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SOLIDS HANDLING & COMPUTER CONTROLLED BATCH FILTRATION

1. SOLIDS HANDLING

1.1 INTRODUCTION

Solids, in general, are more difficult to treat than the liquids, vapors or gases. During the processes, the solids may be presented in various types: big angular pieces, continuous wide sheets or powders pulverized in a refined way. They may be hard and abrasive, resistant or gummy, soft or fragile, dusty, plastic or sticky. Whatever their form, it is necessary to find ways to manipulate the solids and improve their manipulation characteristics if possible.

Of all the shapes and sizes that may be found in solids, the most important from a chemical engineering standpoint is the small particle. An understanding of the characteristics of masses of particulate solids is necessary in designing processes and equipment for dealing with streams containing such solids.[1] Much research regarding handling and storage characteristics of bulk solids has been conducted over the years. Physical properties of granular solids play a significant role in their resulting storage and flow behaviour, and are therefore essential to design appropriate, efficient and economic bulk solids handling and storage equipment and structures. Transportation and storage of many goods can be problematic as it often becomes restricted by caking and bridging which occurs during transportation and storage. This issue probably results from a number of factors, including storage moisture, temperature, relative humidity, particle size, time, or temperature variations, to name a few.[2] Flow is defined as the relative movement of a bulk of particles among neighbouring particles, or along the wall surface of a container.[3] Flow characteristics are of vital significance in bulk material handling and processing, since they impact conveying, blending, and storage options.[2]

The handling, storage, and flow of particulate materials are important in industries associated with agricultural, food, chemical, ceramic, pharmaceutical, metallurgical, and other bulk solids and powder processing.

Solid particles, especially when dry and not sticky, have many properties similar to a fluid. They exert pressure on the walls of a container while flowing through a hole or coming down a hopper.

However, they are different in some ways.

- Solid particles stick as a result of pressure and cannot glide down until the force applied reaches a certain value.
- The granular solids resist distortion permanently when they are subject to moderate force contrary to what happens with most of the fluids. (When enough force is applied, the breaking takes place and one layer of particles glides down over another)

Depending on the flux properties, the solids split into two groups:

- **cohesive** (like humid clay these type of solids are characterized by the *difficulty to flow* through holes.)
- **no cohesive** (like grain, sand or plastic strands that can *flow easily* from containers or silos.) [1]

1.2 THEORY

Individual solid particles are characterized by their size, shape and density. Particles of homogeneous solids have the same density as the bulk material but those obtained by breaking up a composite solid have various densities. Size and shape on the other hand are easily defined for regular particles (like spheres, cubes) but for irregular particles (like sand grains) the terms ***size*** and ***shape*** are not so clear and must be arbitrarily defined.[1]

Particle Shape

The shape of an individual particle is expressed in terms of the sphericity, Φs, which for a spherical particle has the value of 1. For a non-spherical particle, the sphericity is defined by the relation;

$$\phi s = \frac{6V_{\mathrm{p}}}{DpSp} \qquad \text{Eq.1.1}$$

Where;
Dp= equivalent diameter or nominal diameter of the particle, m
Sp= surface area of one particle, m^2
Vp=volume of one particle, m^3

The equivalent diameter is the diameter of a sphere of equal volume. Dp is usually taken to be the nominal size based on screen analysis, the surface area is found from adsorption measurements or from the pressure drop in a bed of particles.

Usually Φs takes values between 0.6 and 0.8.[1]

Particle Size

In general, diameters may be specified for any equidimensional particle. As for non equidimensional particles (that are longer in one direction than the other), often second longest major dimension is used. For needlelike particles, Dp would refer to thickness of particles, not their length. [1]

Size Analysis

In a sample of uniform particles of diameter, Dp (m), total volume of the particles is m/ρ_p where m and ρ_p refer to the total mass (kg) and density of the particles (kg/m^3) respectively. Since the volume of one particle is, Vp (m^3); the number of particles in the sample, N is:

$$N = \frac{m}{\rho_p Vp} \qquad \text{Eq.1.2}$$

The total area, A (m^2), of the particles is:

$$A = NSp = \frac{6m}{\phi_S \rho_p Dp} \qquad \text{Eq.1.3}$$

To apply Eq.1.2 and Eq.1.3 to mixtures of particles having various sizes and densities, the mixture is sorted into fractions, each of approximately constant size. Each fraction is weighed, the equations are applied. The mass or number fraction in each size increment as a function of the average particle size (or size range) in the increment. An analysis tabulated in this way is called a **differential analysis**. The results are often presented as a histogram. A second way is through a **cumulative analysis** obtained by adding, consecutively, the individual increments. [1]
There are a number of methods for measuring particle sizes and size distributions. Some of these methods depend upon calibration with known particle sizes. A number of suppliers now sell small spherical particles of nearly uniform size distributions for calibration purposes.[4]
Some of the more advanced methods of particle size measurement not only measure the particle sizes but they will also provide the size distributions of the particles. For a given material, there are four types of particle size distributions that are possible: (1) by number, (2) by length, (3) by surface, and (4) by mass (or volume). Distributions can be reported either in terms of frequency (differential form) or by cumulative (integral form). [4]

To explain how we mathematically represent the distribution data, lets suppose that you measure the mass of particles by size by some unspecificed process. As an example your measured data may be plotted as shown in Figure 1.1. You can normalize the plot by dividing the masses of each size by the total mass, to obtain the mass fractions as shown in Figure 1.2.

Finally, if we add the mass fractions cumulatively we get the Cumulative Mass Fraction plot, shown in Figure 1.3.[4]

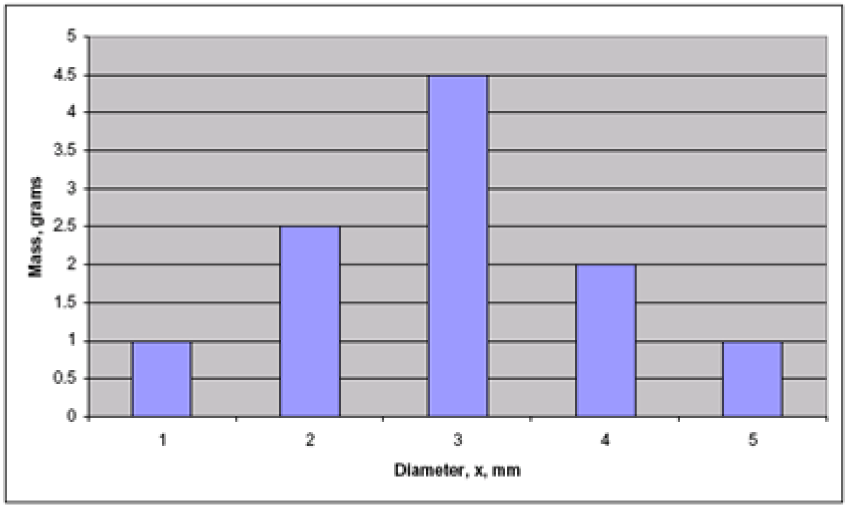


Figure 1.1. Example mass quantities of a imaginary sample of particles[4]

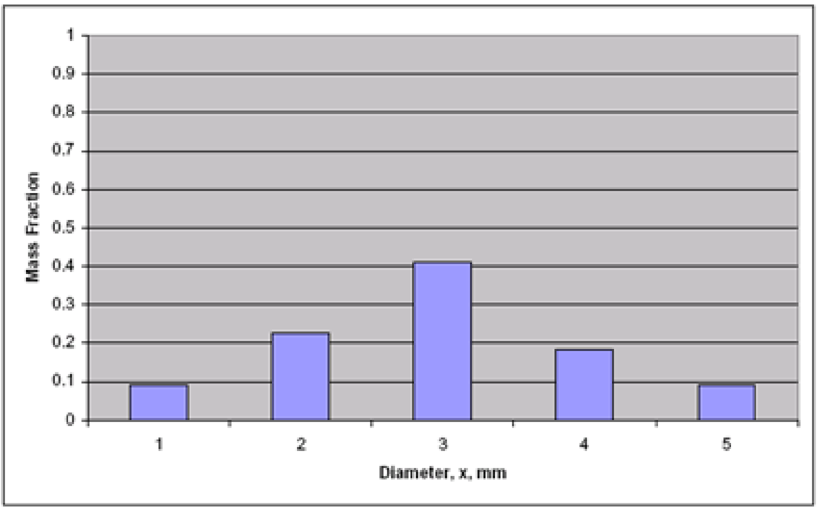


Figure 1.2. Mass fractions from data in Figure 1. 1[4]

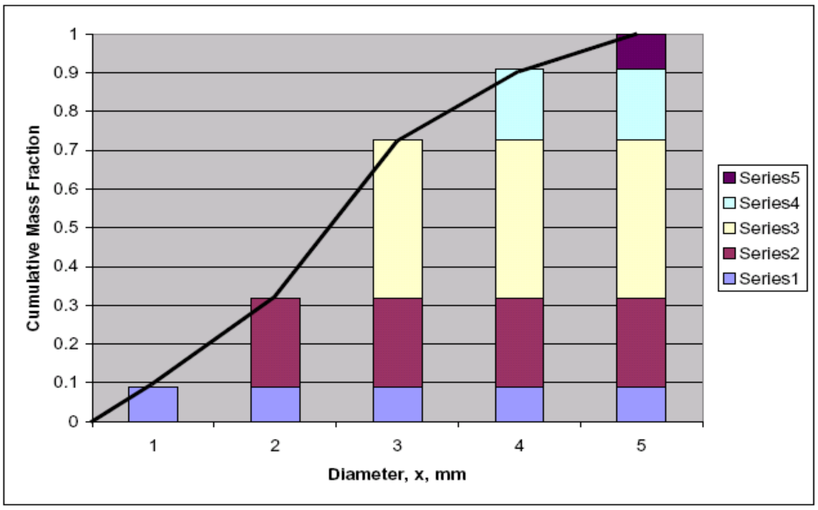


Figure 1.3. Cumulative mass fraction plot of data from Figure 1.1 [4]

Specific Surface of Mixture

If the particle density, ρ_P and sphericity, Φs are known and constant, the surface area of the particles in each fraction may be calculated. The results for all fractions added, give A_w, the specific surface (total surface area of a unit mass of particles, m^2/kg). It is calculated by Eq. 1.14 given below:

$$A_w = \frac{6x_1}{\phi_S \rho_P \mathrm{Dp}_1} + \frac{6x_2}{\phi_S \rho_P \mathrm{Dp}_2} + \ldots + \frac{6x_n}{\phi_S \rho_P \mathrm{Dp}_n} \qquad \text{Eq.1.14}$$

where;

Dpi = average particle diameter (arithmetic average of smallest and largest particle diameters in increment), m

n = number of increments

x_i= mass fraction in a given increment[1]

Average Particle Size

The most common way to describe average particle size is the v**olume-surface mean diameter,** Ds (m), which is related to the specific surface area, A_w (m^2/kg). It is defined as:

$$Ds = \frac{6}{\phi_S \mathrm{Aw} \rho_P} \qquad \text{Eq.1.15}^1$$

Miling and Sieving

Milling is a unit operation that is very important in some industrial processes, despite implying only a physical transformation. By milling, the average volume of the particles in a solid sample is reduced. The most usual methods used on the mill devices are: compression, impact, rubbing of metal shears and cut.

The main types of devices for the mill are:

A. **Crushers** (thick and fine)
  - Jaw crusher
  - Revolving crusher
  - Clod crusher

B. **Mills** (Intermediate fine)
  - Hammer mill
  - Clod crusher
  - Bowl mill
  - Clod crusher
  - Disk mills
  - Revolving mills
  - Bar mills
  - Ball mills
  - Tube mills

C. **Extremely fine mills**
  - Hammer mills
  - Energy flow mills
  - Shaking mills

D. **Cutting mills and mills with razors**

The milling operation takes place in two steps. The first step is to provide that the solids break into pieces. **Crushers** (most common type is **jaw crusher**) and then **primary mills** (usually **hammer mill** used in cement industry) are used.

In the jaw crushers, feed comes through the jaws that are in the shape of a "V". One of the jaws is steady, and the other has an alternative movement (eccentric). As a result, great compression force is applied on the pieces. The opening can be regulated and thus different grain sizes can be obtained from this crusher.

When relatively more controlled reducing is aimed, the type most commoly used in the industry is the **balls mill**. The balls mill carry out the most part of the reduction by impact. When it revolves round an axis, it provokes that the balls fall down in cascade from the maximum height of the mill. This action causes a tapping on the material. Thus, the mill is uniform. At industrial scale, the balls mill works with a continuous flow, having two rooms inside; the first one has big balls of two or three inches of diameter, whereas the second one will have balls of 1 to 1 1/2 inches (in SI units 0.0254 to 0.0127 m).[1]

Sieving

Flowability is the ability of granular solids and powders to flow. Particle size, and particle size distribution, both play significant roles in flowability and other properties, such as **bulk density, angle of repose, and compressibility** of bulk solids. Even a small change in particle size can cause significant alterations in the resulting flowability.[2] Reduction in particle size often tends to decrease the flowability of a given granular material due to the increased surface area per unit mass.[5] Particle size also plays an important role in the compressibility of powders. An increase in particle size generally leads to an increase in compressibility (and thus volume reduction).[6] By Farely and Valentin, 1967, particle size is found to be the most important factor governing the ***'structure'*** of the powder compact, and at the same time, the interparticulate force governs the ***strength of the 'structure'***.[7] The size distribution of particulate matter is defined as the relative percentage by weight of grains of each of the different size fractions represented in the sample.

Size distribution is very important in determining physicochemical properties in a large number of processes of various industries (e.g. production of food powders, chemicals, colorants, paints, and pharmaceuticals).[8]

Sieving or screening has been the oldest yet most important unit operation for industrial separation of solid particles or as a laboratory method in size analysis. Often the term **screening** is used to refer to a continuous sizing operation as distinct from sieving, which usually means a batch process.[9]

The sieves consist of framework and **mesh** which is made of wires. Their openings may be square or round. Each sieve is identified by **the size of the holes called *hole meshes***. **Mesh** is the number of openings per linear inch counting from the center of any wire to a point exactly 25.4mm (1 in) distant or by an opening specified in inches or milimeters, which is understood to be the clear opening or space between the wires. **Aperture**, or screen-size opening is the minimum clear space between the edges of the opening in the screening surface and is usually given in inches or milimeters.

It is essential that standart sieves (normalized sieves) with standart size-openings be used for sieve analyses. Standart screens of U.S. Sieve Series and Tyler Standart Sieve Series are used with almost equal frequency throughout U.S. industry. Standart screens range in mesh size from 4 in. to 400-mesh. Woven metal screens with openings as small as 1 μm are commercially available. Seperation in the size range between 4-mesh and 48-mesh is called “fine screening” and for sizes smaller than 48-mesh, it is considered as “ultrafine”.[8]

With regard to the sieving process, either for industrial separation of solid particles or as a laboratory method in size analysis, a **stack of sieves** or screens ***of decreasing mesh size, also known as a sifter cascade***, is often used.[10] The solid phase is placed on top of a series of screens. Each screen has smaller openings than the one above. Under the bottom screen, a solid pan is put.[8] The sieve stack is typically mounted on a device that provides vibration or shaking to achieve the movement of particles in relation to the sieve surface. At present, the electromagnetic sieves are the ones that are used exclusively. They transmit some vibrations in a vertical direction which are produced by an electromagnet. That will produce the solids to be sieved by a rotational movement which overcomes the usual blockage of the meshes.[9]

As the sieves are shaken, the particles fall through them until a screen is reached in which the openings are too small for the particle to pass. The size of particles found on any screen is expressed as an appropriate mean length between the openings in the screen above and that on which the particles rest.[11] The fraction of the particles that go through the sieve are called ***dust fines***, and the retained fraction is called ***coarse grits***.[8]

An example of sieving process is the case with flour which is a blend of particles. Flours of different particle sizes differ in physical properties and chemical composition. A method to separate flours according to particle size is through sieving. The fractioned flours are characterized by not only the *difference in chemical composition and physical properties* but also **minimal starch damage**.[12] However, fractionating flour by sieving, although relatively simple, is limited by sieve blinding. **Sieve blinding** occurs when particles block up and lodge in the sieving mesh.[13] It reduces the effective transfer area on the surface, resulting in reduction of sieving rates (**sieving performance or capacity**) and the degree of sharpness of particle separation (**sieving efficiency**).

Many factors have been identified to affect this unit operation, including;

- the size and shape of particles relative to the aperture of the sieve
- the mesh size of the sieve itself
- the amount of material on the sieve surface
- the direction of movement of the sieve
- the rate of movement of the material relative to the sieve surface, etc.[14]

Angle of repose

Angle of repose is defined as the angle between the horizontal and the slope of a heap of granular material dropped from some designated elevation. Angle of repose corresponds qualitatively to the flow properties of that material, and is a direct indication of potential flowability. [4]

In other words, the angle of repose is the greatest angle to the horizontal made naturally by the inclined surface of a pile of solid when poured from a designated height. When we pile up a granular mean (think about a salt mound), the angle of repose will take values between θ_r and θ_m (see Figure 1.5). Under θ_r the salt pile always is stable, and over θ_m there will be avalanches on the surface spontaneously. When the angle that the pile forms is between both of them, a bistability becomes evident and there could be an avalanche on the surface or not.[15]

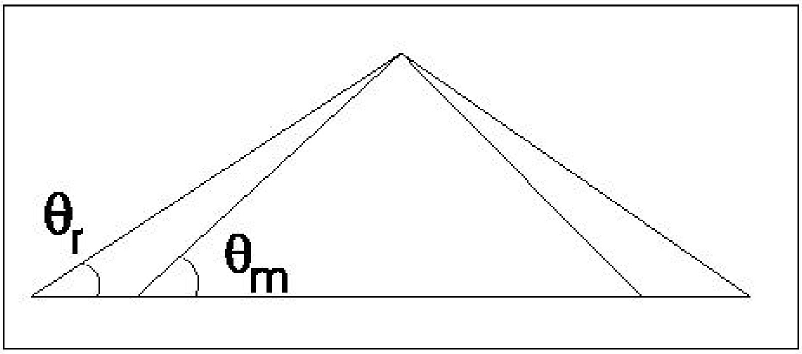


Figure 1.5. Angle of Repose[15]

Apparent density

For granular means, it is necessary to bear in mind the particles and the spaces among them. In general, even if the particles are subjected to the forces of gravity, they get into groups letting spaces among them. Therefore, a granular mean always takes up a volume higher than the one calculated by multiplying the number of particles and the volume of each one. The parameter that indicates the ratio between the volume corresponding to the particles mass, ***Vr*** **(real volume of the grains, m^3),** and its **apparent volume,** ***Va*** (volume including the spaces, m^3), is called ***crushing ratio*****:**

$$\phi = \frac{V_r}{V_a}$$

Eq.1.16

The crushing ratio is always less than one. [15]

An alternative to the crushing ratio is the **apparent density, ρ_a (kg/m^3),** obtained by dividing the granular medium mass, M (kg), by the occupied volume, Va (m^3) :

$$\rho_a = \frac{M}{V_a}$$

Eq.1.17

The apparent density is the product of the material density, ρ (kg/m^3), and the crushing ratio, ϕ :

$$\rho_a = \phi \cdot \rho$$

Eq.1.18

The apparent density will always be less than the real density.

The apparent density may change, depending on the crushing degree of the grains. The apparent density is minimum when the mass is "without cohesion" and reach a maximum when the mass is subjected to vibration or rolling.[15]

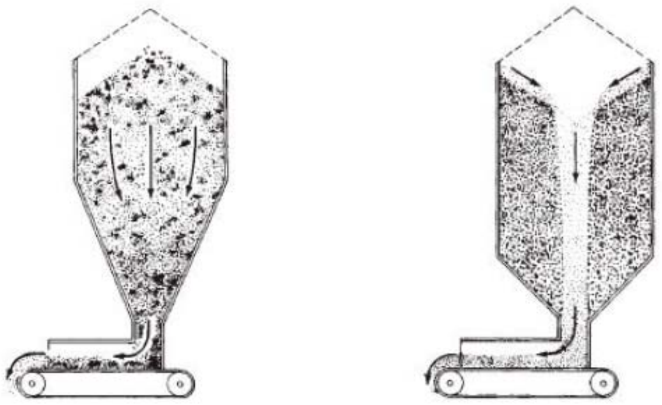
Deposits Unloading

The solids that are too valuable or too soluble to be exposed to elements are stored in deposits, hoppers or silos, which are cylindrical or rectangular recipients of concrete or metal. The silos are usually high or have a relatively small diameter, whereas the deposits are rather wide and not so high. A hopper is a small deposit with an oblique bottom that is used for temporary storage. All these containers are loaded by its top part, using some kind of elevator, whereas the unloading is usually done through the bottom. The main design problem of a deposit is to obtain a satisfactory unloading. [15]

The solids tend to go out by any orifice of a deposit, but they do it better through an orifice situated at the bottom. The pressure in a side exit is less than the vertical pressure for the same level. This causes the exit to get blocked up more easily. Besides, the solids collection by a side exit causes the side pressure to increase heavily. An exit at the bottom does not get blocked so easily and do not generate abnormally high pressures on any point of the walls.

Except for small deposits, it is not possible to open the whole bottom for the unloading. A conic or pyramidal bottom leads usually to a small circular exit, closed by a valve. The vertical pressure fluctuates as the material is unloaded, and on average, it is 5 to 10% higher than when the mass is stable.

Two flux models have been developed depending on the walls inclination in the bottom section of the deposit and on the friction coefficient between the solids and the deposit walls. In deposits of a conic bottom, with a high cone, a **mass flow** grows, where whole material drops uniformly from the top part of the deposit. In deposits with a short cone, or with vertical walls and a central hole on the bottom, a **funnel or internal flow** takes place. In this case, a vertical column of solids, situated on the opening, goes down without disrupting the side material. Eventually, a side flux starts flowing, first from the highest layer of the solids, forming a conic depression on the mass. The solids situated on the deposit bottom or near the walls go out the latest. If more material is added by the top part of the deposit at the same speed with which the unloading is done on the bottom, the solids near the deposit walls stay and do not go out. [15]



Masic flow.

Internal flow.

Figure 1.6. Unloading flow types, masic flow and internal flow[15]

There is a kind of flow called ***"mixed",*** in which a masic flow takes place for the particles that are over a supplied level (around 1 – 2 times bigger than the silo diameter) and internal flux occurs for the particles that are under that level.

The speed of grain solids by gravity through the circle hole on the bottom of a deposit depends on the **diameter of the hole and the solids properties.**

It does not depend on the highness of the solids layer. [15]

There are some models that describe the solids unloading, ***Johansson and Berverloo equations.***

Johansson's expression is theoretical and it is only suitable for mass flow.

Berverloo's one is empiric and it is only suitable for funnel flow.

- **Johansson's equation: (for mass flow)**

There are parameters of Johansson's equation that depend on the kind of hopper used.

For a conic hopper with circular outflow, it comes up under the form:

$$m\& = \rho_a \frac{\pi D^2}{4} \sqrt{\frac{gD}{4.\tan(\theta)}} \qquad \text{Eq. 1.19}^{15}$$

where

$m\&$:Unloading mass flow (kg/s)

ρ_a :Apparent density (kg/m^3)

D :Exit orifice diameter (m)

g :Gravity acceleration (m/s^2)

θ :Hopper angle from the vertical.

Depending on the hoppers included in the unit, $\theta = 30°$ *or* $\theta = 0°$.

- **Berverloo's equation: (for funnel flow)**

$$\dot{m} = 0.58\rho_a g^{0.5}(D - k\mathrm{Dp})^{5/2}$$ **E**q. 1.20[15]

where

$\dot{m}$:Unloading mass flow (kg/s)

ρ_a :Apparent density (kg/m^3)

D :Exit orifice diameter (m)

g :Gravity acceleration (m/s^2)

Dp :Particle diameter (m)

k :Constant (1,3 < k < 2,9) with a typical value of 1,4

In general, it is difficult to start the flow with cohesive solids. However, once started, it is reestablished on the material situated directly on the unloading opening. The adherent solids, and even some dry powders, stick hard to the vertical surfaces and have enough force of metal shears to support a plug of a considerable diameter on the unloading opening. Therefore, to start the flow and keep the material moving; vibrators on the container walls, scraping blades near the container bottom or spurts of air on the unloading opening are often required.

The unloading opening must be small enough to be closed easily when the solids are flowing, but not too much to get blocked. It is preferable to make the opening wide enough to let the whole flow when it is half-open. In this case, it will be possible to open more to counteract the block. However, if the opening is too wide, the control of flow speed will be bad.[15]

Cyclone and pneumatic conveying

Cyclones:

For seperation of small solid particles or mist from gases, the most widely used type of equipment is the cyclone seperator. The cyclones are devices designed to separate the solid part of the flow in two-phase mixtures. If the flow phase is a liquid, they are called **hydrocyclons**, and if it is a gas, **aerocyclons**.[16] Based on air inlet geometry, cyclones are classified into two groups: ***Inverted and direct***. The inverted cyclones are the most common ones, and they consist of a cylindrical body with a conic base, a dust-collecting hopper, and air inlet and outlet. And the direct cyclones are tangential or axial.[15]

The feed mixture goes down rotating in the cyclone. Due to the centrifugal force, the solid phase is thrown to outside walls of the cyclone, goes down and is collected on the lower part, which frequently finishes in a cone. The flow phase, once at the bottom, rise rotating and it is collected through a pipe situated on the cyclone center. In such a center, strong vortex takes place and the low pressure impels the phase upwards. The centrifugal force developed in the vortex tend to move the particles radially to the wall, so that they will be collected as they will go downwards after reaching the wall. Therefore cyclones can be thought as sedimentation devices, in which a strong centrifugal force acts radially (and gravitational force relatively weak vertically). The powder particles that go in the cyclone are accelerated radially, but the force that acts on a particle is not constant due to the change with r and with the distance under the inlet. The calculation of the particles trajectory is difficult, so that the efficiency of a cyclone is predicted usually from empiric correlations.

The **efficiency of seperation** for a given particle diameter is defined as the mass fraction of the size particles that are collected. The cyclone output can be defined as the masic flow of separated solid particles, divided by the masic flow of incoming particles in the cyclone. The output of a cyclone depends on the size of the particles. In general, the small the particles are, the worse output we have, and the bigger the particles are, the better output we have. This is because smaller particles have smaller settling velocities and do not have to reach the wall to be collected. [16]

In general, the cyclone efficiency increases directly proportional to:

- Gas density
- Inlet speed
- Cyclone length
- Gas revolutions number
- Relation between the body and the outlet diameters
- Particles diameter
- Powder amount
- Cyclone wall bumpy texture

The efficiency decreases when there is an increase in the:

- Gas viscosity
- Gas temperature
- Cyclone diameter
- Gas oulet diameter
- Width of the gas inlet pipe
- Inlet surface

The cyclone is one of the few separation devices that operates better at full load than at partial load. [15]

Pneumatic conveying:

In a pneumatic conveyor, the suspension flow is a gas, usually air, which circulates among speeds between 15 to 30 m/s in pipes, with diameters between 50 to 400 mm.[15]

Solids Mixture

In practise the proof of a mixer is in the properties of the mixed material it produces. A well-mixed product is one that does what is required and has the necessary property – visual uniformity, high strength, uniform burning rate, etc. A good mixer is the one that produces well-mixed products at lowest overall cost. There are some types of mixers for dry powders. Many materials are mixed by tumbling them in a container partially full that revolves round a horizontal axis. The tumbling drums mix efficiently thick solids suspensions in liquids and heavy dry powders. As in the double cone mixer, it can contain internal atomizers to introduce small amounts of liquids in the mixer. The drum mixers use generally little less power than the ribbon blenders.[1]

1.3 EXPERIMENTAL PROCEDURE

In the solids handling unit provided in Chemical Engineering Lab. I; the following will be practised.

- Study of the size reduction
- Classification study according to the size
- Solids mixture study
- Solids pneumatic conveyor study
- Solids separation study through the cyclone use
- Study of the angle of repose in solids storage
- Study of the apparent density
- Solids unloading study in hoppers
- Solids weighting

1.3.1 Sieving

- Weigh the solid sample with scales, which has been previously tared. (The sample will have a maximum of 250 grams to assure the correct operation of the sieve).
- Check the set of normalized sieves put, see if they have correctly been fit vertically. (The sieve with the smallest hole mesh will be put at the bottom, over a rack without openings called ***bottom***).
- Keep the particles over the sieve, put the lid and fix the system with bolts. Subject the set to vibrations for a certain time.
- Remove each rejection and weigh. (The total amount of the rejections including the one taken from the bottom must be equal to the total mass of solids that is collected on the sieve, with an permissible error not over 2%).

1.3.2 Angle of repose

- Fill the rotatory cylinder until half of it approximately, using the pneumatic conveyor system, sucking up from a hopper and separating the particles through a cyclone.

- Turn the cylinder slowly, clockwise, until the very moment that the particles start sliding. Note the corresponding angle.
- Repeat by turning the cylinder counter-clockwise.
- Disconnect the tube and turn the cylinder until the contents are emptied into the tray, using the orifice.

1.3.3 Apparent Density

- Take a certain mass of dry solid, for example 500 grams. Pour into the test tube and determine the volume without compacting.
- Then, place the graduated tube in the sieve and subject it to a vibrations cycle for some minutes until the taken volume do not decrease anymore. Then note the volume.

1.3.4 Deposits Unloading

- Select one of the four available hoppers, each one with a different hole size. Fill up the hopper with a dry solid, approximately 2/3 of its total capacity, weighting previously this amount with the scales, and using the tray.
- Open the valve and the hopper starts being unloaded on the collecting tray. Note the unloading time.

1.3.5 Cyclone and pneumatic conveying

- Weight some amount of solid and place the sample on a hopper with discharge valve closed.
- Turn on the blowing and note air flow rate.
- As soon as there is a suction and conveying of the solid begins, start the chronometer.
- First convey the solids to another hopper and note the time and calculate mass flow rate.
- The solid conveyed by the pneumatic system will then be separated by the cyclone.

1.3.6 Ball Mill

- Weight with a balance an amount of solid about 250 grams. Sieve the sample in pieces of 250 grams.

- Take a mass of solid with a heterogeneous size and establish the mill homogeneity.
- Fill up the mill with the sample and 3,5 kg of balls. (The size and the balls proportion may be changed). Before turning on the mill, set the speed regulator to the minimum.
- Afterwards, increase the speed until the balls start falling freely. (The mill time can vary between 10-30 minutes).

1.3.7 Solids Mixture

- **Preparation:** Place the mixer so that the load openings are on the upper part and the discharge opening fully closed and sealed.
- **Load:** To load the mixer, take the lid off of the charge opening. Once charged, be sure that the lids are perfectly secured in the openings.
- **Mixing:** Once the mixer has been charged, the mix process should begin for
- some time.
- **Discharge:** Once the unit has been turned off, turn it down manually if necessary until the discharge opening would be down-facing. Open the discharge mechanism and the product will be collected in the tray.

DATA SHEET

Name Surname:
Group No:
Assistant: **Date:**

Sieving

Hole mesh (mm)	Dust fines (g)	Coarse grits (g)	Dust fines %	Accumulated Dust fines %	Accumulated Coarse grits %

Hole mesh (mm)	Dust fines (g)	Coarse grits (g)	Dust fines %	Accumulated Dust fines %	Accumulated Coarse grits %

Angle of repose

- Calculate the angle of repose.

Apparent Density

- Calculate the material density.
- Then calculate the crushing ratio.

Deposits Unloading

-Calculate the unloading masic flow, check afterwards Johanson and Beverloo equations. Determine the kind of flow.

- *You are not expected to comment on the effectiveness of the equations.*
- *Required data such as the apparent density or the particles size is obtained with the corresponding practices.*

1.4 NOMENCLATURE

ϕ : Crushing ratio

ρ_p : Density of the particles, kg/m^3

θ : Hopper angle from the vertical

Φs : Sphericity

N : The number of particles in the sample

A_w : The specific surface area, m^2

$\dot{m}$: Unloading mass flow

Sp : Surface area of one particle, m^2

Vp : Volume of one particle, m^3

Va : Volume of particles including the spaces between them, m^3

Dpi :Average particle diameter (arithmetic average of smallest and largest particle diameters in increment), m

N : Number of increments

x_i : Mass fraction in a given increment

Ds : The volume-surface mean diameter (m),

Vr : Real volume of the grains, (m^3),

Va : Apparent volume (volume including the spaces, m^3),

ρ_a : Apparent density *(kg/m^3)*,

D :Exit orifice diameter (m)

g :Gravity acceleration (m/s^2)

Dp :Particle diameter (m)

k :Constant ($1,3 < k < 2,9$) with a typical value of 1,4

1.5 REFERENCES

1. McCabe W. , Smith J.C. , Harriott P. , 1993, Unit Operations of Chemical Engineering, McGraw-Hill, 927-1065
2. Ganesan, V., Rosentrater, K.A.K., Muthukumarappan, K., 2008, Flowability and handling characteristics of bulk solids and powders – a review with implications for DDGS5, Biosystems engineering, 101, 425–435
3. Peleg, M., 1977, Flowability of food powders and methods for its valuation – a review, Journal of Food Process Engineering, 1, 303–328
4. Svarovsky, L., 1990, Solid-Liquid Seperation, 3rd Edition, Butterworth, Chapter 2
5. Fitzpatrick,J.J., Barringer,S.A.; Iqbal,T., 2004, Flow property measurement of food powders and sensitivity of Jenike's hopper design methodology to the measured values, Journal of Food Engineering, 61(3), 399–405
6. Yan H; Barbosa-Canovas G.V., 1997, Compression characteristics of agglomerated food powders: effect of agglomerate size and water activity, Food Science and Technology International,3(5), 351–359
7. Farely R; Valentin F.H.H., 1967/68, Effect of particle size upon strength of powders, Powder Technology, 1, 344–354
8. Perry, P.H., Green, D.W., 1999, Perry's Chemical Engineers' Handbook, McGraw-Hill, 1774-1779
9. Liu, K., 2009, Some factors affecting sieving performance and efficiency, Powder Technology, 193,208–213

10. T. Allen, 2003, Particle size analysis by sieving, Powder Sampling and Particle Size Determination, Elsevier, 208–250

11. Foust, A.S., Wenzel, L.A., Clump, C.W., Maus, L., Andersen, L.B., 1960, Principles of Unit Operations, John Wiley&Sons,Inc., 525-528

12. A.C. Apling, 1984, Blinding of screens by sub-sieve sized particles, Transactions of the Institution of Mining and Metallurgy, Section C, Mineral Processing and Extractive Metallurgy, 93, C92–C94

13. T.R. Roberts, J.K. Beddow, 1968, Some effects of particle shape and size upon blinding during sieving, Powder Technology,2, 121–124

14. K. Leschonski, 1979, Sieve analysis, the Cinderella of particle size analysis methods?, Powder Technology, 24,115–124

15. Practices Manual, 2006, EDIBON, S.A., 1-36

16. Geankoplis,C.J., 2003, Transport Processes and Seperation Process Principles, 4th Edition, Prentice Hall, 943-949

2. BATCH FILTRATION

2.1 INTRODUCTION

The filtration is a basic operation, widely used in the chemical industry, namely the separation of solid particles of a suspension through a filter medium that lets the fluid and retains the solid. The solid particles retained on the filter media will form a porous bed (called “filter cake”), through which the fluid flows. In general, the pores of the filter medium have a tortuous shape and they are usually larger than the particles to be separated. Then, the filter just works in a proper way only after an initial deposit has been held in its middle. As the filtering process happens, then the thickness of the cake increases so that the resistance to fluid flow is increased too, being able to carry out the operation in the following ways[1]:

- Filtration at a constant pressure: The flow decreases with time.
- Filtration at a constant flow: The pressure increases with time

In the filtering process, filters can be used widely. The most appropriate choice as well as the optimal operating conditions depend on many factors among which we can include the following[1]:

- The fluid properties, especially viscosity, density and corrosive properties.
- The nature of solid size and shape of particles, size distribution and characteristics of the fill.
- The concentration of suspended solids in the filter.
- The amount of material to be treated.
- If the product we want to extract is the solid, the fluid or both.
- Suspension flow to be treated.
- The need to wash the solid filtered.

Filtration is essentially a mechanical operation, which does not require a lot of energy. Usually progressive establishment of a cake on the filter medium, progressively increases the resistance to flow. In the initial instants of the operation particles are deposited on the surface layers of the support, forming the filter media[4]:

The most important factors which affect to the filtration rate are:

- The pressure drop from the feed to the outlet of the media.
- The filter surface area.
- The viscosity of the filtrate.
- The cake resistance.
- The strength of the filter medium and the initial layer of the cake.

This type of filtering is called **cake filtration**: the proportion of solids in the suspension is high and most of the particles are collected on the filter cake, which is subsequently separated from the medium[4].

The function of the filter medium is generally to act as support for the fitler cake cake while its initial layers provide the real filter. There are some cases in which the solids to be filtered are very small size and they form a dense and waterproof cake, quickly blocking any filter medium that is thin enough to retain them. The filtering of these materials requires that the porosity of the cake increases to allow the passage of fluid with a reasonable speed. This is done by adding a **filter aid** to the suspension before the filtration such as diatomaceous earth, perlite, purified wood cellulose or other porous inert materials. Then, the filter aid can be separated by dissolving the filter cake solids or by burning it directly. Another way to use the filter aid consists on depositing a layer on the fitler media before the operation.

The initial stages of cake formation are of great importance for the following reasons[3]:

- For any pressure filtration value, the flow rate is higher at the beginning of the process and then the resistance is minimal.
- High initial filtration rates may cause the clogging of the pores of the support, causing a very high resistance to the flow passage.
- The orientation of the particles in the initial layers can influence appreciably the structure of the filter cake.

The filter cakes can be divided into two types:

- **Compressible cake**: with this type of cake an increasing of the differential pressure or flow rate leads to the formation of a denser cake with a higher resistance.
- **Non-compressible cake**: the resistance to flow of a given volume of cake is not affected significantly by neither the differential pressure across the cake nor the deposition rate of the material.

2.2 FILTRATION THEORY

The theoretical principle of filtration is based on the quantification of the basic relationship of speed of a fluid

$$\text{speed} = F/R \qquad \text{Eq. 2.1}$$

Where :

- F: driving force force (gravity, thrust of a pump or centrifugal force) (N)
- R: resistance is the sum of that offered by the filter medium and the solid cake formed on it. (kg/s)

The fluid velocity is constrained by the fact that it has to pass through an irregular medium, through some channels formed in the interstices of the cake and filter medium. Then, you can apply the Law of Hagen-Poiseuille[2]:

$$K_2 = \frac{\mu . r}{\Delta P . A} \frac{dV}{A d\theta} = \frac{\Delta P}{\mu \left[\alpha \left(W / A \right) + r \right]} \qquad \text{Eq.2.2}$$

Where:

- V: filtered volume (m^3)
- θ: filtering time (s)
- A: Surface (m^2)
- P: total pressure fall (N/m^2)
- μ: filtrate viscosity (kg/ms)
- r: filtering medium resistance (1/m)
- W: cake weight (kg)
- α: specific cake resistance (m/kg)

If we consider the approximation that the cake is non-compressible and uniformly compacted, the mass of filter cake (W) is related to the volume of filtrate (V) using the following mass balance[5]:

$$W = wV = \frac{\rho . c}{1 - m.c} V \qquad \text{Eq.2.3}$$

Where:

-W: cake weight (kg)
- w: solid mass per filtered unit (kg/m^3)
- c: fraction of solids in the flow of supplies or concentration
- ρ: filtered density (kg/m^3)
- m: Difference of mass between the wet cake and the dry cake (kg)

The cake´s specific resistance constant(α) depends on the pressure with the following formula[5]:

$\alpha = \alpha\,' \cdot PS$

Where α' is another constant depending on the size of the particles which make up the cake.

2.3 EXPERIMENTAL STUDIES

Laboratory studies or “at small scale” can get a trial basis the time variation of the volume filtered (speed) and pressure, according to two types of flow:

- Constant pressure
- Constant Speed (flow)

• Constant pressure filtration testing

The practice of filtering can be done by controlling the pressure difference so that it remains constant throughout the process. It is clear that at a constant pressure the rate of filtration will decrease as the thickness of the cake increases and also its resistance to filtration. For the study of filtration in these circumstances we can start from the Hagen-Poiseuille law as described above, which can be written as follows[4]:

$$K_2 = \frac{\mu . r}{A} \qquad \text{Eq.2.4}$$

As we have a constant pressure process...

$$\frac{d\theta}{dV} = K_1 . V + K_2 \qquad \text{Eq.2.5}$$

Where:

$$K_1 = \frac{\mu . \alpha . \omega}{\Delta P . A^2} \qquad \text{Eq.2.6}$$

$$K_2 = \frac{\mu . r}{\Delta P . A} \qquad \text{Eq.2.7}$$

According to the above equation and ploting Δθ versus filtrate volume ΔV we obtain a line of slope K_1 and intercept K_2 as shown in the following illustration:

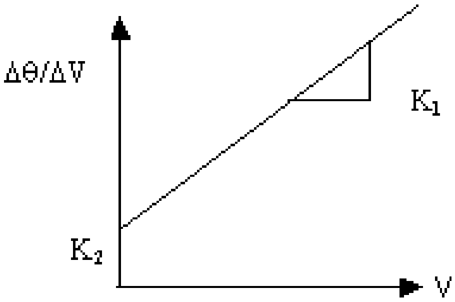


Figure 2.1. The graph of Δθ versus filtrate volume ΔV

After obtaining these constants, you can find the values of α and r.

In order to determine the effect of pressure change is necessary to perform multiple tests at different pressures and calculate the compressibility constant S. To do this, plot the logα versus logΔP, thus obtaining a line of slope S and intercept α '.

- The experimental determination of the parameters K_1 and K_2 and for these r, α, S and α' are necessary for the designing or selection of a filter for a specific system, for the election of the filter medium to provide the maximum solids retention and for the determination of operating conditions (temperature, pressure ...).

- **Constant rate filtration testing**

The practice of filtering can be done by controlling the speed so that it remains constant throughout the process. In this case, the pressure will increase as the filtering process happens due to increasing of the thickness of the cake and also the resistance to leakage. For the study of filtration in these circumstances we can start from the Hagen-Poiseuille law as described above, which can be written as follows[7]:

$$\frac{d\theta}{dV} = \frac{\mu.\alpha.\omega}{\Delta P.A^2}.V + \frac{\mu.r}{\Delta P.A} \qquad \text{Eq.2.4}$$

Solving the above expression:

$$\Delta P = \frac{\mu.\alpha.\omega}{A^2}.\frac{dV}{d\theta}V + \frac{\mu.r}{A}.\frac{dV}{d\theta} \qquad \text{Eq.2.8}$$

Where:

$$\frac{dV}{d\theta} = speed = q \qquad \text{Eq.2.9}$$

As this is a process at constant speed, the above expression is as follows[7]:

$$\Delta P = K_1.V + K_2 \qquad \text{Eq.2.10}$$

Where

$$K_1 = \frac{\mu.\alpha.\omega.q}{A^2} \qquad \text{Eq.2.11}$$

$$K_2 = \frac{\mu.r}{A}.q \qquad \text{Eq.2.12}$$

According to the above equation and plotting ΔP versus the volume of filtrate, we obtain a line of slope K_1 and intercept K_2 as shown in the following illustration:

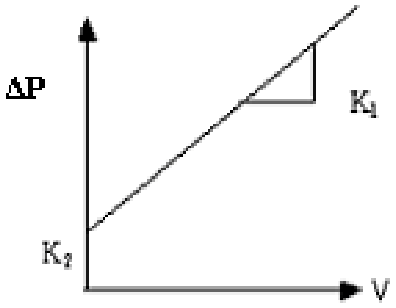


Figure 2.2 The graph of ΔP versus the volume of filtrate

After obtaining these constants, you can find the values of α and r.

EQUIPMENT USED IN FILTRATION

There are several types of industrial filters but the most common are[8]:

- **Press Filter**: they can work at a constant pressure or variable pressure. Sometimes, pressure may be increased gradually to keep constant the volume of filtrate. Centrifugal pumps are commonly used for these purposes.

Filtration is given initially at a constant rate followed by a constant pressure period. The pressure ranges used in this kind of units is 25-75psig (1,7- 5,1 bar). The plates of these units can be horizontal or vertical.

- **Vacuum filter:** In these filters, pressure is kept below atmospheric pressure under the filter medium and the same as atmospheric when being on it.

1. Rotatory drum vacuum filters: they consist of a rotating drum which rotates around its horizontal axis. The drum surface consists of a set of shallow compartments formed by dividing strips that run the drum lake. Each compartment is connected by one or more pipelines to a centrally located automatic rotatory valve at one end of the drum. The drum is partially submerged into an open tank containing the suspension to be filtered. The filter media covers the surface of the drum and it is supported by perforated plates. As the drum rotates, compartments submerged in the suspension make some vacuum.

   The filtrate flows through the filter medium and gets out through the drain pipe, while the solids form a cake on the outer surface of the media. As the compartment appears from the suspension, the cake layer is scraped off.

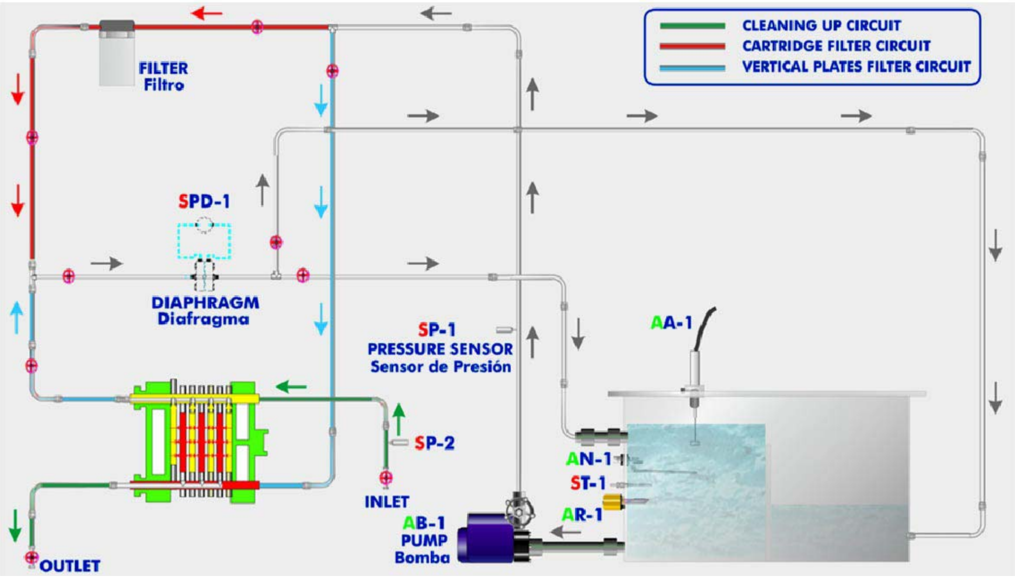
2. Centrifugal filters: these filters use the centrifugal force as the driving force. These filters really are centrifuges fitted with perforated container which is placed on a filter medium. The suspension to be filtered is fed into the container subjected to centrifugal forces, then the filtrate flows through the filter medium and cake builds up inside the container.

2.4. UNIT DESCRIPTION

2.4.1 Description

(a) (b)

a)Unit TFUC. Batch Filtration Unit b)Control Interface Box



ST=Temperature sensor.

SP=Pressure sensor.

AR=Heating resistance.

AN=Level switch.

AA=Stirrer.

SPD=Differential pressure sensor (flow measurement).

AB=Pump.

Figure 2.3 Process Diagram and Elements Allocation[6]

This filtration unit lets the study of the filtration process for two different filters. On one side a vertical plates filter, composed of sheets of nylon, 5 microns in diameter, allowing us to filter the CaCO suspension of known concentration. In addition, a filter cartridge, more suitable for continuous filtration of material in a larger size, will filter and “clean” water with small pieces of paper sample.

To carry out the experiments, the mixture is taken from a tank through a centrifugal pump, which sends it to one of the filters depending on the position of the valves. A motor-driven stirrer will make more homogeneous the sample inside the tank.

Along the way, the mixture goes through one pressure sensor and differential pressure sensor, which will help us to determine the flow through the circuit. The initial temperature of the sample can be controlled by a resistance and a temperature sensor, which will give the temperature value at any time.[6]

2.5 EXPERIMENTAL PROCEDURE

Practice 1: Study of the filter plate at a constant pressure

Prepare a suspension of calcium carbonate in water at a known concentration. It is important to know the proportion and concentration of the same with reasonable accuracy to perform the practice. Then put the suspension inside the feed tank of the unit. Write the height of water in the tank, and multiply it by the other two dimensions of it, to know the starting volume of the suspension. Then start to stirr it.

After a stirring time, switch the pump to begin the process of filtration and setting the pressure value to keep it constant. The time spent during the process will depend on the starting concentration of the sample. As the concentration is lower, a longer time will be spent until seeing a change in the flow. Write the filtering volume value each some time. In order to obtain the filtering volume for a given time, the Arquimedes Principle will be used.

By using the milimetric scale on the tank, we will be able to know the volume of filtered solid. To do this, as we have written the starting volume of the sample, we just need to see again the height of the sample we have at a given time. In order to get a higher accuracy, stop the stirrer before. The difference between the current volumeand the starting one will be the volume of filtered solid. Do this experiment at some different pressure values.

Once saved all these experimental data, do the next plots:

− $\Delta\theta/\Delta V$ versus filtered volume: in this plot you should obtain **α** (specific resistance of the cake) an r (filtrating media resistance).

Practice 2: Study of the filter plate at a constant flow.

As in the previous practice, prepare a suspension of calcium carbonate in water at a known concentration. It is important to know the proportion and concentration of the same with reasonable accuracy to perform the practice. Then put the suspension inside the feeding tank of the unit. Write the height of water in the tank, and multiply it by the other two dimensions of it, to know the starting volume of the suspension. Then start to stir it.

After a stirring time, switch the pump to begin the process of filtration and setting the flow value to keep it constant.

The time spent during the process will depend on the starting concentrationof the sample. As the concentration is lower, a longer time will be spent until seeing a change in the pressure. Write the filtering volume value each some time. In order to obtain the filtering volume for a given time, the Arquimedes Principle will be used. By using the milimetric scale on the tank, we will be able to know the volume of filtered solid. To do this, as we have written the starting volume of the sample, we just need to see again the height of the sample we have at a given time. In order to get a higher accuracy, stop the stirrer before. The difference between the current volume and the starting one will be the volume of solid filtered.

Do this experiment at some different flow vales.

Once saved all these experimental data, do the nex plots:

− $\Delta\theta/\Delta V$ versus filtered volume: in this plot you should obtain **α** (specific resistance of the cake) an **r** (filtrating media resistance).

Practice 3: Viewing the filtration process.

Prepare a suspension of calcium carbonate in water at a known concentration (not higher than 2g/l more or less, in order not to overload the turbidimeter). Then pour the suspension into the feeding tank and begin to shake it. Take a sample of the suspension and measure turbidity.

After some stirring time, switch the pump to start the process. This practice can be performed either at a constant flow and constant pressure. Take turbidity measurements (of the sample in the tank) at some different times and see how it goes down. The turbidity of the sample should decrease because part of the solute remains in the filters, so that the concentration of the mixture decreases.

NAME-SURNAME:

GROUP NO:

ASSISTANT: **DATE:**

DATA SHEET

Practice 1: Study of the filter plate at a constant pressure

	1	2	3	4	5	6	7	8
Volume filtrated (ml)								
Time (min)								

Practice 2: Study of the filter plate at a constant flow.

	1	2	3	4	5	6	7	8
Volume filtrated (ml)								
Time (min)								

2.6 NOMENCLATURE

A: Surface (m^2)

c: fraction of solids in the flow of supplies or concentration

F: driving force force (gravity, thrust of a pump or centrifugal force) (N)

m: Difference in mass between the wet cake and the dry cake (kg)

P: total pressure fall (N/m^2)

R: resistance is the sum of that offered by the filter medium and the solid cake formed on it. (kg/s)

r: filtering medium resistance (1/m)

V: filtered volume (m^3)

θ: filtering time (s)

μ: filtrate viscosity (kg/ms)

α: specific cake resistance.(m/kg)

W: cake weight (kg)

w: solid mass per filtered unit (kg/m^3)

ρ: filtered density (kg/m^3)

2.7 REFERENCES

1. Geankoplis,C.J., 2003, Transport Processes and Seperation Process Principles,4th edition, Prentice Hall, 906-919.
2. Geankoplis,C.J., 2003, Transport Processes and Seperation Process Principles,4th edition, Prentice Hall, 92-93.
3. Tien,C.,2006, Introduction to cake filtration analyses,experiments and applications,Elsevier, 1-10.
4. McCabe W., Smith J.C, Harriott P., 1993, Unit Operations of Chemical Engineering, 5th edition, McGraw-Hill, 1016-1026.
5. P. Park, C. Lee, S. Lee, 2006, Variation of specific cake resistance according to size and fractal dimension of chemical flocs in a coagulation-microfiltration process, Euromembrane 2006 , 199, 213-215.
6. Practices Manual, 2006, Edibon, S.A. , 1-24.
7. S.A. Lee, A.G. Fane, R. Amal and T.D. Waite, 2003, The effect of floc size and structure on specific cake resistance and compressibility in dead-end microfiltration, Separation Science and Technology, 38, 869–887.
8. Y, Endo, M. Alonso, 2001, Physical Meaning of Specific Cake Resistance and Effects of Cake Properties in Compressible Cake, Filtration & Separation, 38, 42-46.