOL OF  
SCIENCE & TECHNOLOGY

# Course: OSF

# Operações Sólido Fluido

# Solid Fluid Operations

LEQB/MIEQB, 2023/24

Chemical and Biological Engineering Section, Department of Chemistry, FCTNOVA

OSF/FCTNOVA

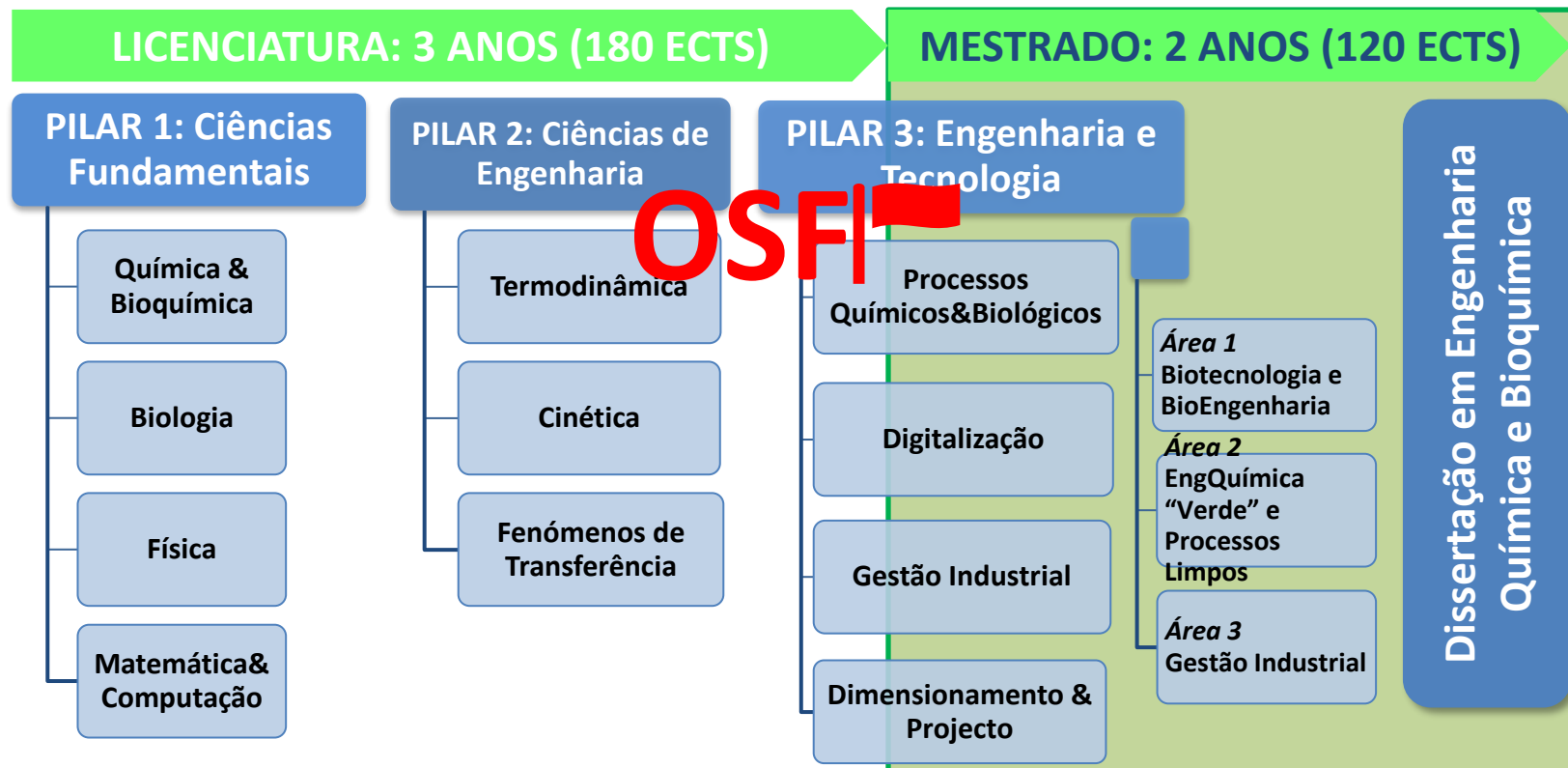
# Instructors

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- **Prof. Rui Oliveira**
  - Office 628 DQ
  - Email: [rmo@fct.unl.pt](mailto:rmo@fct.unl.pt)
  - Tutoring: **WED 14:00-15:00 AM**
- **Prof. Isabel Esteves**
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  - Tutoring: **WED 14:00-15:00 AM**
- **Prof. Rafael Costa**
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  - Tutoring: **WED 14:00-15:00 AM**

# OSF topics of study

Mechanisms and processes involving the handling and/or processing of bulk solids (e.g. powders, granules, pellets) and fluids (gas, liquid, flow deforming material) in the chemical & biological industry.



<https://www.thechemicalengineer.com/features/bulk-solids-handling-perspective-on-a-professional-blind-spot/>

“It is estimated that >70% of everything we use or consume involves **bulk solids** handling somewhere in its lifecycle”



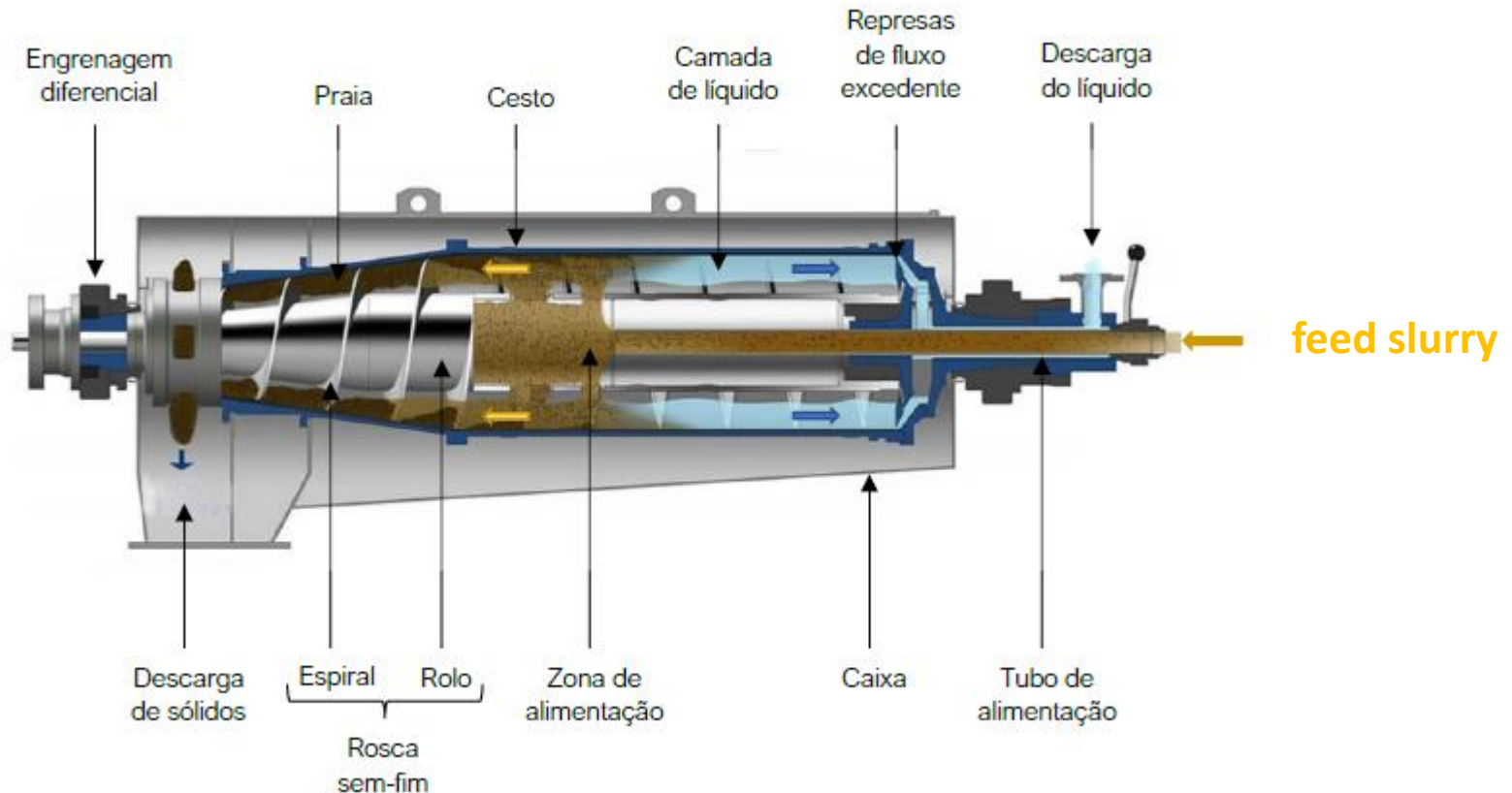


# Example 1: Design of a settling tank



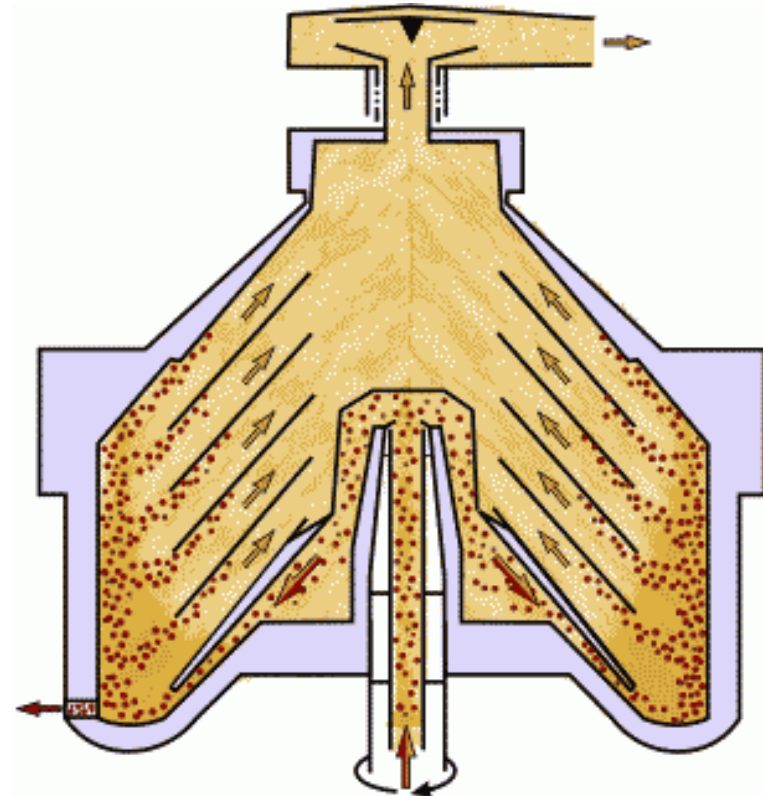
# Example 2: Decanter centrifuge

Continuous separation of the solids and liquids in a feed slurry by centrifugation



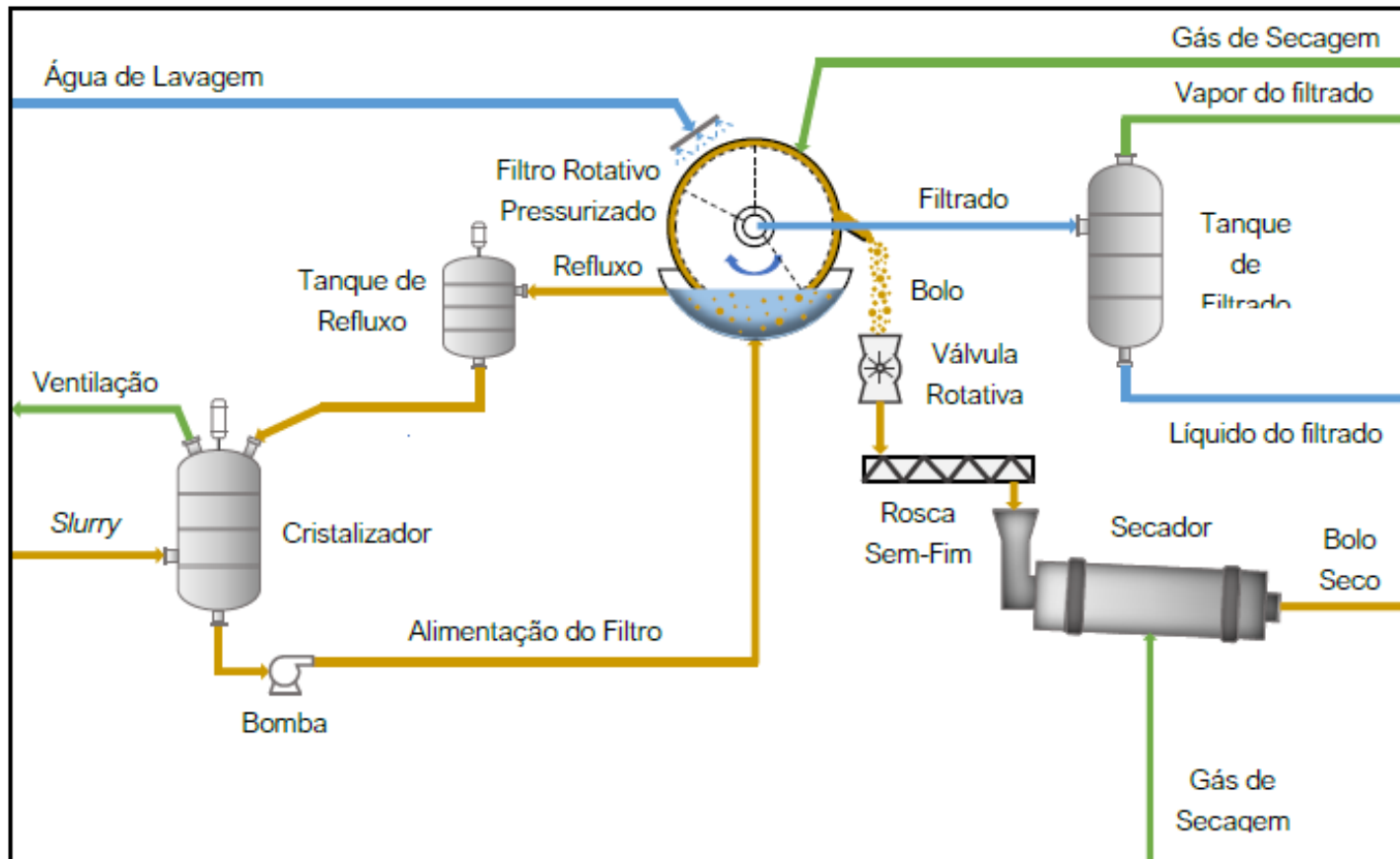
Adapted from: <https://www.flottweg.com/pt/linha-de-produtos/decanter/>

# Example 3: Disk stack centrifuge



[https://www.youtube.com/watch?v=GhT\\_N\\_-TIBY](https://www.youtube.com/watch?v=GhT_N_-TIBY)

# Example 4: Pressurized Rotary Filter



Adapted from a Purified Terephthalic Acid (PTA) process flowsheet

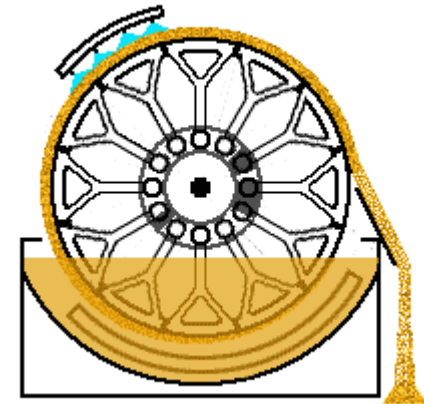
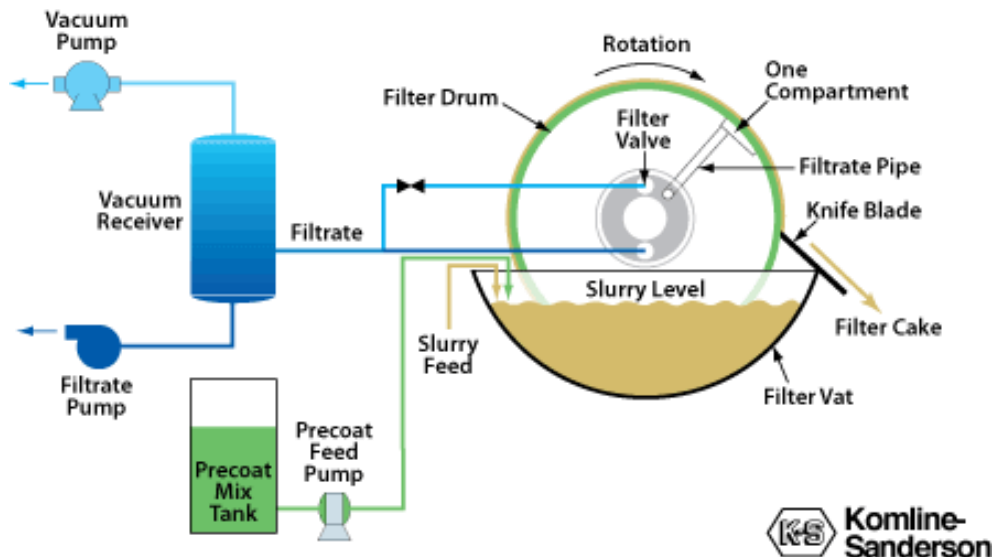


# Example 5: Rotary vacuum filter

Continuous separation of a slurry feed in its solid and liquid components by filtration. Integrated filtration + washing + drying of the solids cake.

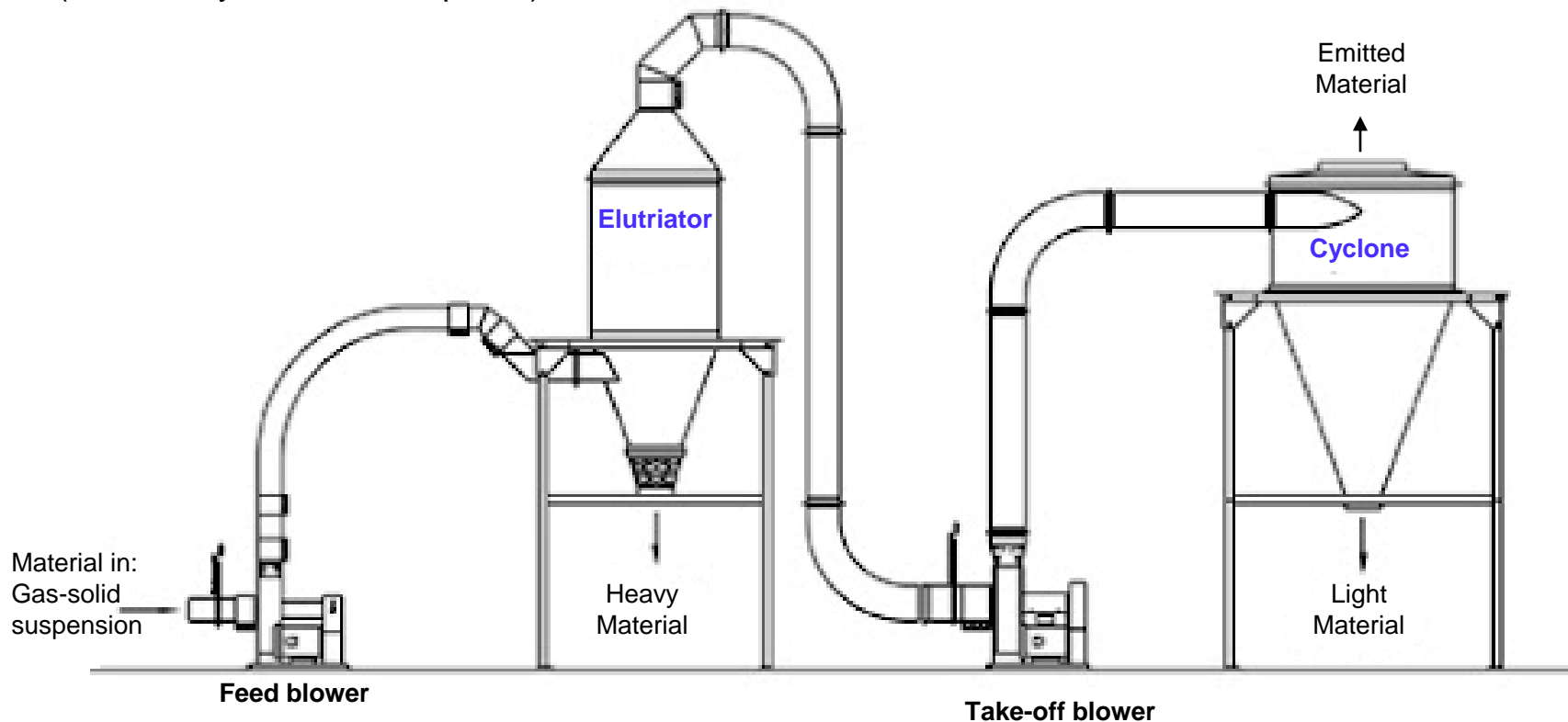


**Rotary Drum Vacuum Filter  
Precoat Discharge**



# Example 6: Gas elutriator + Cyclone

This plant separates heavy solids from light solids in a two-stages separation process. The first stage is an elutriator that uses the gravity force to separate the heavy/light solids streams. The second state is a cyclone separation unit that uses the centrifugal force to separate the heavy/light solids streams (more to Cyclones in Chapter 7)



# Syllabus

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## **Chapter 1. Characterization of solid particles**

General concepts. Single particles characterization. Particle size distributions based on weight, number and surface area. Mean size based on number or weight data. Particles shape. Methods for particle size measurement.

## **Chapter 2. Size reduction of solids**

Mechanisms for size reduction. Dynamics of size distribution. Energy for size reduction. Equipment for size reduction.

## **Chapter 3. Motion of particles in a fluid**

Characterization of flow around a sphere (laminar and turbulent flow). Skin and form drag friction. Stokes law. Newton law. Terminal settling velocities. Extension to non-spherical particles. Transient motion of particles: vertical acceleration under gravity.

# Syllabus

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## Chapter 4. Sedimentation

Free and hindered settling. Fine and coarse settling. The thickening process. Kynch method. Design of settling processes.

## Chapter 5. Centrifugal separation

Types of centrifuges. Mechanical design. Fluid pressure and liquid surface form. Separation of two liquids. Separation between suspension solids and liquids. Filtration using centrifuges. Prediction of separation efficiency. Gas cleaning. Cyclone design. The theoretical cut-off model. Recovery efficiency. Pressure drop in cyclones. Electrostatic separators.

**Teste-1: Chapters 1-5**





# Syllabus

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## Chapter 6. Flow of fluids in packed columns.

Characterization of flow in packed columns. Characterization of packings. Calculation of friction factors and pressure drop. Extension to vacuum columns. Economical design of packed bed columns. Heat and mass transfer.

## Chapter 7. Fluidization

Description of fluidization phenomena. Gas and liquid fluidization. Bubbling behavior. Calculation of minimum fluidizing velocity. Calculation of bed expansion.

## Chapter 8. Filtration

Filtration theory. The general filtration equation. Cake and filter resistance. Compressible and incompressible cakes. Filtration equipment. Design of plate and frame filters and design of rotary vacuum filters

**Teste-2: Chapters 6-8**



# Laboratory sessions

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- 2 experiments (size reduction + sedimentation + filtration)
- 2 weeks (25.11 – 6.12)
- Chemical engineering lab 5<sup>th</sup> floor DQ
- Groups of 4 students in the same P session
- Frequency mandatory
- 1 report

# Frequency & grading

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## Frequency

Mandatory frequency to P (laboratory) and 2/3 of TP.

## Continuous grading

**A** = Laboratory experiment + report; Mandatory

**B** = (Test-1 + Test-2 )/2; Minimum grade = 9.5

**Final grade** = 20% **A** + 80% **B**

## Final exam

**C** - All topics in a single exam at the end of the semester;

**Final grade** = 20% **A** + 80% **C**

# Teaching material

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## THEORY

- **[THE BEST]** J.M. Coulson and J.F. Richardson, Chemical Engineering, II Vol., 2<sup>a</sup> Ed., 1965, Pergamon Press, London
- Slides (always work in progress)

## PROBLEM-SOLVING (TP)

- **List of exercises @ CLASSROOM** (taken from J.M. Coulson and J.F. Richardson, Chemical Engineering, II Vol., 2<sup>a</sup> Ed., 1965, Pergamon Press, London)
- **List of exercises @ HOME** (taken from J.M. Coulson and J.F. Richardson, Chemical Engineering, II Vol., 2<sup>a</sup> Ed., 1965, Pergamon Press, London)
- Calculating machine or LAPTOP  
**NOTE:** Solution of exercises is also available from J.M. Coulson and J.F. Richardson, Chemical Engineering, II Vol., 2<sup>a</sup> Ed., 1965, Pergamon Press, London



# Bibliography

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1. **J.M. Coulson and J.F. Richardson**, Chemical Engineering, II Vol., 2<sup>a</sup> Ed., 1965, Pergamon Press, London
2. **J. P. K. Seville, U. Tüzün and R. Clift**, Processing particulate solids, 1<sup>a</sup> Ed., 1997, Blackie Academic & Professional, London, UK, ISBN: 0751403768
3. **Philip A Schweitzer**, Handbook of Separation Techniques for Chemical Engineers, 3<sup>a</sup> Ed, 1996, McGraw-Hill, New York, NY, ISBN: 0070570612
4. **Albert Rushton, Anthony S. Ward, Richard G. Holdich**, Solid-Liquid Filtration and Separation Technology (Hardcover), 2<sup>a</sup> Ed, 2000, Wiley-VCH, Germany

# Chapter 2. Size reduction of solids

*2.1 Introductory concepts*

*2.2 Mechanisms of size reduction*

*2.3 Energy for size reduction*

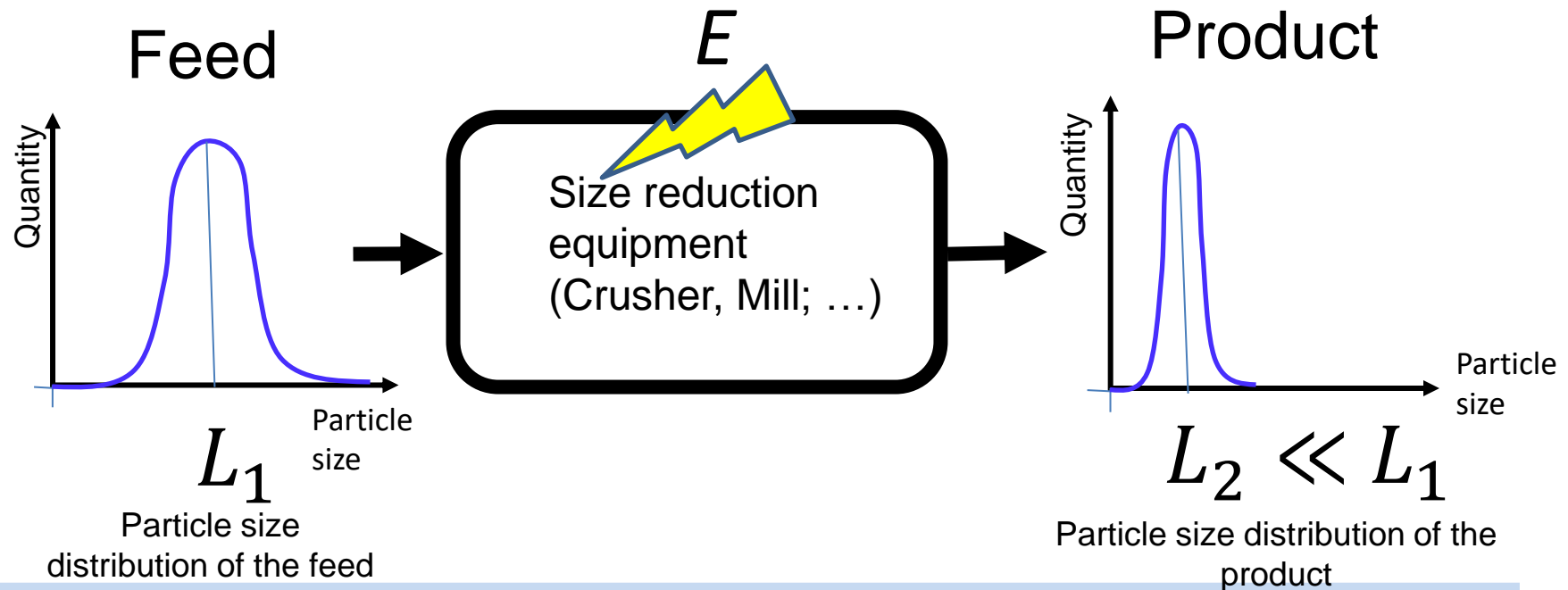
*2.4 Equipment for size reduction*

*2.5 Exercises.*

*J.M. Coulson and J.F. Richardson pp 95 - 144*

# Size reduction unit operation

A size reduction unit operation (a crusher or a mill) spends energy ( $E$ ) to reduce the size of solids from the average size  $L_1$  to the final average size  $L_2 \ll L_1$



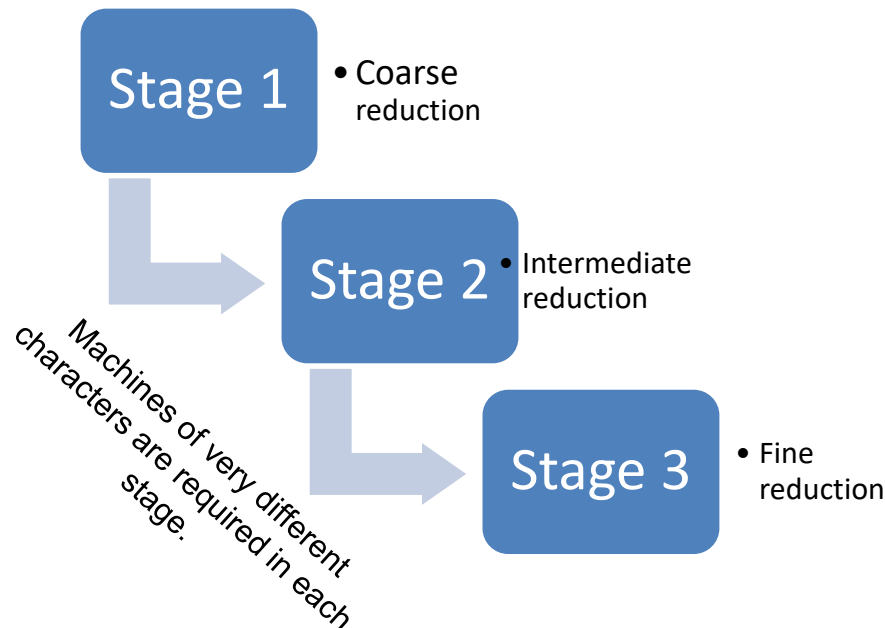
# Multi-stage size reduction

In chemical and biological engineering, it is often necessary either to decrease or to increase the particle size. For example, the starting material is too coarse, and possibly in the form of large rocks, and the final product needs to be a fine powder. The particle size will have to be progressively reduced in stages.

## Feed: Large rocks



### Sequence of size reduction stages



## Product: Fine powder





# Solids properties that affect size reduction

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Solid properties that highly impact the performance of a size reduction process and that influences the choice of the equipment:

- **Size of the feed-** depending on the size of the feed and product, reduction is classified as coarse, intermediate and fine reduction; the energy needed is very different
- **Compressive strength** - minimum compressive (slow) strength that causes solid fracture
- **Brittleness (“fragilidade”):** Brittle materials are characterized by little deformation, poor capacity to resist impact and vibration of load, [high compressive strength](#), and low [tensile strength](#). Most of inorganic non-metallic materials are brittle materials, e.g. glass
- **Stickiness (“pegajosidade”):** Stickiness is a property that causes considerable difficulty in reducing the size because the material gets to adhere to the equipment
- **Soapiness (propriedade de sabão):** a measure of the friction coefficient of the material surface
- **Humidity:** humidity content between 5-50% should be avoided as solids tend to cake, do not flow well; energy increases
- **Friability (“Friabilidade”):** it is the tendency of the material to fracture during normal handling; a crystalline material will break along well defined planes; the energy to break the material will increase as the material gets smaller.

# Types of forces in size reduction equipment

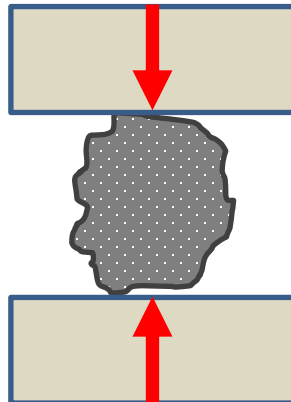
Four main types of forces develop simultaneously in any size reduction equipment. Different machines will however develop a predominant type of force.

## 1- Impact



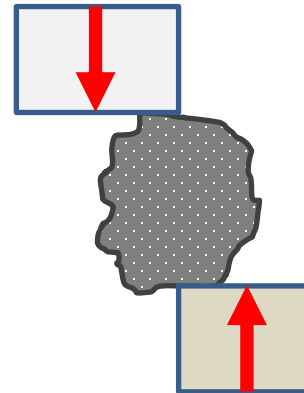
particle  
concussion by a  
single rigid force

## 2 - Compression



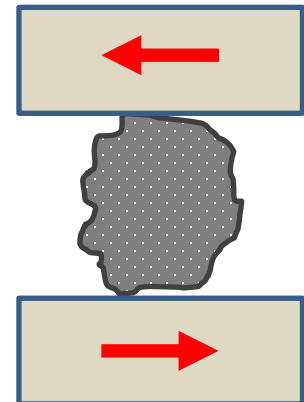
particle  
disintegration by  
two rigid forces

## 3 - Shear



produced by a  
fluid or by  
particle-particle  
interaction

## 4 - Attrition



arising from  
particles scraping  
against one  
another or against  
a rigid surface

# Compressive strength, $f_c$ [N/m<sup>2</sup>]

**Compressive strength (resistência ao esmagamento) ,  $f_c$  [N/m<sup>2</sup>]** is the compressive force required to cause rupture of the particle

Rock	Tensile Strength (MPa)	Compressive Strength (MPa)
Limestone	18.00 ± 0.62 (20)	41.45 ± 3.52 (4)
Sandstone	19.17 ± 0.21 (23)	77.59 ± 1.59 (5)
Sandstone	23.10 ± 0.48 (19)	80.83 ± 2.21 (10)
Sandstone	24.21 ± 0.83 (8)	90.48 ± 3.86 (4)
Mudstone	35.17 ± 3.17 (4)	50.07 ± 3.79 (4)
Limestone	36.28 ± 1.24 (24)	142.55 ± 6.14 (5)
Limestone	38.76 ± 2.69 (23)	142.97 ± 19.10 (8)
Ironstone	44.28 ± 4.48 (5)	190.69 ± 17.93 (4)
Sandstone	65.66 ± 0.83 (11)	167.66 ± 9.86 (5)

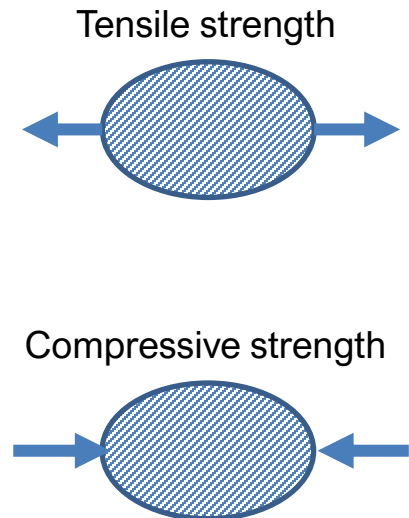
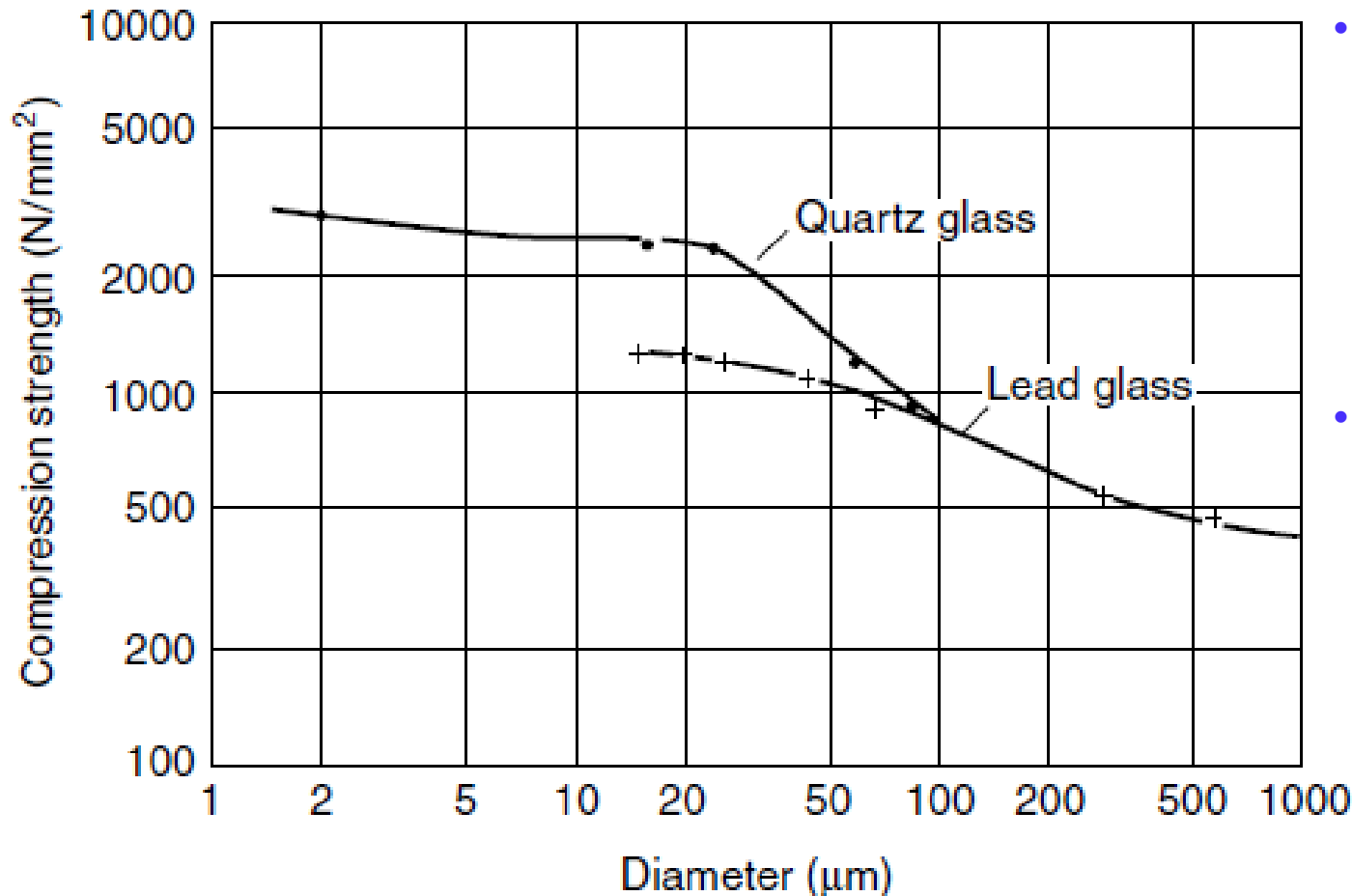


Table 2. Mean tensile strength and compressive strength for selected sedimentary rock types (after Johnson and Degraff, 1988).

# Compressive strength, $f_c$ [N/m<sup>2</sup>]

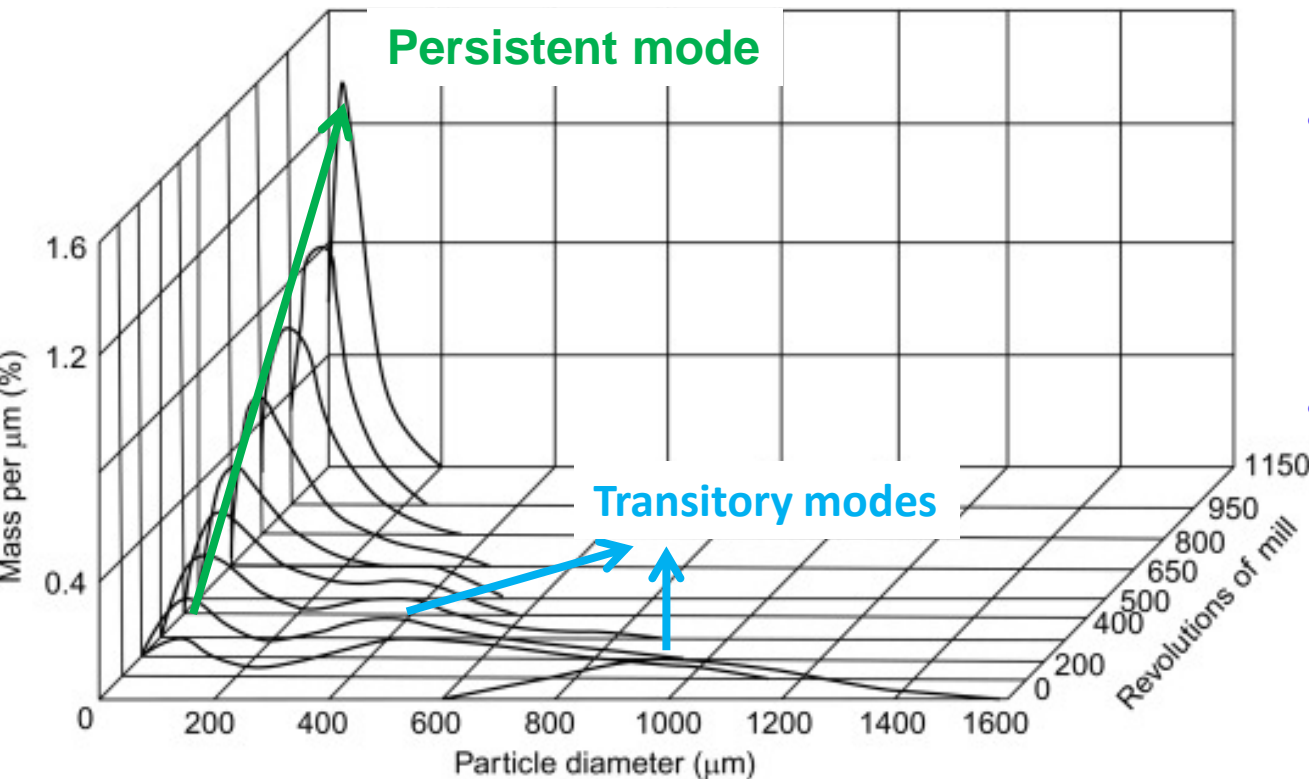
(taken from Coulson & Richardon)



- The compressive strength,  $f_c$  [N/m<sup>2</sup>], of a solid material tends to increase as the size of the solid decreases
- As consequence, the energy per unit mass [J/Kg] spent to reduce the size of a given material is higher in the small size range

# Transition and persistent modes

Effect of progressive gridding on the solid size distribution. The size distribution dynamically changes with mill revolutions



- **Persistent mode** (stable final size) is determined by the internal structure of the material
- The relative quantities **persistent** and **transitory modes** is determined by the size reduction machine (predominant type of force and power)

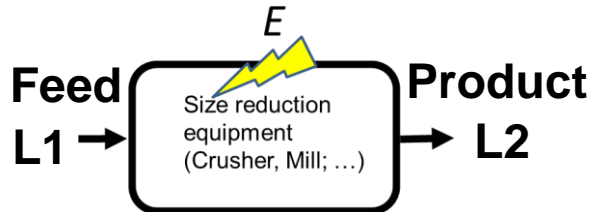
(taken from Coulson & Richardson)

# Energy for size reduction

Very low energy utilization efficiency between 0,1 – 2,0%

- **In producing inelastic deformation which results in effective size reduction  $L1 \rightarrow L2$  and creation of new surface.**
- In producing elastic deformation of the particles before fracture occurs
- In causing elastic deformation of the equipment.
- In friction between particles, and between particles and the machine.
- In noise, heat and vibration in the plant, and
- In friction losses in the plant itself.

# Energy for size reduction



Depending on the size of the feed and of the desired product, reduction equipment is classified as:  
(1) Fine, (2) Intermediate, (3) Coarse; different laws of energy are applied in each case

	<b>FINE</b>	<b>INTERMEDIATE</b>	<b>COARSE</b>
<b>Feed size (L1)</b>	5-2 mm	50-5 mm	1500-40 mm
<b>Product size (L2)</b>	<0,1 mm ( <i>powder</i> )	5-0,1 ( <i>granular/powder</i> )	50-5 mm ( <i>large/granular</i> )
<b>Examples of equipment</b>	<b>Ball mill</b> Buhrstone mill Roller mill NEI pendulum mill Griffin mill Ring roller mill Tube mill ...	<b>Crushing rolls</b> Disc crusher Edge runner mill Hammer mill Single roll crusher Pin mill Symons disc crusher ...	<b>Blake jaw crusher</b> Stag jaw crusher Dodge jaw crusher Gyratory crusher ...



# Energy laws

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*Empirical law of energy for size reduction:*

$$\frac{dE}{dL} = -C L^p$$

***E*** – energy spent for size reduction [kJ/kg]

***L*** – size of solids [m]

***C*** – empirical constant related to the solid properties and equipment properties

***p*** - empirical constant related to the size of solids

$p = -1$ : coarse reduction,  $p = -3/2$ : intermediate reduction,  
 $p = -2$ : fine reduction

# Energy laws

## Rittinger's law ( $p=-2$ )

$$E = C \left( \frac{1}{L_2} - \frac{1}{L_1} \right)$$

$$E = K_R f_c \left( \frac{1}{L_2} - \frac{1}{L_1} \right)$$

### Fine reduction

- Energy is utilized more efficiently
- Energy increases as the feed size decreases

## Bond's law ( $p = -3/2$ )

$$E = 2C \left( \frac{1}{\sqrt{L_2}} - \frac{1}{\sqrt{L_1}} \right)$$

$$E = E_i \sqrt{\left( \frac{100}{L_2} \right)} \left( 1 - \frac{1}{\sqrt{q}} \right)$$

### Intermediate reduction

- Intermediate efficiency
- Energy increases as the feed size decreases

## Kick's law ( $p = -1$ )

$$E = C \ln \left( \frac{L_1}{L_2} \right)$$

$$E = K_K f_c \ln \left( \frac{L_1}{L_2} \right)$$

### Coarse reduction

- Energy is utilized less efficiently
- Energy increases with the ratio of feed/product sizes

# Energy laws

## Rittinger's law

$$E = K_R f_c \left( \frac{1}{L_2} - \frac{1}{L_1} \right)$$

Fine reduction

## Bond's law

$$E = E_i \sqrt{\left( \frac{100}{L_2} \right) \left( 1 - \frac{1}{\sqrt{q}} \right)}$$

Intermediate reduction

## Kick's law

$$E = K_K f_c \ln \left( \frac{L_1}{L_2} \right)$$

Coarse reduction

**$E$**  – energy spent for size reduction, [KJ/kg]

**$K_R, K_K$** – Rittinger, Kick constant respectively; empirical constant related to the equipment; without physical meaning

**$f_c$** – Compressive strength [MPa]; characterizes the solid material that is being reduced

For bond's law only:

**$E_i$**  - the work index: amount of energy required to reduce unit mass of material from  **$L_1 = \infty$**  to a size  **$L_2 = 100 \mu\text{m}$**

$$q = L_1 / L_2$$

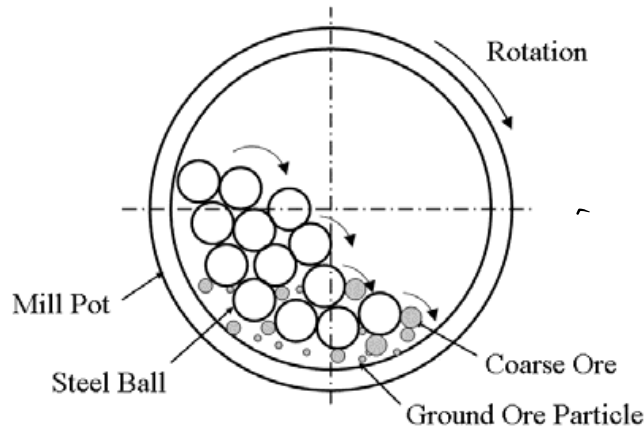
# Energy laws

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Determination of average size (L1, L2)

- **L1** or **L2** are calculated as the **mean diameter based on volume** ( $d_V$  - see chapter 1) from the feed and product size distributions
- Alternatively, **Bond's diameter** ( $d_{Bond}$ ) provides a good estimate. **Bond's diameter** ( $d_{Bond}$ ) is defined as the mesh size through which 80% of material passes, in a sieving characterization experiment

# Ball mill (Moinho de bolas)



- Hollow rotating cylindrical chamber
- Grinding balls inside [0,3-0,4 v/v]
- The balls are typically made of steel
- Prevalent forces: **Impact/Attrition**
- Classified as **fine size reduction**
- Rittinger's energy law

$$E = K_R f_c \left( \frac{1}{L_2} - \frac{1}{L_1} \right)$$

# Ball mill (Moinho de bolas)

- A **ball mill** has a critical rotation speed ( $w_c, rad/s$ ) that must be avoided. At the critical point, the ball (with mass  $m$ ) is subject to a centrifugal force ( $mu^2/r$ ) equal to the gravitational force ( $mg$ )



$$m \frac{u^2}{r} = mg \Leftrightarrow m r w_c^2 = mg \Leftrightarrow$$

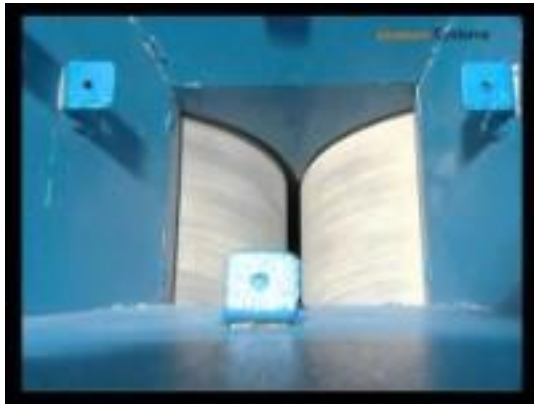
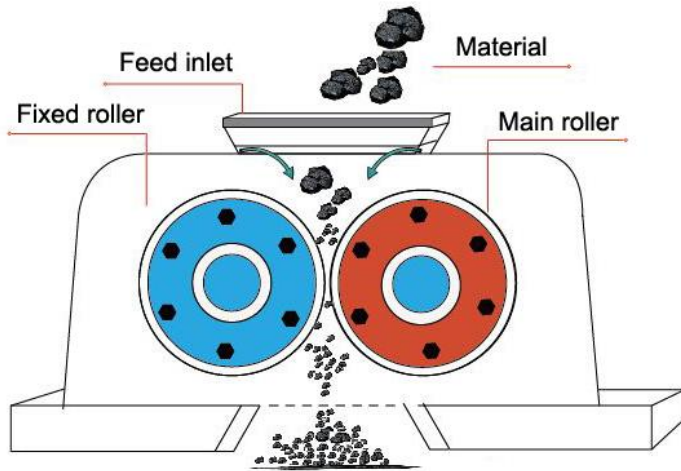
$$w_c = \sqrt{\frac{g}{r}}$$

- The optimal rotation speed ( $w_o, rad/s$ ) should be chosen below the critical value ( $w_c, rad/s$ ) in order to maximize milling efficiency:

$$w_o \sim [1/2, 3/4] \times w_c$$

$w_c$  - critical rotation speed [rad/s],  $r$  – mill internal radius [m],  $g = 9,81$  [m/s<sup>2</sup>]

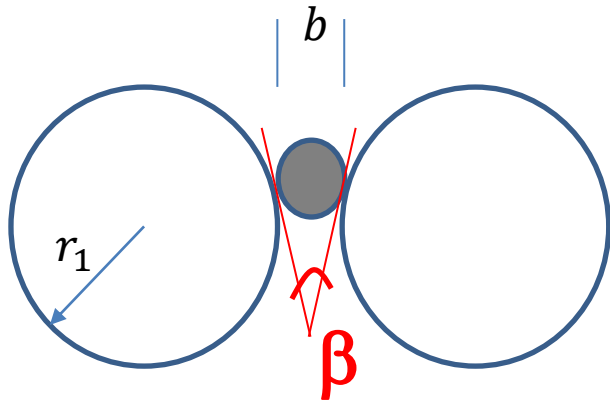
# Crushing rolls (triturador de rolos)



- Two parallel rollers (compact cylinders) rotating in opposite direction (inwards)
- In its simple version, only one of the rollers is mechanically driven
- The distance between the rollers may be adjusted to the feed size and desired product size
- Main forces: **compressive/attrition**
- Classified as **intermediate size reduction**
- Bond's energy law:

$$E = E_i \sqrt{\left(\frac{100}{L_2}\right)} \left(1 - \frac{1}{\sqrt{q}}\right)$$

# Crushing rolls (triturator de rolos)

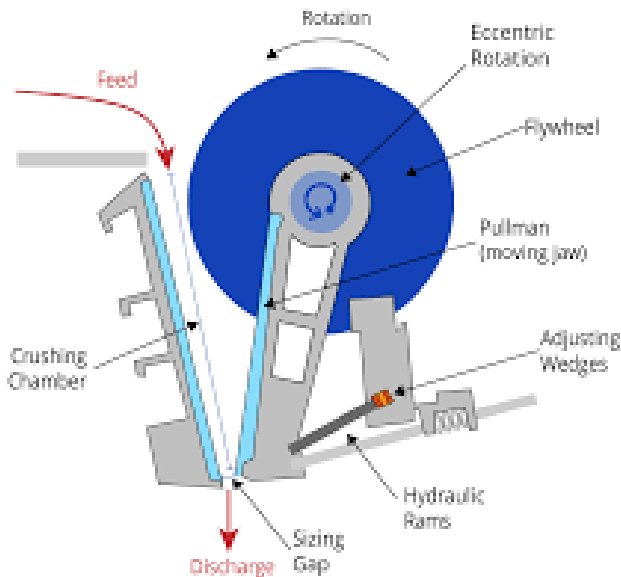


$$\cos\left(\frac{\beta}{2}\right) = \frac{r_1 + b/2}{r_1 + r_2}$$

- Roll radius,  $r_1$
- The distance between the rolls,  $b$ , must be adjusted to the feed particle size,  $r_2$
- **Nip angle,  $\beta$** , should be at most  $31^\circ$  degrees



# Jaw crusher (Triturador de maxilas)



- Two jaws with compatible toothed surface
- Fixed jaw + swing jaw
- Angle of nip  $\sim 30^\circ$  degree
- Main force applied: **compressive**
- Classified as **coarse reduction**
- Kick's energy law:

$$E = K_K f_c \ln \left( \frac{L_1}{L_2} \right)$$

# Exercises II.1–7

## II - REDUÇÃO DA GRANULOMETRIA DE SÓLIDOS

1. Tritura-se um material num triturador de maxilas Blake e reduz-se o tamanho médio das partículas de 50 mm para 10 mm, com um consumo de energia de  $13.0 \text{ kw s kg}^{-1}$ . Qual será o consumo de energia necessário para tritura o mesmo material do tamanho médio 75 mm até à dimensão média de 25 mm.

- (a) supondo aplicável a lei de Rittinger, e  
(b) supondo aplicável a lei de Kick?

Qual destes resultados considera de maior confiança e porquê?

2. Usou-se um triturador para tritura um material cuja resistência à compressão era de  $22.5 \text{ MN/m}^2$ . O tamanho da alimentação era menor que 50 mm, maior que 40 mm e a potência necessária era  $13.0 \text{ kw s kg}^{-1}$ . A análise por peneiração do produto produziu o seguinte resultado:

Dimensão da abertura (mm)	Percentagem do produto (% em número)
Passando por 6.00	100
Retido em 4.00	26
Retido em 2.00	18
Retido em 0.75	23
Retido em 0.50	8
Retido em 0.25	17
Retido em 0.125	3
Passando por 0.125	5

Qual seria a potência necessária para tritura um kg por segundo de um material com resistência à compressão de  $45 \text{ MN/m}^2$  a partir de uma alimentação de menor que 45 mm, maior que 40 mm para dar um produto de tamanho médio de 0.50 mm?

3. Um triturador para moer cal de  $70 \text{ MN/m}^2$  de resistência à compressão desde o tamanho médio de 6 mm de diâmetro até ao tamanho médio de 0.1 mm de diâmetro, precisa de ter 9 kw. A mesma máquina usa-se para tritura domolite ao mesmo ritmo de produção desde o tamanho médio de 6 mm de diâmetro até um produto que contém 20% com um diâmetro médio de 0.25 mm, 60% com um diâmetro médio de 0.125 mm, tendo o restante um diâmetro médio 0.085 mm. Fazer a estimativa da potência em kw necessária para accionar o

triturador, supondo que a resistência ao esmagamento da domolite é  $100 \text{ MN/m}^2$  e que a trituração obedece à lei de Rittinger.

4. Se se regularem uns rolos de moagem de 1 m de diâmetro de tal modo que as superfícies de moagem fiquem à distância de 12.5 mm e o ângulo de presa for  $31^\circ$ , qual é o tamanho máximo de partículas que se deveria introduzir nos rolos?

Se a capacidade real da máquina é 12% da teórica, calcular o ritmo de produção em kg por segundo, quando a funcionar a 2.0 Hz, se a superfície de trabalho dos rolos tiver 0.4 m de comprimento e se a alimentação pesar  $2500 \text{ kg/m}^2$ .

5. Um triturador mói sal desde um tamanho médio de partícula de 45 mm até um produto assim

Dimensão (mm)	% do produto em número
12.5	0.5
7.5	7.5
5.0	45.0
2.5	19.0
1.5	16.0
0.75	8.0
0.40	3.0
0.20	1.0

e ao fazer isto consome  $21 \text{ kJ/kg}$  de material triturado.

Calcular a potência necessária para tritura o mesmo material ao mesmo caudal, a partir de uma alimentação com um tamanho médio de 25 mm até um produto com um tamanho médio de 1 mm.

6. Um moinho de bolas com 1.2 m de diâmetro está a trabalhar a 0.80 Hz verificando-se que o moinho não está a trabalhar satisfatoriamente. Sugere alguma modificação nas condições de funcionamento?

7. É preciso fornecer 3 kw a uma máquina para esta tritura material ao caudal de  $0.3 \text{ kg/s}$  desde cubos de 12.5 mm até um produto com os seguintes tamanhos (% em número):

80%	3.175 mm
10%	2.5 mm
10%	2.25 mm

Que potência em kw teria de fornecer-se a esta máquina para tritura  $0.3 \text{ kg/s}$  do mesmo material de cubos de 7.5 mm até cubos de 2.0 mm?