

CRACOW UNIVERSITY OF TECHNOLOGY
Faculty of Chemical Engineering and
Technology



DETERMINATION OF EQUIVALENT
DIAMETERS FOR IRREGULAR
PARTICLES

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*Authoress: **Ángela Santiago Dehesa**
UNIVERSIDAD DE CANTABRIA
Escuela Técnica Superior de Ingenieros
Industriales y de Telecomunicación*

*Supervisor: Dr. W. Figiel, prof.
PK POLITECHNIKA KRAKOWSKA
Faculty of Chemical Engineering and Technology*



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INTRODUCTION

1. INTRODUCTION

In the ideal world of the particle characterization, all the particles would be homogeneous spheres. Moreover, they would have uniform properties such as density, chemical composition, color, and opacity. Then, all the size measurement methods would yield the same size for a particle, being its diameter, and the same particle size distribution for a collection of particles, regardless of the principle applied in the technique. It will be clear that such particles would constitute the ideal standard reference material for particle size measurement methods. It will also be clear that this world would be rather dull.

In the real world, most particles are not spherical, but have different shapes and often rough surfaces. Materials of different chemical composition have different properties and, even when the bulk composition is the same.



Spherical particle



Irregular particle

The size of spherical homogeneous particle is uniquely defined by its diameter. For regular, compact particles such as cubes or regular tetrahedral, a single dimension can be used to define the size. With some regular particles it may be necessary to specify more than one dimension: for a cone the base diameter and height are required whilst for a cuboid three dimensions are needed.

Derived diameters are determined by measuring a size-dependent property of the particle and relating it to a single linear dimension. The most widely used of these are the equivalent spherical diameters.



1.1. JUSTIFICATION: OBJECTIVE OF STUDY

The aim of the study is to obtain a good method for finding the equivalent diameter of irregular stones, taking account likewise what is the best way to carry out the analysis.



DEFINITIONS

2. DEFINITIONS

2.1 EQUIVALENT DIAMETERS

The equivalent diameter is defined as "the diameter of a spherical particle that would pass through a sieve with a certain opening x". This is done in order to harmonize the irregularities of shapes in a form close to the sphere. Obviously it is assumed as an inherent error in a granulate know that most of the particles have different shapes.

Different types of equivalent diameters:

2.1.1. Diameter of a sphere of equivalent volume (d_v).

Is the diameter of a sphere having the same volume (V) to the desired particle characterization.

$$d_v = \sqrt[3]{\frac{6V}{\pi}}$$

2.1.2. Diameter of a sphere of equivalent area (d_s).

Is the diameter of a sphere having the same external surface (S) to the desired particle characterization.

$$d_s = \sqrt{\frac{S}{\pi}}$$

2.1.3. Surface- volume diameter (d_{sv}).

Diameter of a sphere having the same eternal surface (S) to volume ratio (V) as the particle

$$\frac{S}{V} = \frac{\pi d_{sv}^2}{\frac{\pi d_{sv}^3}{6}} = \frac{6}{d_{sv}}$$

$$d_{sv} = \frac{6V}{S}$$

2.1.4. Projected area diameter (d_a).

Diameter of a circle having the same projected area (A) as the particle in stable orientation.

$$d_a = \sqrt{\frac{4A}{\pi}}$$

Figure 2.1 shows a projection of an irregular particle (blue trace). Setting the area of this projection is possible to estimate the diameter of a circle equivalent (figure red line). The microscopy allows estimation of d_a . Figure 2.2 indicates that depending on which direction the projected area is obtained, the areas will differ and consequently different equivalent diameters are obtained.

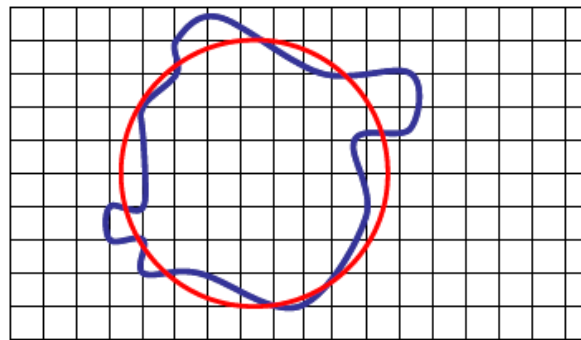


Fig. 2.1 Conceptualization of projected area.

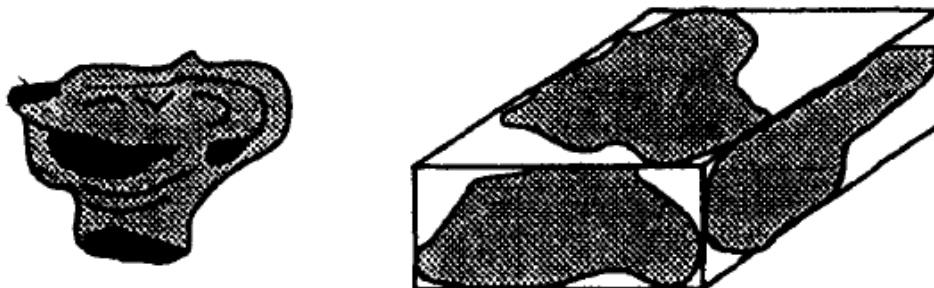


Fig. 2.2 View of the projected areas according to the orientation.

2.1.5. Perimeter diameter (d_c).

Is the diameter of a sphere having equal perimeter (P) that the particle to be characterized. This measure also depends on the orientation of the particle at the time of measurement.

$$d_c = \frac{P}{\pi}$$

2.1.6. Stokes diameter (d_{ST}).

A small, spherical, homogeneous particle settling in a fluid, rapidly reaches a constant 'terminal' velocity which is uniquely related to the diameter of the sphere. If an irregularly shaped particle is allowed to settle in a liquid, its terminal velocity may be compared with the terminal velocity of a sphere of the same density settling under similar conditions. The size of the particle, defined as its Stokes diameter, is then equated to the diameter of that sphere. In the laminar flow region particles settle in random orientation and a single particle generates a range of equivalent diameters depending its orientation.

The Stokes diameter is some average of these. Outside the laminar flow region particles orientate themselves to give maximum resistance to motion and the free falling diameter that is generated will be the smallest of these diameters.

In laminar flow the terminal velocity is:

$$V_T = \frac{d_{ST}^2 (\rho_P - \rho_f) g}{18\mu}$$

Therefore the Stokes diameter is:

$$d_{ST} = \sqrt{\frac{18 \cdot V_T \cdot \mu}{(\rho_P - \rho_f) g}}$$

Thus μ and ρ_f is the fluid viscosity and density, respectively. P_p is the particle density.

2.1.7. Martin diameter (d_M)

Martin's diameter is used in microscopy, represents the length of the line that bisects the projected area. Figure 2.3 is introduced to clarify this concept. Depending on which direction the line is drawn bisecting the area, you will get different diameters Martin.

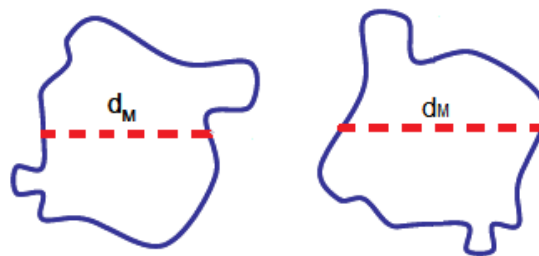


Fig. 2.3 Martin diameter

2.1.8. Feret diameter. (d_F)

The Feret diameter, as the diameter of Martin, is used in microscopy. In this case represents the distance between two parallel lines which are tangential to the contour of the projection of the particle, as seen in Figure 2.4. As with the Martin diameter can be determined Feret different diameters according to the direction that the tangents are drawn.

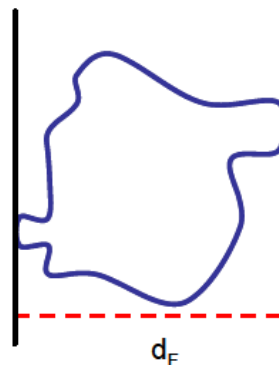


Fig. 2.4 Feret Diameter

2.1.9. Sieve diameter (d_A).

A method of measuring particle size is sieved particles which have mesh sieve with square openings, as seen in Figure 2.5 the diameter corresponds to the sieve mesh opening.

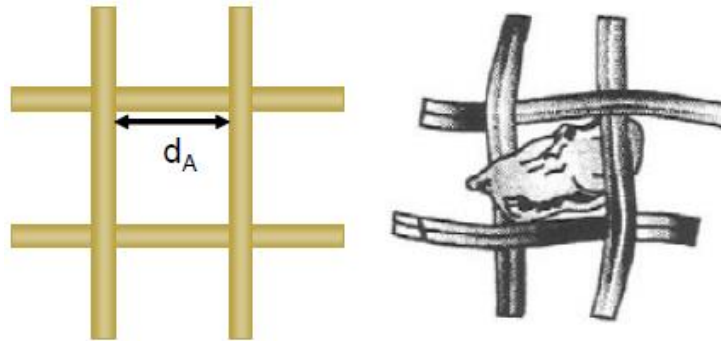


Fig. 2.5 Sieve diameter

The equivalent diameter best suited for a particular particle depends on what this value will be used. For example if desired design a settler, Stokes diameter is a good representation of the particles. If the calculated diameter is used to study chemical reactions or surface heat transfer phenomena and the mass ratio maintained diameter outer surface per unit of volume is adequate. For the use of pigments of the projected area diameter is recommended, since the adhesion is related to the projection of the particle on the surface to cover.

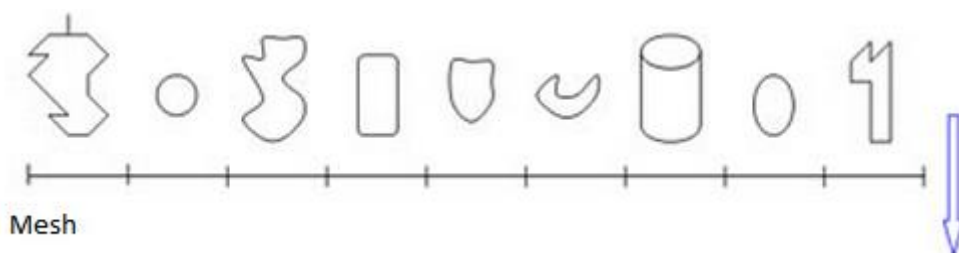


Fig. 2.6 Possible forms of particles that can pass through a screen with the same diameter.

2.2. SPHERICITY.

Sphericity (Ψ) is a measure of how spherical (round) an object is. As such, it is a specific example of a compactness measure of a shape. The definition is “the ratio of the surface area of a sphere (with the same volume as the given particle) to the surface area of the particle”.

$$\Psi = \frac{\sqrt[3]{\pi} \cdot \sqrt[3]{(6 \cdot V_p)^2}}{A_p}$$

The sphericity of a sphere is 1 and, by the isoperimetric inequality, any particle which is not a sphere will have sphericity less than 1.

2.3. SIZE MEASUREMENT

Use of a microscopy should always accompany size analysis by any method because it permits an estimate to be made of the range of sizes present and the degree of dispersion. In most instances, particle size measurement rarely follows, as other procedures are faster and less stressful to the operator. Microscope size analysis is used primarily as an absolute method of size analysis, as it is the only one in which individual particles are seen. It is used perhaps more for shape/morphology analysis than size analysis and for beneficiation studies of minerals when combined with x-rays analysis. It is also used when other methods are not possible.

Clearly, the range of sizes and their degree of dispersion strongly influence the ease and reliability of size measurement. For instances where particles are not already deposited on surfaces, sample preparation is a critical step. This is discussed in the detail by Allen. Typically, samples can be extracted from an agitated well-dispersed suspension, but dry, temporary, or permanent mounts also possible. For dry mounts, particles can be dusted onto a surface; for temporary slides, powder can be dispersed-held in viscous media.

When a satisfactory dispersion of particles on a relevant substrate has been achieved, particle size analysis can follow. Points to consider are resolution, the total number of particles to be counted), and the effect of material properties.

The microscope gives us data as aspect ratio, area, CE diameter, circularity, convexity, elongation, length or width.

2.4. PARTICLE SHAPE CHARACTERIZATION

Particles have various shapes depending on their manufacturing method and mechanical properties. Unlike a sphere or a rectangular parallelepiped, which are objects with clear geometrical definitions, powder particles are very complicated objects with no definite form. It is generally very difficult to describe their shapes. They have therefore been conventionally described and classified by the use of various terms.

On the other hand, along with recent rapid advances in computer and information technologies, image processing technology has remarkably progressed in both software and hardware, making it easy to obtain image information and geometrical features of particles. Accordingly, various quantitative methods for expressing particle shape have been proposed and adopted.

The particle shape description or expression is classified by several criteria. In the most primitive manner, the descriptions are classified into quantitative methods and qualitative ones.

In qualitative description, the shape is expressed by several terms such as “spherical”, “granular”, “blocky”, “flaky”, “platy”, “prismatic”, “rodlike”, “acicular”, “fibrous”, “irregular”, and so on. The verbal description is sometimes convenient to express irregular shape and makes it easy to understand the shape visually. But how are platy shape and a flaky shape distinguished? Under present conditions, this distinction must depend on human visual judgments in many cases. Therefore, quantitative descriptions of particle shape will be necessary.

Quantitative shape descriptors can be calculated from 2 or 3 dimensional geometrical properties and can be calculated by comparing with physical properties of the reference shape.

The following are required for the shape descriptor:

- ❖ Rotation invariance: values of the descriptor should be the same in any orientation.
- ❖ Scale invariance: values of the descriptor should be the same for identical shapes of different size.
- ❖ Reflection invariance.
- ❖ Independence: if the elements of the descriptors are independent, some can be discarded without the need to recalculate the others.
- ❖ Uniqueness: one shape always should produce the same set of descriptors, and one set of descriptors should describe only one shape.
- ❖ Parsimony: it is desirable that the descriptors are thrifty in the number of terms used to describe a shape.

2.5. SETTLING VELOCITY OF AN ISOMETRIC PARTICLE

The experimental device includes a mechanism of conditioning the sedimentation of solid particles and a system of measurement based on a space-time location. This method offers the advantage to not disturb the flow. The sedimentation device consists of a glass column of 10.7 cm of radio and 1.20 m height, bearing in minds only 80cm of route.

The dimensions of the measurement column are selected by taking in to account the practical requirements of the laboratory and the experimental conditions but also having in mind the minimization of the influence of the wall on the settling velocity.

The sedimentation test velocity is higher in infinite medium than the sedimentation velocity in column. in an unlimited field, the fall of particle induces the same speed direction in all points of the fluid and the drive effects is felt far from the particle. On the other hand, in a limited field, the flow of the fluid is constrained. The drive effect is induced near by the particle, generating a return movement of the fluid, associated with viscous frictions at the wall.

2.6. STOKES LAW

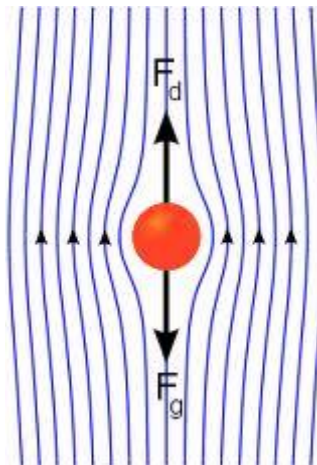
In 1851, George Gabriel Stokes derived an expression, now known as Stokes law, for the frictional force – also called drag force – exerted on spherical objects with very small Reynolds numbers (e.g., very small particles) in a continuous viscous fluid. Stokes' law is derived by solving the Stokes flow limit for small Reynolds numbers of the Navier–Stokes equations:

$$F_d = 6\pi \mu R v_s$$

where:

- ❖ F_d is the frictional force – known as Stokes' drag – acting on the interface between the fluid and the particle (in N),
- ❖ μ is the dynamic viscosity (N s/m²),
- ❖ R is the radius of the spherical object (in m), and
- ❖ v_s is the particle's settling velocity (in m/s).

The condition of low Reynolds number laminar flow means which can be translated by a relative speed between the field and the lower half at a certain critical value. Under these conditions the resistance of the medium is almost exclusively due to the friction forces that oppose the sliding layers on other fluid from the boundary layer adhered to the body. Stokes law has been verified experimentally in a variety of fluids and conditions.



If the particles are falling vertically into a viscous fluid due to its own weight can be calculated sedimentation velocity equaling or fall of frictional force with the apparent weight of the particle in the fluid.

$$v_s = \frac{2(\rho_p - \rho_f)}{9\mu} g R^2$$

where:

- ❖ v_s is the particles' settling velocity (m/s) (vertically downwards if $\rho_p > \rho_f$, upwards if $\rho_p < \rho_f$),
- ❖ g is the gravitational acceleration (m/s^2),
- ❖ ρ_p is the mass density of the particles (kg/m^3), and
- ❖ ρ_f is the mass density of the fluid (kg/m^3).

Stokes' law makes the following assumptions for the behavior of a particle in a fluid:

- ❖ Laminar Flow
- ❖ Spherical particles
- ❖ Homogeneous (uniform in composition) material
- ❖ Smooth surfaces
- ❖ Particles do not interfere with each other.

2.7. DENSITY OF SOLIDS

An important feature of the solid particles (both small powders, such as large, fruit) is its density. We should begin by distinguishing between the density per unit density sometimes called "real", and the overall density or "apparent". The first is the average mass per unit volume of the individual particles. Is determined by weighing the particles in air and determining its volume by displacement of a liquid, generally water. The ratio of weight (kg) divided by volume (m^3) is the actual density. If the particle size is small, employing a gradient tube filled with two miscible liquids of different densities and allowed to equilibrate for several days. Are introduced into the glass beads of known

density are measured height which are, at constant temperature, and is a graph representing the density function of the height. And calibrating the gradient, the sample is introduced and its density is determined based on the height reached in the tube by reference to the calibration graph.

The absolute density: is defined as the ratio of the mass per unit volume of a body at a given temperature.

$$\rho = \frac{m}{V}$$

Where:

Ψ M: mass in Kg

Ψ V: Volume in m³

The absolute density is a function of temperature and pressure. The density of some liquids in function of the temperature. The variation of the density of the liquid is very small, except at very high pressures and for all practical calculations can be neglected.

Relative density is defined as the ratio of the density of the substance with respect to the pattern density, which leads to a relationship between the mass of the test substance to the mass of the same volume of distilled water at pressure atmosphere and 4 ° C. It is evident that the relative density is a dimensionless quantity:

$$\rho_r = \frac{\rho_s}{\rho_w}$$

$$\rho_s = \frac{m_s}{V}$$

$$\rho_w = \frac{m_w}{V}$$

Substituting

$$\rho_r = \frac{\frac{m_s}{V}}{\frac{m_w}{V}} = \frac{m_s}{m_w}$$

Pycnometer

Using the pycnometer (Fig 2.7) we can determine the relative density of irregular stones. This makes weighing of the empty pycnometer perfectly dry and subtracting the heavy pycnometer with the analyte and with distilled water. Thus, for the same volume gives the mass of the test substance and the mass of distilled water, using the last equation.



Fig. 2.7 Pycnometer.

To calculate the density of a solid with a pycnometer, which has to be dimensioned to pycnometer, weighing the pycnometer with the solid and fill with distilled water, we can get the volume of the solid. If in addition we have calculated the volume of the pycnometer. Moreover, once the mass of the solid obtained we can calculate its density.

P_P	$P_P + M$	$P_P + M + A_F$	$P_P + A_F$
1) Weigh the empty pycnometer conveniently clean empty: P_P	2) Put the sample into the pycnometer and weighed: $P_P + M$	3) The pycnometer filled with distilled water. Put the stopper, is flush, the outside was dried and weighed: $P_P + M + A_F$	4) The pycnometer was filled with distilled water and weighed: $P_P + A_F$

Procedure should be repeated at least 2 times.

Calculations:

2) – 1) = Solid mass **M**

4) – 1) = Water mass **A**

3) – 2) = not dislodged water **A_F**

$A - A_F$ = Dislodged water \rightarrow Solid volume

$$\rho = \frac{m}{V} = \frac{m}{A - A_F}$$

Archimedes Principle

"All solid volume V immersed in a fluid experiences an up thrust equal to the weight of the fluid evacuated."

Determining the solids density by the Archimedes principle consists in determining the thrust (E) which is carrying out the difference between the weight of the solid in air (W_s) and the apparent weight of the solid immersed in the liquid (W_a). The volume of liquid corresponding to the volume vacated submerged solid. This method is perfect for irregular solids.

$$E = W_{des} = W_s - W_a = Vd_L$$

Where W_{des} is displaced fluid weight, V volume of the solid and liquid density d_L . Weigh a beaker (in place you can use a plastic container) partially filled with water (w_b). The solid is then tied with a thin thread and is suspended in the beaker of water as shown in Fig. 2.8. Make sure not to touch the solid walls of the vessel. Obtained system weight and its weight are recorded as W_T .

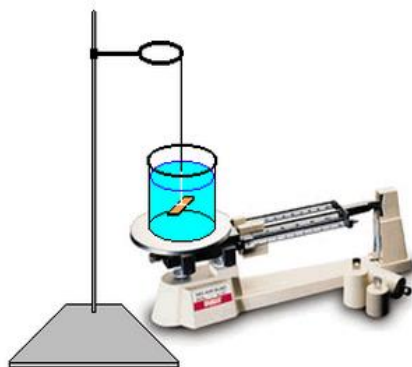


Fig. 2.8 Archimedes Principle

The rope weight solid supports but does not nullify the push, so that W_T equals the weight of the container with more water thrust (weight of water displaced by the solid W_{des}).

$$E = w_{des} = W_T - w_b = Vd_L$$

Bearing in mind that in the above equation, the density can be calculated from the expression:

$$d_s = \frac{W_s}{V} = \frac{W_s}{(W_T - W_b)} d_L$$

Where, if the liquid is water, d_L corresponds to 1.00 g / ml.

Test tube method

Is a very common method for irregular solids. The problem that arises is that the solid has to be quite large, so to properly appreciate the volume change in the test tube.

The solid was carefully and completely immersed in a beaker containing water exact volume (V_o). Then carefully reads the final volume (V_f). The volume of the solid is the difference:

$$V = \Delta V = V_f - V_o$$

As found with this method the particle volume by measuring the mass easily obtain the density of said solid.

Geometrical method

This is a method that can be used only for geometric particles. The particle is to weigh and get your objects geometrically volume measurement precision.



METHODOLOGY OF WORK

3. METHODOLOGY OF WORK

3.1. LABORATORY MATERIAL

3.1.1. GLYCEROL

Glycerol (Fig.3.1) is a simple polyol compound. It is a colorless, odorless, viscous liquid that is widely used in pharmaceutical formulations. Glycerol has three hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature. The glycerol backbone is central to all lipids known as triglycerides. Glycerol is sweet-tasting and of low toxicity.

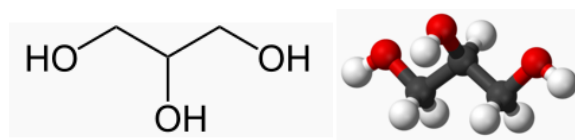
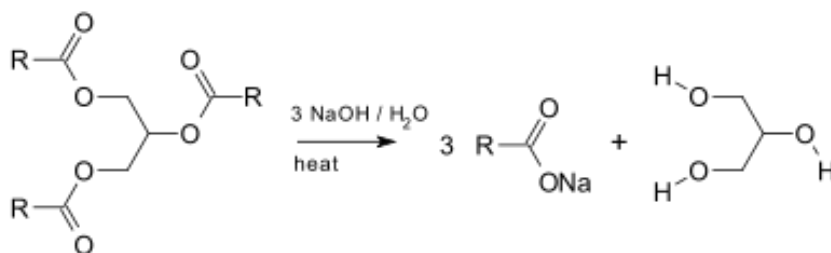


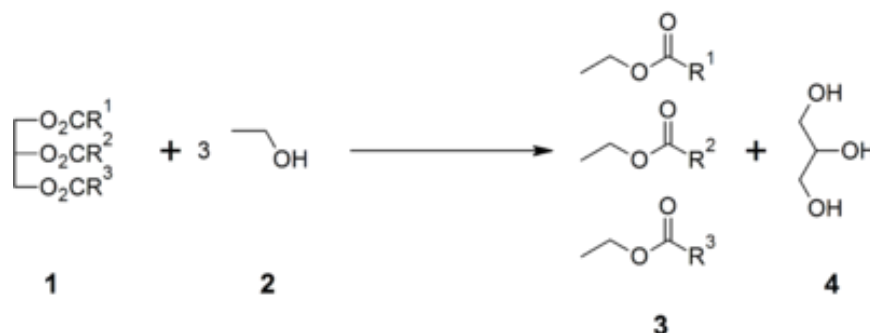
Fig.3.1 Glycerol's structure

From fats and oils

Triglycerides found in fats and oils are by definition esters of glycerol with long-chain carboxylic acids; the hydrolysis (saponification) or transesterification of these triglycerides produces stoichiometric quantities of glycerol. In this scheme, glycerol is produced as a co-product in the production of long-chain carboxylate salts used as soaps (see soap-making):



It is also a byproduct of the production of biodiesel via trans esterification. This form of crude glycerin is often dark in appearance with a thick, syrup-like consistency. Triglycerides (1) are treated with an alcohol such as ethanol (2) with catalytic base to give ethyl esters of fatty acids (3) and glycerol (4):

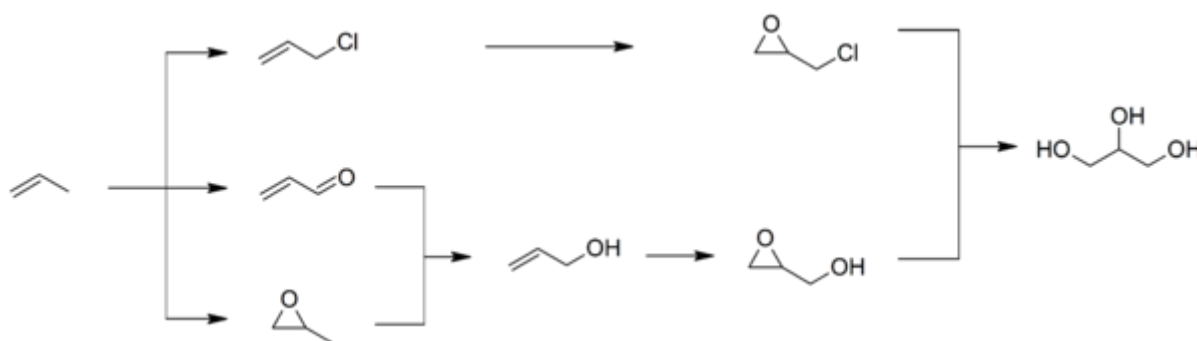


Glycerol from triglycerides is produced on a large scale, but the crude product is of variable quality, with a low selling price of as low as 1-8 U.S. cents per pound in 2011. It can be purified, but the process is expensive. As a result, a good fraction of crude glycerol is disposed of as waste. Some glycerol is burned for energy, but the heat value is low.

That said, crude glycerol from the hydrolysis of triglycerides can be purified by treatment with activated carbon to remove organic impurities, alkali to remove unreacted glycerol esters, and ion exchange to remove salts. High purity glycerol (> 99.5%) is obtained by multi-step distillation; vacuum is helpful due to the high boiling point of glycerol (290 °C).

Synthetic glycerol

Synthetic glycerol refers to material obtained from non-triglyceride sources. Glycerol may also be produced by various routes from propylene. The epichlorohydrin process is the most important; it involves the chlorination of propylene to give allyl chloride, which is oxidized with hypochlorite to dichlorohydrins, which reacts with a strong base to give epichlorohydrin. Epichlorohydrin is then hydrolyzed to give glycerol. Chlorine-free processes from propylene include the synthesis of glycerol from acrolein and propylene oxide.



Because of the emphasis on biodiesel, where glycerol is a waste product, the market for glycerol is depressed, and these old processes are no longer economical on a large scale. In fact, due to the glycerol glut, efforts are being made to convert glycerol to its precursors, such as acrolein and epichlorohydrin.

Applications

- ❖ The manufacture of cosmetics such as soaps. The glycerin increases detergency, whiteness gives a smoother and softer skin.
- ❖ In the field of medicine used in the preparation of medicaments in the form of syrups (as excipient; as antiseptic to prevent infections in wounds, as inhibitor of enzymatic changes during the fermentation of ointments, pastes or creams, as a solvent for iodine, bromine, phenol, thymol, tannins, alkaloids and mercuric chloride). It is also used for ophthalmic lubricants and moisturizers.
- ❖ As heating bath to temperatures above 250 ° C.
- ❖ Specific machinery lubrication. For example, production of food and drugs (not to be toxic), oil, etc.
- ❖ In military disciplines for the manufacture of explosives, such as nitroglycerin and to cool the barrels of guns.
- ❖ Antifreeze (low melting point of the water, by the cryoscopic drop).
- ❖ Preparation of alkyd resins which are used as insulators.
- ❖ Fluid separator instruments capillary tubes.

- ❖ Paint and varnish industry. Key component varnishes used for finishes. In some cases, use of 98% glycerol to prepare varnishes electro insulating.
- ❖ Tobacco industry. Due to the high binding capacity of glycerin, the moisture can be adjusted in order to eliminate the unpleasant taste and irritating smoke snuff.
- ❖ Textiles. Provides elasticity and softness to fabrics.

3.1.2. SOLIDS

For the selection of the particles used in this project I made a selection process, focusing especially on the particle shape and the relationship of this feature with its free-fall conditions within a fluid. Thus various types of shapes selected (analyzed fall within a water pipe):

- ❖ Shaped boulders half ellipse: not fall straight but sway even colliding with the walls of the tube.
- ❖ Angled triangular stones: fall too fast to determine the time, also without fall straight.
- ❖ Very angled porous: Some break to hit the tube, and not collide, they lose small particles as they descend.
- ❖ Angled with rounded edges: at 10 cm from start turning on themselves and do tours spiral inside the tube.
- ❖ Angulated without rounded corners: 90% of them fall in a straight line, also cross the tube long enough to measure time.

After having selected particles is necessary know various aspects thereof. Some example might be the projection area, the convexity or length data provides a microscope, and others are the mass and density.

Obviously, we calculate the mass without any problem with a precision balance. Furthermore, to calculate the density, it would be easier to calculate the volume of the same. The first problem we face when having irregular particles, even so, there are methods described above with which you can calculate this density.

The most affordable option in material and the simplicity of the experiment would be to use the method of the test tube. On the other hand, this method is discarded without needing an attempt to support this. This is because our sample particles are too small to observe a change in the height of the liquid would not be possible to obtain a precise volume.

The next case would be the method to analyze the principle of Archimedes. The characteristics of the sample are usually good for this method. The problem is that a university laboratory usually not equipped with a scale with sufficient precision for it. For this reason, this method is also discarded.

In the case of the pycnometer, nothing seemed to indicate it was not a suitable method. After the experiment, to perform calculations and get the densities, we note that these are not correct. Observing this thought, probably the mass of the particles is too low for this method, or even, the sample is very small, as our final sample consists of only 12 particles.

Due to the problems encountered during the process, we finally decided to assume an approximate density for each particle, between 2.5 and 3.3 g/cm³, due to the material. Thus can calculate each limit velocity by Stokes law.

3.1.3. INSTRUMENTS.

Refractometer

Called refractometry, optical method of determining the speed of propagation of light in a medium/ compound/ substance/ body, which is directly related to the density of the medium/ compound/ substance/ body. To use this principle uses the refraction of light, (which is a fundamental physical property of a substance), and the measurement range of this principle is called refractive index, refractometers (Fig. 3.2) are instruments that employ this either refraction (using several prisms), or the critical angle (prism using only one), and its primary scale of measurement is the refractive index, from which specific construct different scales, Brix (sugar), specific gravity, % salts.



Fig. 3.2 Refractometer ABBE.

Refractometers, used to measure concentrations in liquids, usually offer a reading of Brix. To find the concentration of the liquid to be measured should be the conversion from Brix refractometer reading multiplying it by a specific constant value or by using a correspondence table of the measured solution itself, in this case glycerol.

By definition, the brix is a measure of density. Brix is a density that is at 20 ° C, a solution of 1% sucrose.

The refractive index has many applications in the field of chemistry, including product identification, quantitative analysis of solutions, determination of the purity of samples, and are useful for determining dipole moments, molecular structures and molecular weights approximate.

Tensiometer

I used the tensiometer methods uses elastic, which means that the surface is maintained over time. This tensiometer combines two methods:

- ❖ Plate method (Whilhelmy 1863): It uses a rectangular plate geometry suspended well known vertically on a precision balance. The underside of the plate is contacted (horizontally) with the liquid surface to become wet. Then a vertical force is exerted on the plate to lift. The plate was slowly raised, and each side forming a curved interface, the plate is lifted until starting occurs.

In the position just before the start can calculate the power balance between the tensile forces applied to the part and another plate (hence the factor 2) and the lifting force F (Fig. 3.4).

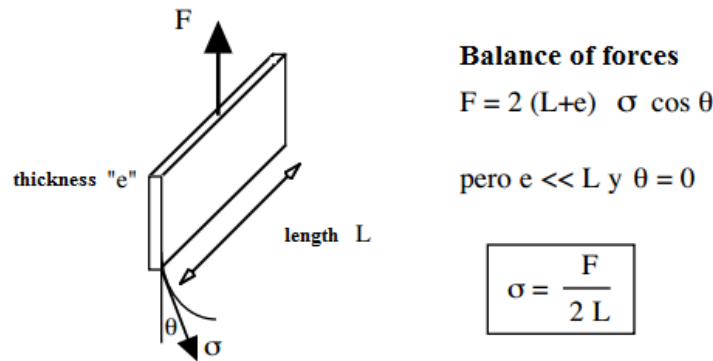


Fig. 3.4 Balance of forces

- ❖ Ring method (Nouy 1919): In Nouy method, an O-ring is used suspended horizontally, perfectly parallel to the surface or interface. The ring has a radius R , and is made with a wire of radius r , resulting in a total circumference $L = 4\pi R$. Note that this is an approximation perimeter; it does not take into account the exact position of the three phase contact line relative to the ring. In any case is valid if $r \ll R$. To measure the surface tension, one proceeds as in the case of plate method.

First it gets wet (completely) the ring and then proceeds to lift up the boot. However in this case, the situation is slightly different for two reasons:

Whatever the contact angle, the direction of application of the tensile force varies as it is extracted from the liquid ring. There is a position of the contact line at which the tensile force is vertical. In this position the vertical projection of the tension force is maximum. The experimental method takes into account this feature, since it measures the maximum force.

Furthermore one should consider that except in the case where $r \ll R$, then the medial meniscus and the lateral meniscus do not have the same shape (Fig. 3.5). Consequently there are actually two positions in the force passes through a maximum. To avoid this problem is that there is always $r \ll R$.

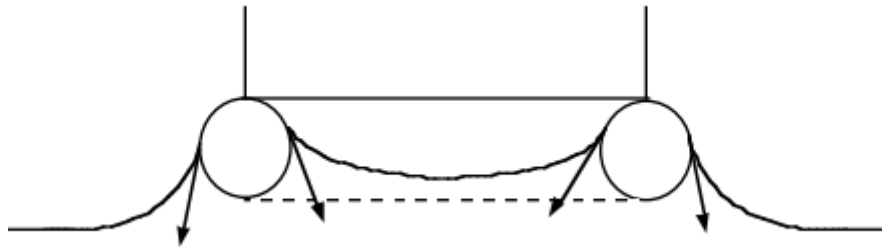


Fig. 3.5 Difference between the internal meniscus and the external meniscus.

Electronic microscope

The instrument that I used (Fig. 3.6) is based on automated image analysis, is a new concept in characterization of particles provides microscopy images of high quality and very important statistical information about the size and shape of the particle, through rapid analysis of hundreds of thousands of particles, practically without user intervention.



Fig. 3.6 Morphologi G3

An additional capability allows the automated detection, enumeration and classification of foreign particles sized on a filter.

The G3-ID system Morphologi particle characterization combined with automatic image analysis chemical identification of individual particles by Raman spectroscopy. This fully automated measuring particle size, particle shape and chemical identification using a single platform.

I got this instrument measure data as complicated as the equivalent diameter, the convexity or the area of the particles in the sample. A very important due to the large irregularity thereof.

Rotational viscometer

Rotational viscometers are standard in all industries. Viscosity measured using the torque required to rotate a spindle immersed in a liquid at a constant speed. The torque is proportional to the spindle drag in the fluid, and therefore the viscosity.

Rotational viscometers employ the idea that the force required to rotate an object immersed in a fluid may indicate the viscosity of the fluid. Some of them are:

The most common rotational viscometer of Brookfield type is determining the force required to rotate a disk or spangle in a fluid at a known speed.

The viscometer of 'Cup and bob' that work by determining the torque required to achieve a certain rotation. There are two classical geometry in this type of rotational viscometer, systems known as "Couette" or "Searle".

'Cone and plate' the viscometers used a cone is introduced into the fluid at a very shallow depth into contact with the plate.

The Stormer viscometer. A rotating device is used to determine the viscosity of paints; it is widely used in the paint processing industries. Is a kind of type paddle bladed rotor which is immersed in a liquid and begins to rotate at 200 revolutions per minute, the engine load measured for doing this is in viscosity ASTM D 562 tables which determine the viscosity in Krebs units. The method applies to both brush paintings and roll.

Experiment installation

The installation of the experiment consists of a glass tube about a meter high and 10.7 cm in diameter. The pipe provided in the bottom of a mesh for collecting the particles and thereby facilitates the experiment.

Likewise, the tour which measures the time of fall of the particles is determined by two lines delimiting. These lines serve the function that when the particle arrives at the first point has already reached its limit speed. The second line is to delimit the travel 80cm representing the experiment, and thus to calculate the velocity.

Of course, to the aforementioned speed control, a stopwatch was used analog. Moreover, for greater control over the temperature has been obtained at which the temperature fell each particle in each fluid with the help of a thermometer.

3.2. EXPERIMENTAL PROCEDURE

After find out which are the best stones for our experiment, change the tube for liquid glycerin 85%. At this time I take a larger sample of the type of stones only I selected (without rounded edges angled) and do a more thorough study on how such stones and fall exactly as each.

In conducting this experiment thorough we can see that within the particles with these characteristics (angled flat without rounded edges), there are a particularly useful for our study, these are the addition to the features mentioned are quadrilateral. This is because 90% of them fall in a straight line and take a considerable time in traversing to be measured.

To continue the experiment, glycerol change by 85% to 74% glycerol. Again I throw stones selected, so I can see the difference in behavior between the two particles have concentrations.

Concentration of glycerol solutions the measure with a refractometer. As the first solution (85%) were of unknown concentration, and the second (74%) was created following the first by adding water. So using the refractometer and a calculator to the glycerol concentrations get.

To find the density of the two liquids, I made use of a Kruss K11 tensiometer. Data necessary to identify the speed limit using Stokes' law.

The tensiometer gives the measurement of the 85% glycerol has a density of 1.227g/ml (21.5 ° C) and 74% glycerol has a density of 1.191g/ml (21.9 ° C).

After this, we obtain both the viscosity of liquids using a rotational viscometer.

3.3. WORKING TOOLS

For laboratory results, and then to manage, I used the following tools:

- ❖ Microsoft excel with solver option.
- ❖ Morphologi software



RESULTS

4. RESULTS

4.1. PROCESS OF SAMPLE SELECTION

First, we take a very large sample stones of the same material. Fallin observe their behavior in a water-filled tube, just to preview it on forms that are disposable from the beginning.

Taking a closer idea about what type of particles are suitable for our experiment, we establish a distance of 80cm in the water-filled tube. This helps us to determine a more specific behavior of particles, said particles having previously separated into seven major groups:

- ❖ Shaped boulders half ellipse
- ❖ Rounded angled
- ❖ Elongated boulders
- ❖ Triangular beaked
- ❖ Porous very angled
- ❖ Angled with rounded edges
- ❖ Angled beaked

After the previous experiment, we selected only peaked angular particles. Thus, having a larger sample of a single group, we can divide these into several subtypes.

Change the water by glycerol. Observe two subtypes that are good candidates for the experiment. Angled flat square particles and particles with high convexity.

PARTICLE	1	2	3	4	5	6
WEIGHT	0,4500	0,3413	0,1817	0,1643	0,1250	0,3523
TIME glycerol 85% (s)	9,2	9,4	11,2	12	16	7,2
TEMPERATURE (°C)	20,4	20,4	20,4	20,5	20,6	20,5

PARTICLE	7	8	9	10	11	12
WEIGHT	0,1024	0,1534	0,1192	0,0782	0,1176	0,0635
TIME glycerol 85% (s)	15,2	14,8	14,6	15,2	13,8	15,2
TEMPERATURE (°C)	20,5	20,4	20,4	20,4	20,4	20,4

After obtaining these results, we empty part of glycerol that is in the pipe installation, and fill with water to lower the concentration. Thus repeat the experiment with the same particles but at a lower concentration.

PARTICLE	1	2	3	4	5	6
WEIGHT	0,4500	0,3413	0,1817	0,1643	0,1250	0,3523
TIME glycerol 74% (s)	5	5	5,4	5,4	7	3,4
TEMPERATURE (°C)	21,0	21,0	21,1	21,2	21,1	21,2

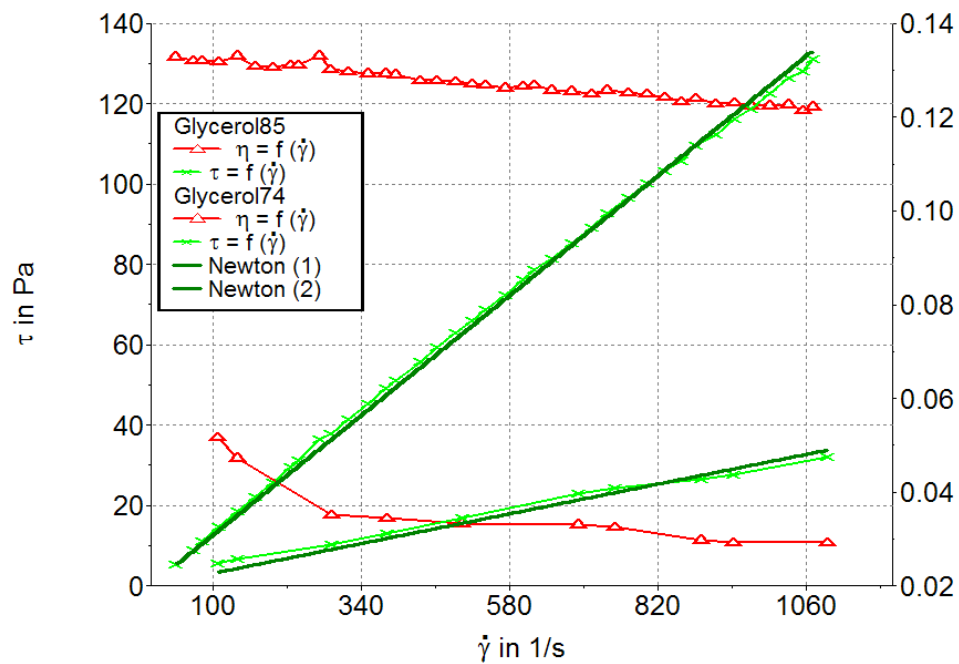
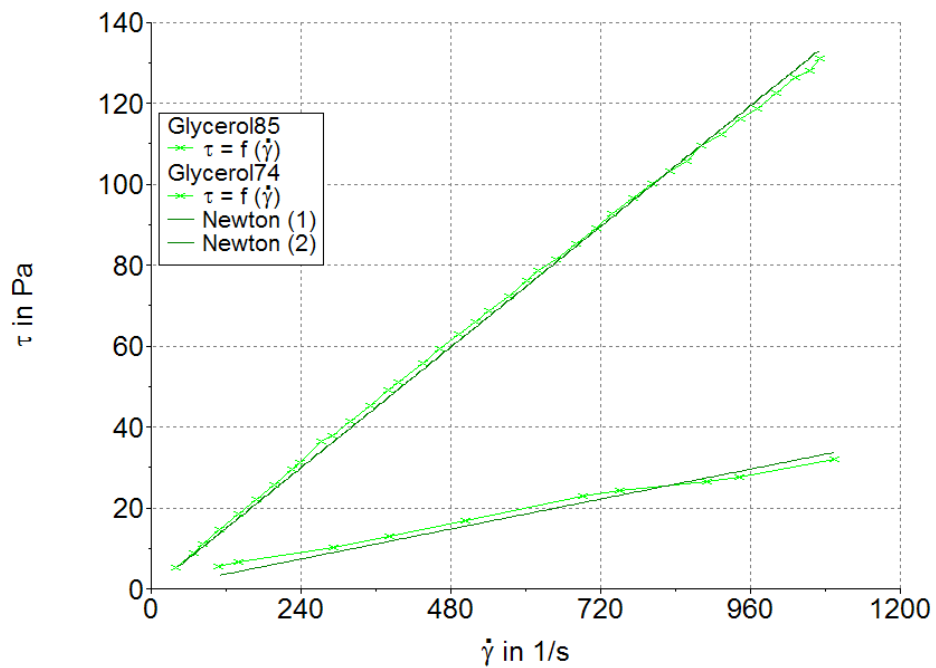
PARTICLE	7	8	9	10	11	12
WEIGHT	0,1024	0,1534	0,1192	0,0782	0,1176	0,0635
TIME glycerol 74% (s)	7,8	6,6	6,6	7,6	6,2	7,8
TEMPERATURE (°C)	21,2	21,1	21,0	21,0	21,0	21,1

4.2. CHARACTERISTICS OF GLYCEROL

Since we know the concentrations of the two solutions of glycerol, the refractometer used to determine the same. This instrument tells us that the concentration of the first solution is 85%, and that the second is 74%.

One thing we also need is the density of both solutions. We use a tensiometer with temperature control. The first solution has a density of 1,227 g / ml (21.5°C) and the second solution has a density of 1,191 g / ml (21.9°C).

To find the viscosity of the two solutions used a rotational viscometer. Which gives us the following results:



With these image data, we observed that the viscosity of glycerol (85%) is from 0.1244 Pa / s, with an uncertainty of 0.9036, whereas the viscosity of glycerol (74%) is from 0.03087 Pa / s with an uncertainty of 8,212.

4.3. DATA PARTICLES

4.3.1. Identification of particles

Because our experiment is repeated in different solutions, we must have a clear identification of all particles. In this way we avoid possible confusion or exchanges. To facilitate this, we photographed all of the test particles:

Particle 1



Particle 2



Particle 3



Particle 4



Particle 5



Particle 6



Particle 7



Particle 8



Particle 9



Particle 10



Particle 11



Particle 12



4.3.2. Microscope data

If we keep in view the identification of the particles, we have to keep in mind that we also have to join the data we get with the microscope with the data obtained in the installation of the experiment.

The microscope provides a picture of the projected area of the particles, but these do not have to take the same position as when the photos were taken. at first this was a little problem, which involved the analysis of microscope repeat a few times. Finally, we realized that between microscope data were positioning coordinates. Knowing this and placing the particles sufficiently separated, we could identify all particles.

PARTICLE	1	2	3	4	5	6
ASPECT RATIO	0,733	0,985	0,783	0,673	0,767	0,566
AREA (μm^2)	109795184	81014200	41429944	43244192	36205000	52661240
CE DIAMETER (μm)	11823,52	10156,30	7262,94	7420,26	6789,52	8188,43
CIRCULARITY	0,856	0,940	0,885	0,876	0,923	0,865
CONVEXITY	0,969	0,974	0,957	0,940	0,957	0,937
ELONGATION	0,267	0,015	0,217	0,327	0,233	0,434
LENGTH (μm)	15455,19	11119,92	9174,82	8932,92	7790,47	10306,12
WIDTH (μm)	11328,79	10949,24	7342,75	6010,44	5975,44	5837,15

PARTICLE	7	8	9	10	11	12
ASPECT RATIO	0,764	0,85	0,847	0,897	0,688	0,803
AREA (μm^2)	36258428	47037212	37859352	28348954	31861000	24586344
CE DIAMETER (μm)	6794,53	7738,84	6942,91	6007,91	6369,20	5595,02
CIRCULARITY	0,913	0,892	0,879	0,879	0,917	0,944
CONVEXITY	0,964	0,961	0,918	0,963	0,977	0,975
ELONGATION	0,236	0,150	0,153	0,103	0,312	0,197
LENGTH (μm)	7842,11	9144,03	7652,40	6746,83	7854,65	6237,67
WIDTH (μm)	5994,54	7772,63	6480,73	6048,95	5404,58	5009,00

With all these data described above, we only need the density of the particles to find the limit velocity the same by Stokes law.

4.4. Solids density

As I described above, there are different ways to calculate the density of an irregular solid. Which was better suited to our properties, both particle and facilities, was the pycnometer method.

Thus, we perform the procedure in this case. This is weighed the empty pycnometer filled with distilled water, only with solid inside and with solid and flush with water. This procedure could not be performed for particles 1 and 2, due to an inadequate size for the pycnometer we had.

Whole procedure was successful. Still, the results were not correct densities for the solid problem.

First, calculate the density as well is described in the introduction. The results were:

particle	1	2	3	4	5	6
empty pycnometer (g)	–	–	33,842	33,842	33,842	33,842
solid in pycnometer (g)	–	–	34,0247	34,006	33,9662	34,1935
solid+water pycnometer (g)	–	–	60,2043	60,2363	60,1025	60,276
full water pycnometer (g)	–	–	60,0834	60,0834	60,0834	60,0834
solid weight (g)	–	–	0,1827	0,164	0,1242	0,3515
water weight	–	–	26,2414	26,2414	26,2414	26,2414
not displaced water mass	–	–	26,1796	26,2303	26,1363	26,0825
density 1	–	–	2,9563	14,7748	1,1817	2,2121

particle	7	8	9	10	11	12
empty pycnometer (g)	33,842	33,842	33,842	33,842	33,842	33,842
solid in pycnometer (g)	33,9442	33,9951	33,9616	33,9214	33,9595	33,9053
solid+water pycnometer (g)	60,1703	60,1521	60,0987	60,1541	60,1796	60,107
full water pycnometer (g)	60,0834	60,0834	60,0834	60,0834	60,0834	60,0834
solid weight (g)	0,1022	0,1531	0,1196	0,0794	0,1175	0,0633
water weight	26,2414	26,2414	26,2414	26,2414	26,2414	26,2414
not displaced water mass	26,2261	26,157	26,1371	26,2327	26,2201	26,2017
density 1	6,6797	1,8140	1,1467	9,1264	5,5164	1,5945

If we believe it is the same material, there are many variations on the same; there can be some differences so great between densities and other. We also know that this solid density is between 2.5 and 3.3 g/cm³.

After repeatedly reviewing the calculations, and check that there is no miscalculation, decided to try another method of calculation for the same pycnometer.

$$\rho = \frac{\text{solid + water pycnometer (g)}}{\text{full water pycnometer (g)}}$$

particle	1	2	3	4	5	6
density 2	–	–	1,0020122	1,0025448	1,00031789	1,00320554

particle	7	8	9	10	11	12
density 2	1,0014463	1,00114341	1,00025465	1,0011767	1,00160111	1,00039279

Since these results are not correct, the discard. This leads us to think that was the problem, if at first it seemed that was the best method.

Considering such disparate results obtained with the same data, we think that maybe the particles have a very low weight for this experiment, and on the other hand, having a representative sample so small it does not help to find a better way the problem.

As the objective of our work is to find out the speed limit of particles in our sample, we assume an average density for all particles, so that we can get an approximation of that velocity.

4.5. Stokes law

Stokes' Law relates to the frictional force experienced by the spherical objects moving in a viscous fluid within a laminar flow of low Reynolds numbers.

If the particles are falling vertically into a viscous fluid due to its own weight can be calculated sedimentation velocity equaling or fall of frictional force with the apparent weight of the particle in the fluid.

$$V_s = \frac{2}{9} \frac{r^2 g (\rho_p - \rho_f)}{\eta}$$

- ❖ V_s is the fall velocity of the particles (speed limit)
- ❖ g is the acceleration of gravity,
- ❖ ρ_p is the density of the particles and
- ❖ ρ_f is the density of the fluid.
- ❖ η is the fluid viscosity.
- ❖ r is the radius of the particle equivalent.

As density data for all the particles take 2.9 g/cm³ because it is an average value of the expected range.

Attention must be given in the units when applying equation.

These are the results of the speeds for the 85% glycerol:

PARTICLE	1	2	3	4	5	6
Equivalent Radius	5,912E-03	5,078E-03	3,631E-03	3,710E-03	3,395E-03	4,094E-03
Densiti of fluid (kg/m3)	1227	1227	1227	1227	1227	1227
Densiti of solid (kg/m3)	2900	2900	2900	2900	2900	2900
Fluid viscosity (kg/m.s)	0,1244	0,1244	0,1244	0,1244	0,1244	0,1244
Speed limit (m/s)	1,0236	0,7553	0,3862	0,4032	0,3375	0,4909

PARTICLE	7	8	9	10	11	12
Equivalent Radius	3,397E-03	3,869E-03	3,471E-03	3,004E-03	3,185E-03	2,798E-03
Densiti of fluid (kg/m3)	1227	1227	1227	1227	1227	1227
Densiti of solid (kg/m3)	2900	2900	2900	2900	2900	2900
Fluid viscosity (kg/m.s)	0,1244	0,1244	0,1244	0,1244	0,1244	0,1244
Speed limit (m/s)	0,3380	0,4385	0,3529	0,2643	0,2970	0,2292

These are the results of the speeds for the 74% glycerol:

PARTICLE	1	2	3	4	5	6
Equivalent Radius	5,912E-03	5,078E-03	3,631E-03	3,710E-03	3,395E-03	4,094E-03
Densiti of fluid (kg/m3)	1191	1191	1191	1191	1191	1191
Densiti of solid (kg/m3)	2900	2900	2900	2900	2900	2900
Fluid viscosity (kg/m.s)	0,03087	0,03087	0,03087	0,03087	0,03087	0,03087
Speed limit (m/s)	4,2136	3,1091	1,5900	1,6596	1,3894	2,0210

PARTICLE	7	8	9	10	11	12
Equivalent Radius	3,397E-03	3,869E-03	3,471E-03	3,004E-03	3,185E-03	2,798E-03
Densiti of fluid (kg/m3)	1191	1191	1191	1191	1191	1191
Densiti of solid (kg/m3)	2900	2900	2900	2900	2900	2900
Fluid viscosity (kg/m.s)	0,03087	0,03087	0,03087	0,03087	0,03087	0,03087
Speed limit (m/s)	1,3915	1,8051	1,4529	1,0879	1,2227	0,9435

4.6. Equivalent diameters

With the data I could get, considering the difficulties we have had with the volume of the particles and thus the density, I can only calculate two equivalent diameters, projected area diameter and Stokes diameter.

Projected area diameter
$$d_A = \sqrt{\frac{4 \cdot A}{\pi}}$$

Particle	Area (cm ²)	d _a
1	1,0980	1,1824
2	0,8101	1,0156
3	0,4143	0,7263
4	0,4324	0,7420
5	0,3621	0,6790
6	0,5266	0,8188
7	0,3626	0,6795
8	0,4704	0,7739
9	0,3786	0,6943
10	0,2835	0,6008
11	0,3186	0,6369
12	0,2459	0,5595

Stokes diameter
$$d_{ST} = \sqrt{\frac{18 \cdot V_T \cdot \mu}{(\rho_p - \rho_f) \cdot g}}$$

The results of the 85% solution are:

PARTICLE	Stokes Diameter cm
1	1,182352
2	1,01563
3	0,726294
4	0,742026
5	0,678952
6	0,818843
7	0,679453
8	0,773884
9	0,694291
10	0,600791
11	0,63692
12	0,559502

The results of the 74% solution are:

PARTICLE	Stokes diameter cm
1	1,182352
2	1,01563
3	0,726294
4	0,742026
5	0,678952
6	0,818843
7	0,679453
8	0,773884
9	0,694291
10	0,600791
11	0,63692
12	0,559502

As might be expected, the results are the same in both solutions, indicating that other steps taken, as the fluid densities, viscosities, etc. are correct.



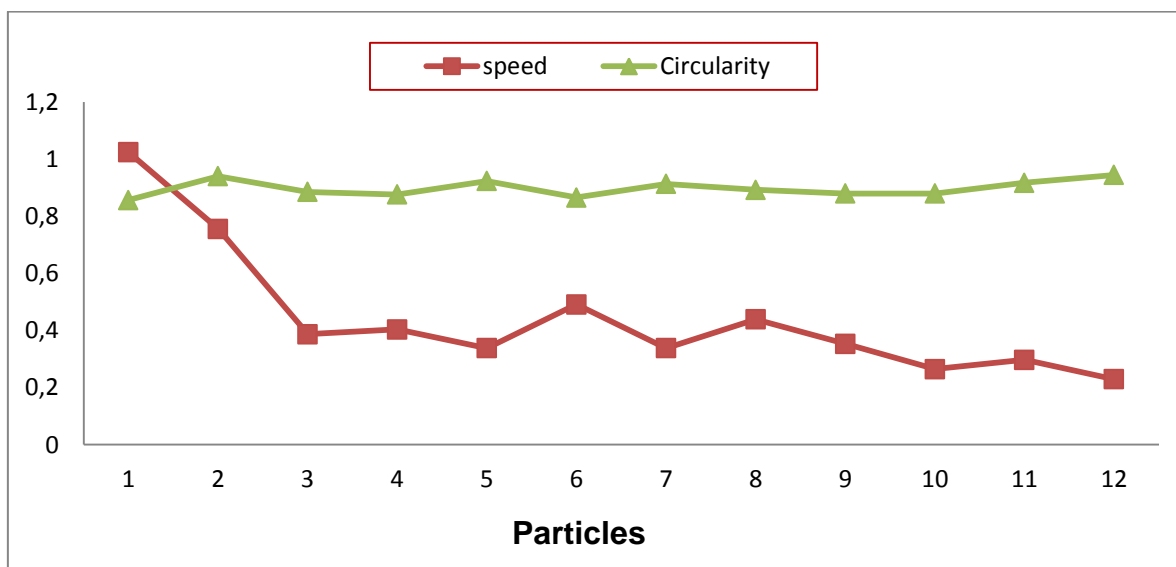
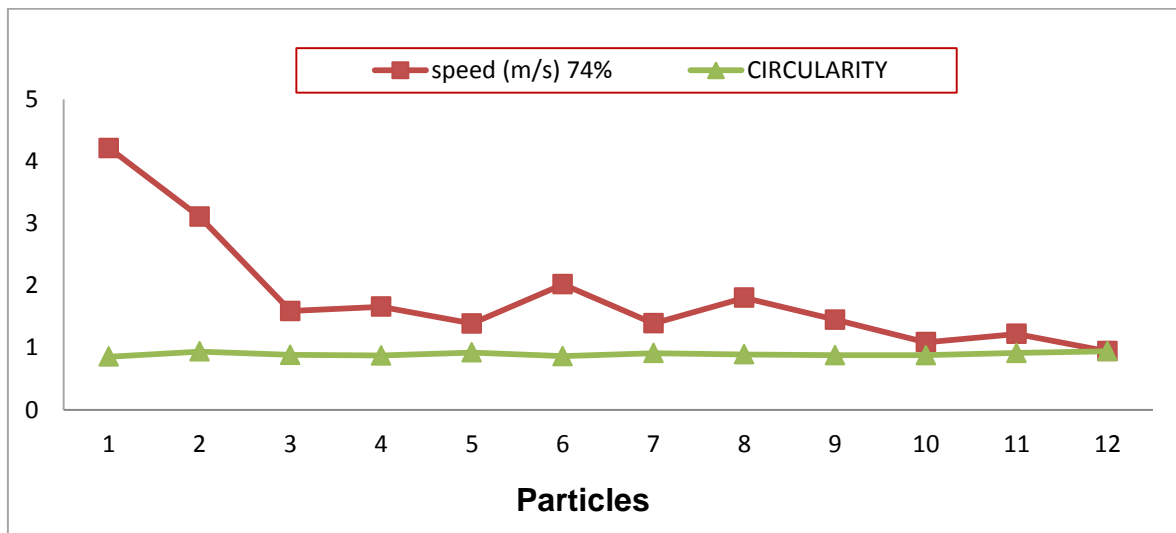
CONCLUSION

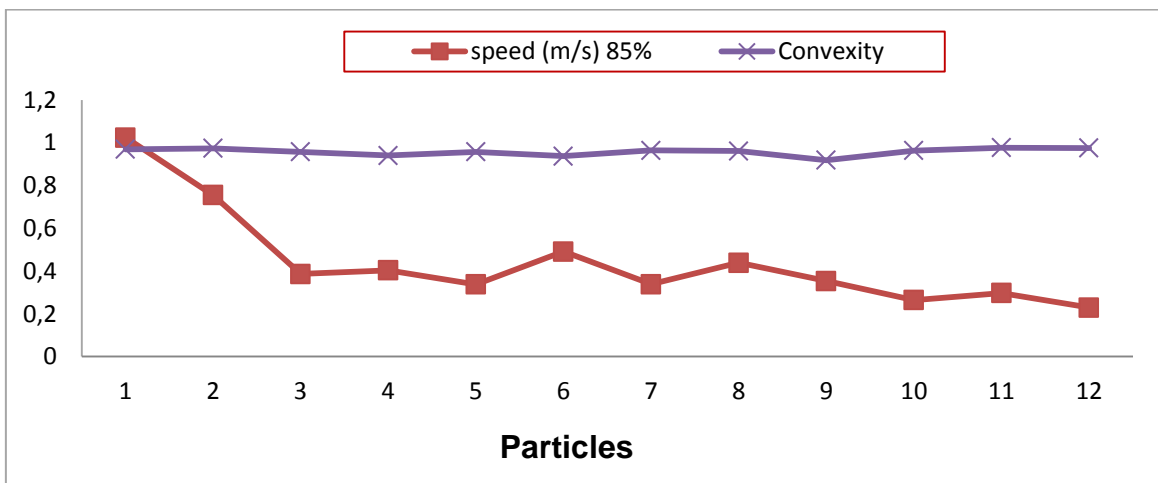
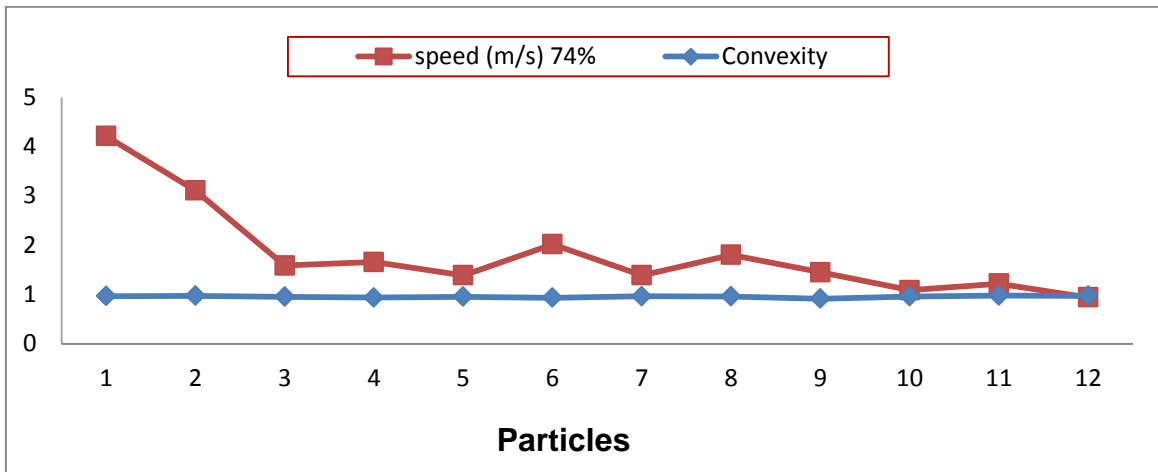
5. CONCLUSION

First, we must mention the importance of checking the data you obtain while you are performing the experiment; this would facilitate avoiding problems in the final calculations.

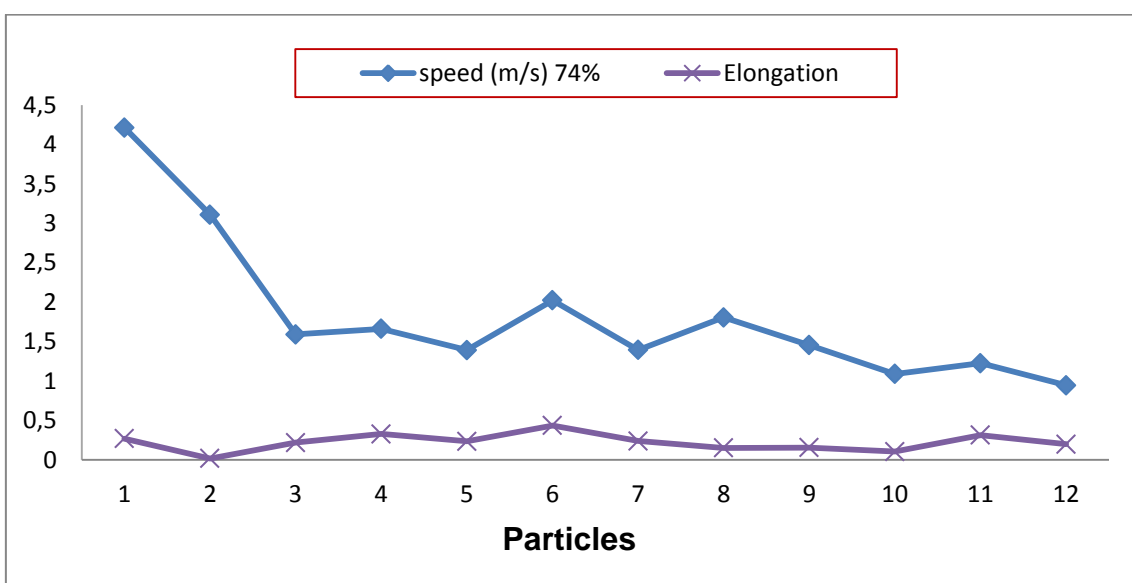
Moreover, we can relate the speed limit of particles with equivalent diameter. One might think that a larger equivalent diameter, longer, due to the higher friction surface, and consequently lower speed. This is not what the data is translated. In fact, we see a significant relationship between these data, ie, the higher the observed equivalent diameter greater speed.

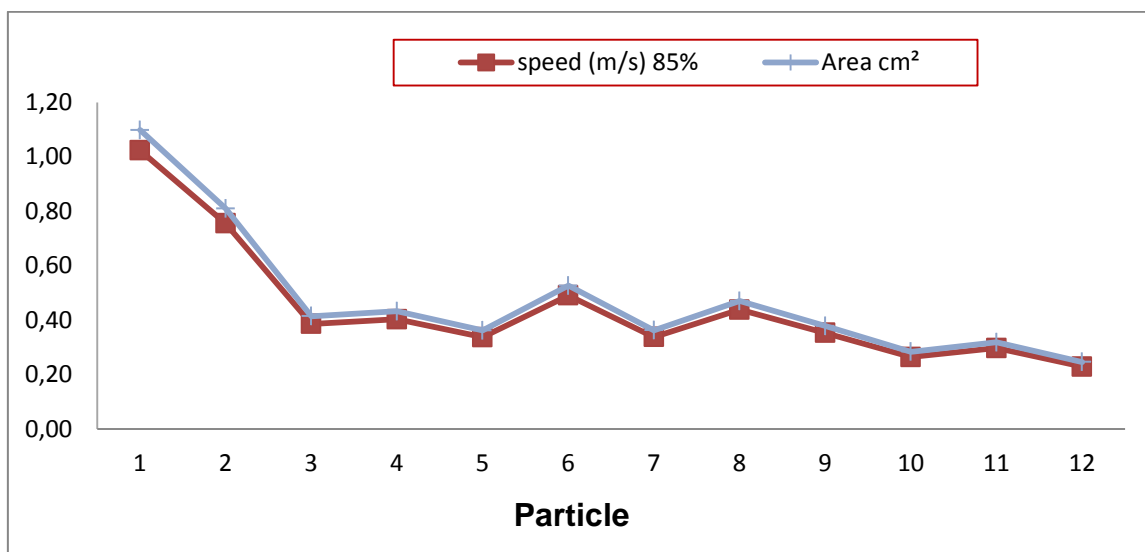
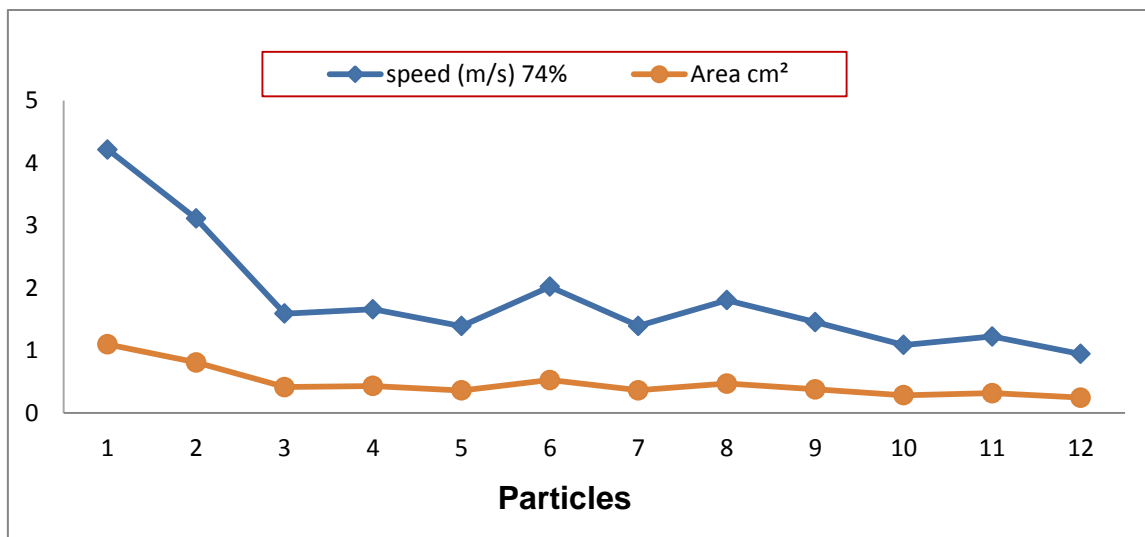
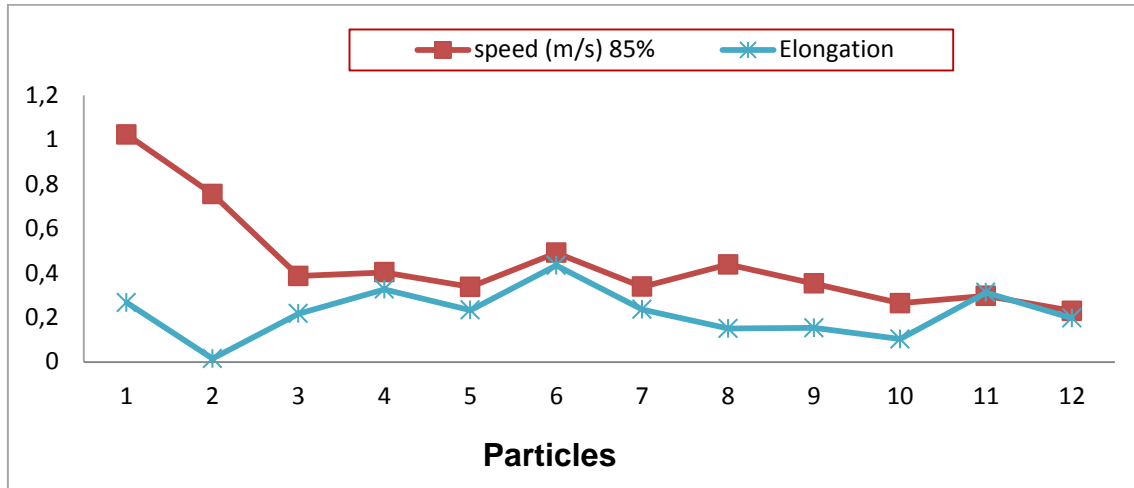
Here we show some characteristics of the stones that have no apparent relation to the speed of the particles themselves.

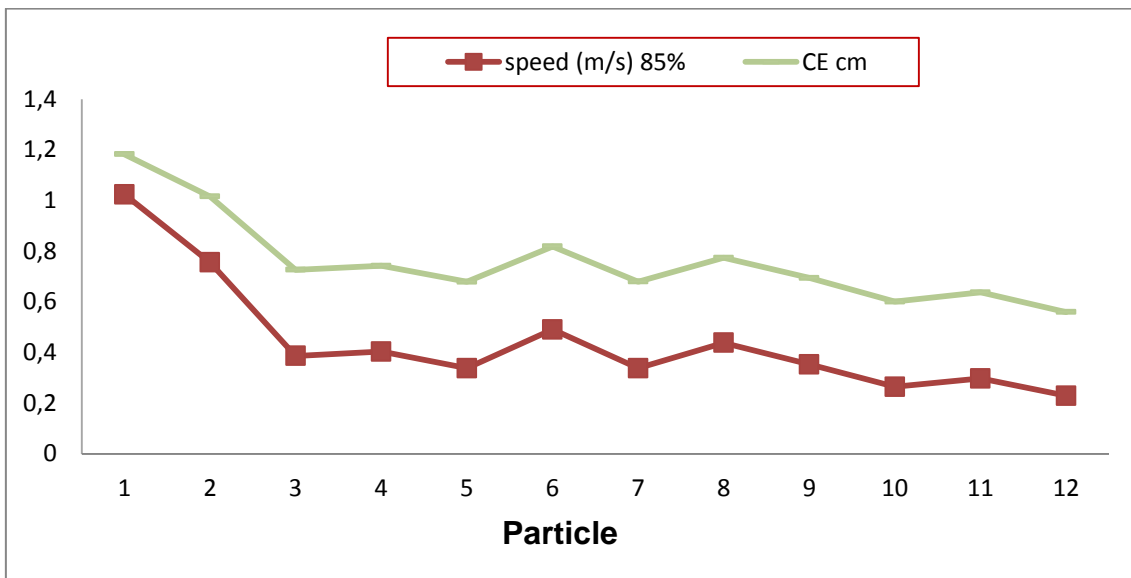
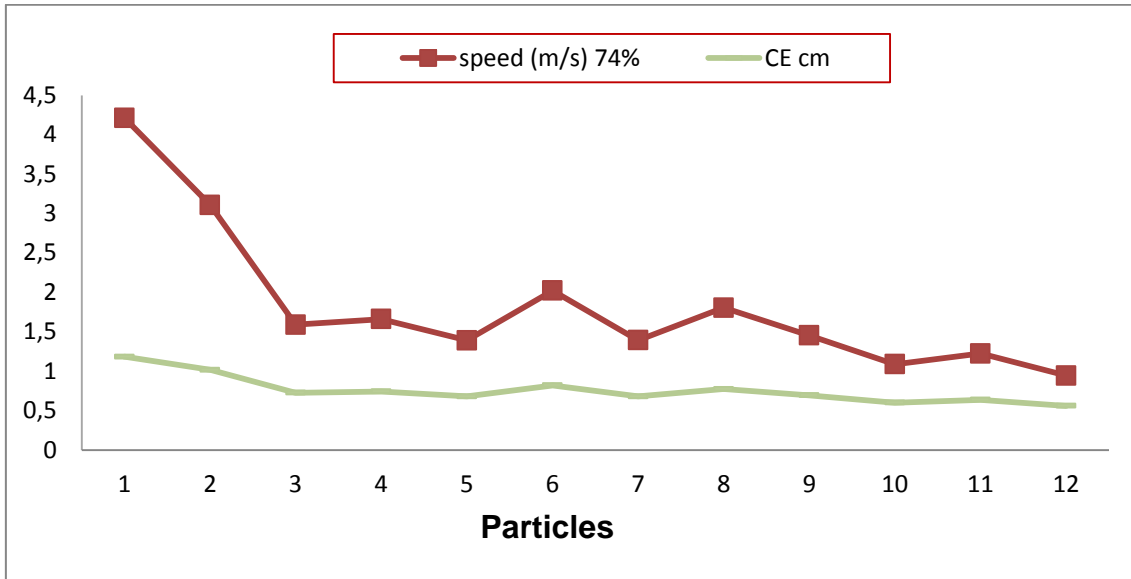




The following is some relationship between the features shown and speed.









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6. BIBLIOGRAPHY

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- ❖ Elaboraron: I.Q.I Minerva Juárez Juárez, Dr. Gustavo Valencia del Toro,
M. en C. Juan Ramírez Balderas, I.Q.I. Linaloe Lobato Azuceno, I.B.Q.
Emma Bolaños Valerio, I.B.Q. Pedro Miranda Reyes
<http://www.biblioteca.upibi.ipn.mx> (Date of reference: 5th January)