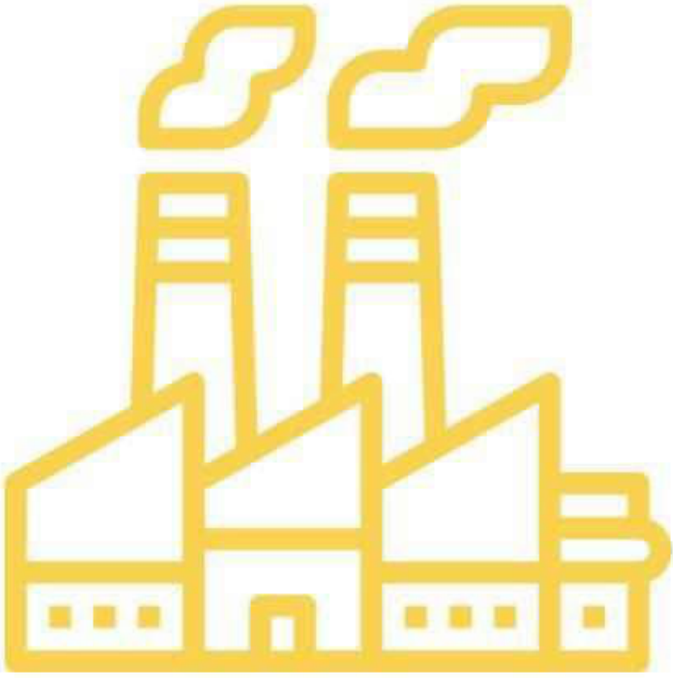


MIRACLE Academy

صناعية 1

زميلتكم رانيا سميح

لجان الرفعات

قال تعالى (يَرْفَعِ اللَّهُ الَّذِينَ آمَنُوا مِنكُمْ وَالَّذِينَ أُوتُوا الْعِلْمَ دَرَجَاتٍ)

Description of particle size

انواع الـ dist.

قريبة من الـ mean

Types of distributions

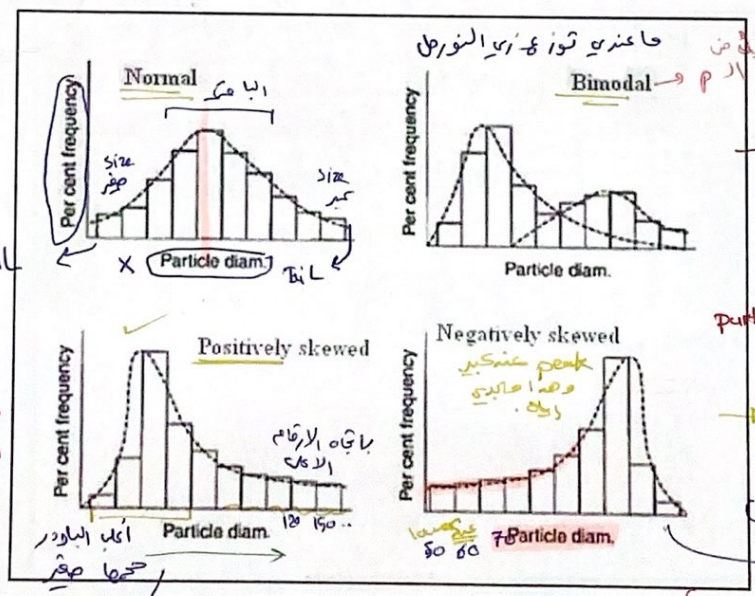
- ***Normal distribution***: The mode separates the curve into two symmetrical halves.
- ***Positively skewed***: A frequency curve with an elongated tail towards the higher size range.

له ذيل باتجاه الرقم الأكبر

- ***Negatively skewed***: A frequency curve with an elongated tail towards the lower size range.

له ذيل باتجاه الرقم الأصغر

- ***Bimodal***: The frequency curve containing two peaks (two modes)



Tail

من هنا هنأخذ الرسمة اللي جاية بعد أعرف الحاجة

IQCS ①

Span ②

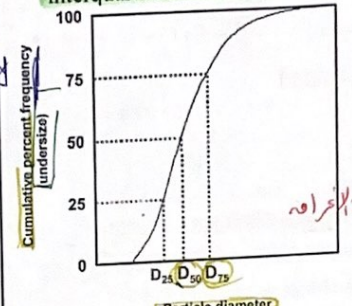
D_{50} اللي جنب median ③

Presentation of size distribution

Evaluation of degree of skewness

درجة الانحراف

- The degree of skewness can be estimated by determining interquartile coefficient of skewness (*IQCS*)



$$IQCS = \frac{(D_{75} - D_{50}) - (D_{50} - D_{25})}{(D_{75} - D_{50}) + (D_{50} - D_{25})}$$

upper — median — lower

الجواب هو درجة الانحراف

Cumulative frequency distribution curves. Point D_{50} corresponds to the median diameter; D_{25} is the lower quartile point and D_{75} is the upper quartile point.

IQCS هي أحسن طريقة بتكون عندكم بباقي

cumulative frequency — p.diameter

IQCS

-1 negatively skewed — 0 normal — +1 positively skewed

* D_{50} يعني 50٪ من Particle size أقل منه 50

هل البيانات سالكة من skewed أو لا ؟ سؤال امتحان

-0.5 — -0.5 (أو مابين)

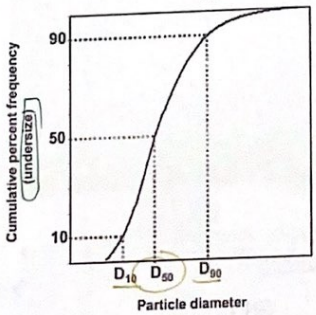
وإذا رقم طبيعي ← اعلم أنها skewed .

Presentation of size distribution

Evaluation of distribution width

مدى

- The size distribution width can be estimated by determining Span



$$Span = \frac{D_{90} - D_{10}}{D_{50}}$$

size distribution

- **Note**: D_{90}, D_{50}, D_{10} are values corresponding to 90, 50 and 10% in the cumulative undersize curve.

* هو نبتسم wide narrow.

* هو نبدي نعرف (p.d.) x-axis عشان استخدمها بالـ cumulative frequency 90٪ 50٪ 10٪

عشان لقدش عندك فروقات باحجام الـ p بالعينة كل ما يكون الـ curve عريض أكثر كل ما كان الاختلاف باحجام الـ p أكثر.

أما لو كان narrow يكون الفرق قليل

* ليش الـ width ؟ لأنه بأكثر حالات التوضيح ممكن احتاج نيمرة يكون عندي مجموعة أحجام من الـ curve الواحد وممكن نيمرة أكون محتاج الـ p يكونوا اقرب ما يمكن لبعض شي الاثنين

* ممكن يكون الرسمتين نوعال لكن width (العرض) لهم مختلفين

narrow size — كل ما كان span صغير أذا

~~span~~ — كل ما تو سع Different size

واضح بقى

mean ← متوسط أحجام الـ p

mode ← أكثر p موجودة بالـ Freq

midian ← الـ p تقسم الـ curve لنصين

M = M = M ← بالـ normal تكون

اختصار P: Particle

اشو الميزة المستخدمة كرفة حجم الـ p ؟

Particle size analysis methods

Microscope methods

Direct → اكرم مباشرة

Equivalent diameters بقياس

InDirect → ما اوخذ القيمة من معادلة

d_a, d_p, d_F and d_M can be determined

Range of analysis

- Light microscope (1 - 1000 μm)
- Scanning electron microscope (0.05 - 1000 μm)
- Transmission electron microscope (0.001 - 0.05 μm)

انواع مايكروسكوب !

الاكثر بيز لحيد

ds dst dv

SEM. TEM

اوقة واصغر

surface ما بيعطي ديتيلس من characteristic.

حسب الحجم

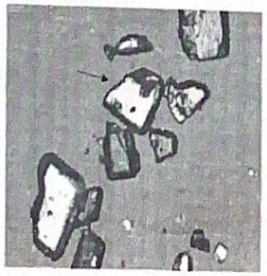


Image by light microscope

2D صورة

Image by scanning electron microscope

3D لشوفها

TEM

smooth ←

اذا بدنا شوف الشي smooth شو فيها تحققان ؟

ذهب Au →

صيرة المايكروسكوب بصور ويكتفط بالصورة

لازم يكون فيها ذهب تكلفها اكثر

Direct مباشرة بعطي العينة

Particle size analysis methods

1. **Microscope methods**

Sample preparation

Techniques

- manual
- Semiautomatic
  - Particle comparator
  - Image shearing eyepiece (double prism arrangement)
- Automatic (video camira)
  - A video camera is used to transform the image to a microprocessor where manipulations and calculations are done

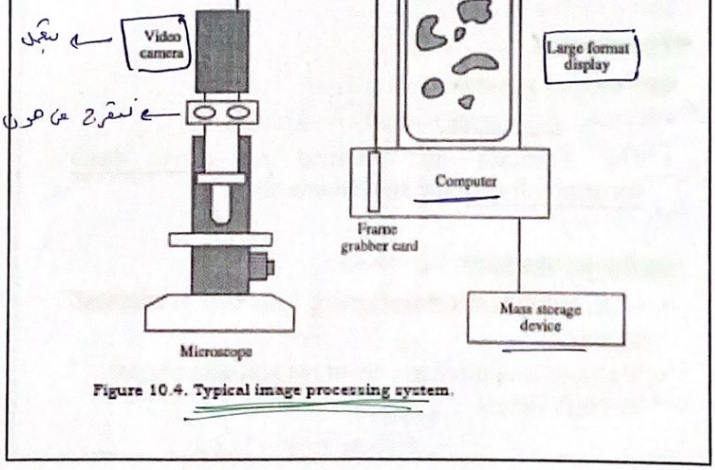


Figure 10.4. Typical image processing system.

Particle size analysis methods

2 Sieve methods

Equivalent diameter

Sieve diameter (d_S)

Range of analysis

Available range: (5 - 125 000 µm)

ISO range: (45 - 1000 µm)

Sample preparation

Dry sieving: for non cohesive powders

Wet sieving: for suspensions and cohesive powders

Particle size analysis methods

Sieve methods

Techniques

1) Vibrated sieving:

- Uses a sieve stack (usually 6 –8 sieves)
- The Particles are retained on sieve mesh corresponding to the sieve diameter.

2) Air-jet sieving:

- Uses individual sieves starting from that of smallest aperture.
- Vacuum is applied to encourage particles to pass through sieves.

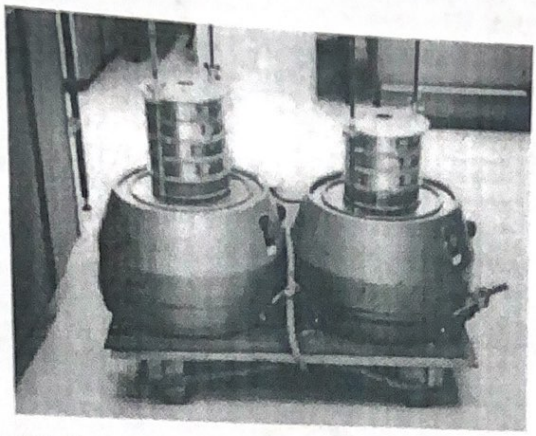


Figure 3.2. Vibratory sieve shakers in action.

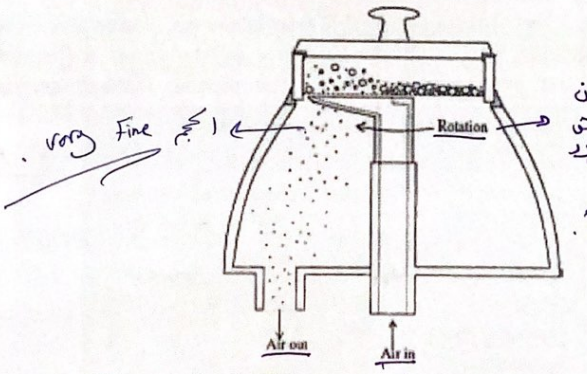
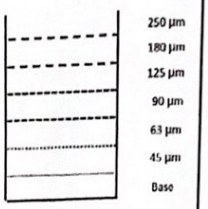
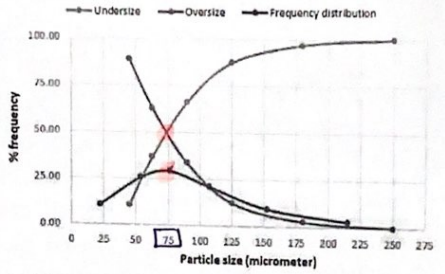


Figure 3.4. The airjet sieve.

(1) Sieve size range (μm)	(2) mean of size range	(3) Sieve fractions		(4) Nominal aperture size (μm)	(5) % Cumulative undersize	(6) % Cumulative oversize
		wt (g)	wt%			
>250		0.02	0.04	250	99.96	0.04
180-250	215	1.32	2.96	180	96.99	3.01
125-180	152.5	4.23	9.50	125	87.49	12.51
90-125	107.5	9.44	21.19	90	66.30	33.70
63-90	76.5	13.1	29.41	63	36.89	63.11
45-63	54	11.56	25.95	45	10.93	89.07
<45	22.5	4.87	10.93	0	0	100
		Sum=44.54				





75 median.
possibly coarse.

Standards for powders based on sieving

- Standards for pharmaceutical powders are provided in pharmacopoeiae, which indicate the degree of coarseness or fineness depending on percentage passing or not passing through certain sieves.
- Ex. BP

Table 12.1 Powder grades specified in British Pharmacopoeia

Description of grade of powder	Coarsest sieve diameter (μm)	Sieve diameter through which no more than 40% of powder must pass (μm)
Coarse	1700	355
Moderately coarse	710	250
Moderately fine	355	180
Fine	180	—
Very fine	125	—

1700
40%
355

50mm ← وزن وزنه فيها الـ d أكبر من 10٪

5mm ← أقل من 90٪

اذا بطلقوا ultra. fine

Standards for powders based on sieving

- Some Pharmacopoeiae define another size fraction, known as 'ultrafine powder'. كثير ناقص
- In this case it is required that the maximum diameter of at least 90% of the particles must be no greater than 5 µm and that none of the particles should have diameters greater than 50 µm.

50µm حاجز بيمنعه p.2 لازم *

5µm p.2 لازم يكون 90٪ على الأقل *

ultra powder لحتى نسميها

≤ 90٪ 5 µm

من

sample كل

0٪

~~50 µm~~ نكون

(*) all the particles

50 µm أقل من

Particle size analysis methods

(3) **Electric stream sensing zone method (Coulter counter)** ضد كهربائية

Equivalent diameter:

Volume diameter (d_v)

Range of analysis

(0.1 - 1000 µm)

Particle size analysis methods

Electric stream sensing zone method (Coulter counter)

Principle of measurement

- Powder samples are dispersed in an electrolyte solution to form a very dilute suspension.
- The particle suspension is drawn through an orifice where electrodes are situated on either side and surrounded by electrolyte solution.
- As the particle travels through the orifice, it displaces its own volume of electrolyte solution.
- The change in electrical resistance between the electrodes is proportional to the volume of the particle (volume of electrolyte displaced)

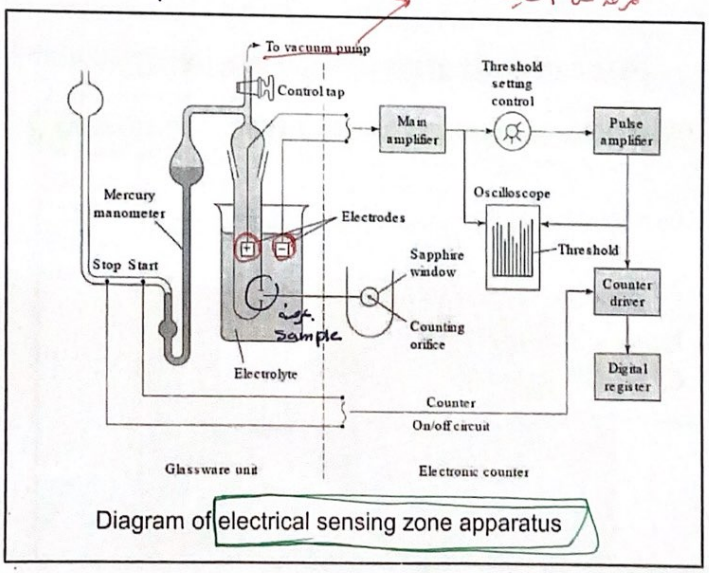


Diagram of electrical sensing zone apparatus

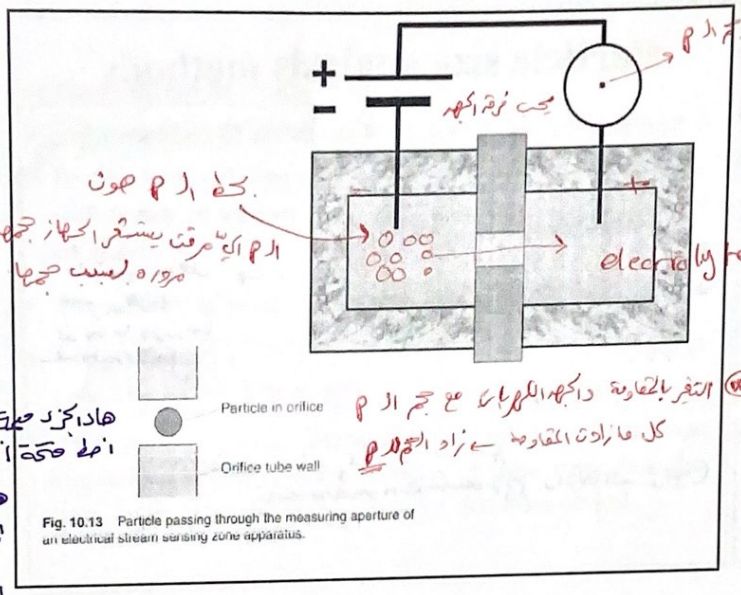


Fig. 10.13 Particle passing through the measuring aperture of an electrical stream sensing zone apparatus.

Particle size analysis methods

4 Sedimentation methods

Equivalent diameter: Stokes diameter (d_{st})

- Stokes equation:

$$d_{st} = \sqrt{\frac{18\eta h}{(\rho_s - \rho_f) g t}}$$

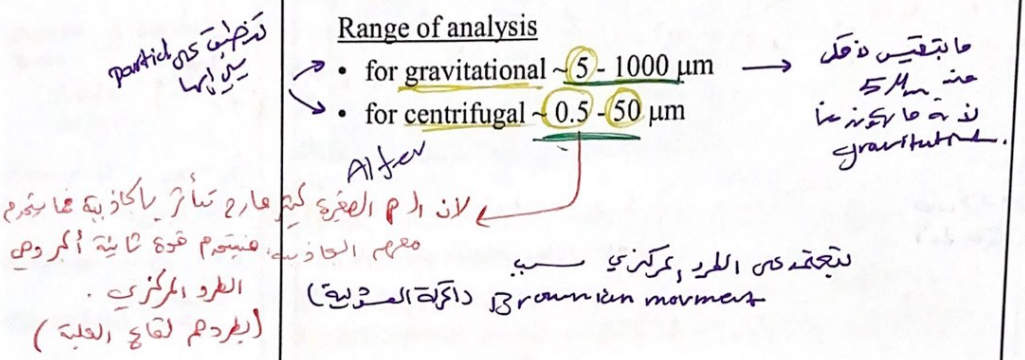
- d_{st} = Stokes diameter, η = viscosity of fluid, h = height or sedimentation distance, ρ_s = density of solid, ρ_f = density of fluid, g = the acceleration due to gravity, t = time

Particle size analysis methods

Sedimentation methods

Range of analysis

- for gravitational ~ 5 - 1000 μm
- for centrifugal ~ 0.5 - 50 μm



Particle size analysis methods

Sedimentation methods

Principles of measurement

- Particle size distribution can be determined by examining the powder as it sediments out.
- The powder is dispersed uniformly or introduced as a thin layer in a fluid.
- Techniques can be divided into two main categories.

Sedimentation methods

Pipette method (Andreasen pipette)

- In this method known volumes of the suspension are withdrawn, at various time intervals, from bottom (lower set limit).
- The amount of solid is determined in each volume.
- The particle diameter corresponding to each time period is calculated from Stokes' law.
- The amount of solid determined for each time interval is the weight fraction having particles of sizes more than the size obtained by the Stokes' law for that time period.

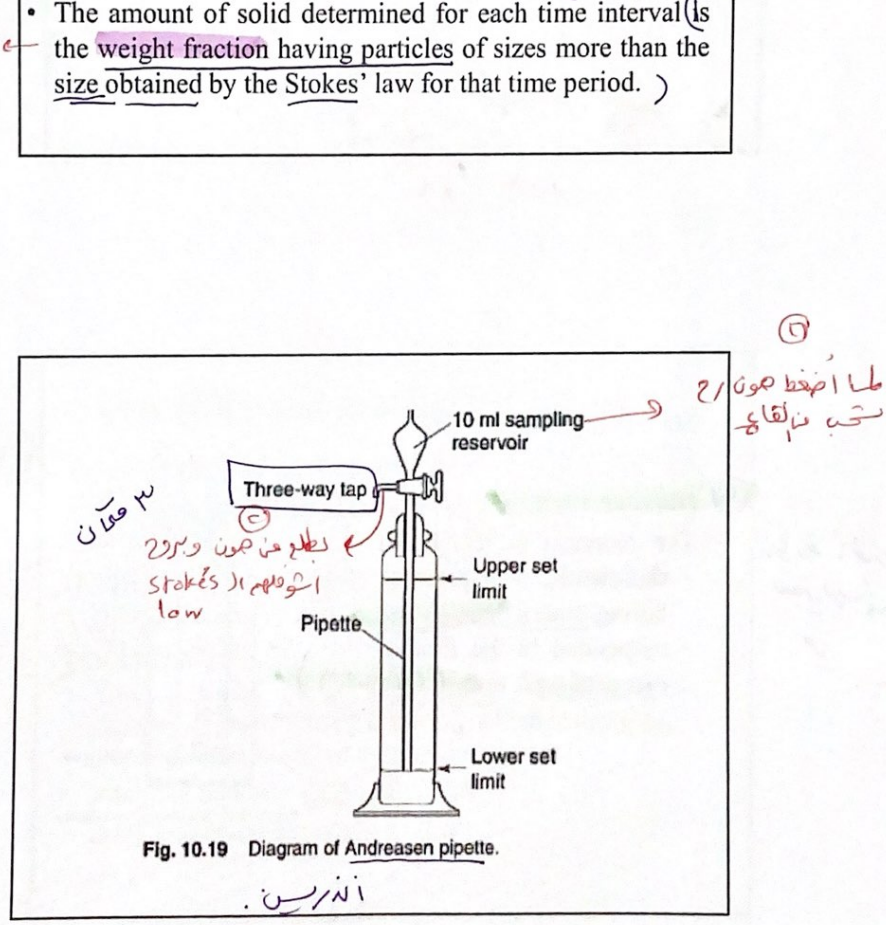


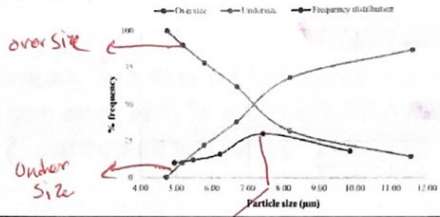
Fig. 10.19 Diagram of Andreasen pipette.

لو اجاني سؤال صيك فأنا هعين كل المعطيات بطريقة مباشرة على القانون مع أخذ أي وقت .

- A suspension of 5 g of ZnO_2, density 5.60 g/cm^3, in 50 ml of water was prepared containing 2.75 g sodium citrate as deflocculating agent was transferred to Andreasen pipette (h = 20 cm) and volume made up to 550 ml using distilled water. The suspension was shaken and allowed to settle under the acceleration of gravity, 981 cm/sec^2, at 25 °C. the density of the medium is 1.01 g/cm^3, and its viscosity is 1 centipoise = 0.01 poise or 0.01 g/cm sec.

الوقت اللي استنيته حتى تاخد العينة

Time (sec)	Particle size (µm)	Size range (µm)	Mean of size range (µm)	wt of sample collected (g)	wt (%)	Cumulative undersize (%)	Cumulative Oversize (%)
600	11.54	>11.54		0.7	14	86	14
1200	8.16	8.16-11.54	9.85	0.9	18	68	32
1800	6.66	6.66-8.16	7.41	1.5	30	38	62
2400	5.77	5.77-6.66	6.22	0.8	16	22	78
3000	5.16	5.16-5.77	5.47	0.6	12	10	90
3600	4.71	4.71-5.16	4.94	0.5	10	0	100
				Σ = 5			



oversize
Under Size
mode = 7.5

Sedimentation methods

(b) *Balance method*

The increase in weight of sedimented particles falling onto a balance pan suspended in the fluid is recorded with time. (Gravity)

ميزان

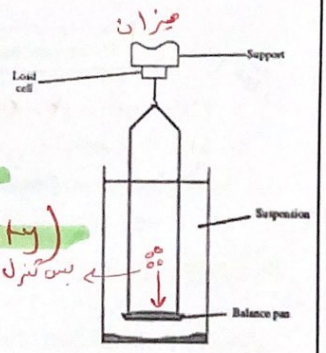


Figure 9.3. Sedimentation balance.

زيادة الوزن بسبب sedimented على الـ Pan .

Sedimentation methods

c) Alternative technique

- Alternative technique is the application of centrifugal sedimentation to make quicker the sedimentation of small particles.

Particle size analysis methods

a) Laser light scattering methods

Equivalent diameters: Area diameter, d_a, volume diameter, d_v.

Principle of measurment: Interaction of laser light with particles

1) Fraunhofer diffraction

- This is based on forward scatter (small angle change) of laser light by particles, which is detected, amplified and analyzed by microprocessor.
- Range of analysis = 0.5 - 1000 µm
- sample is liquid or air suspended

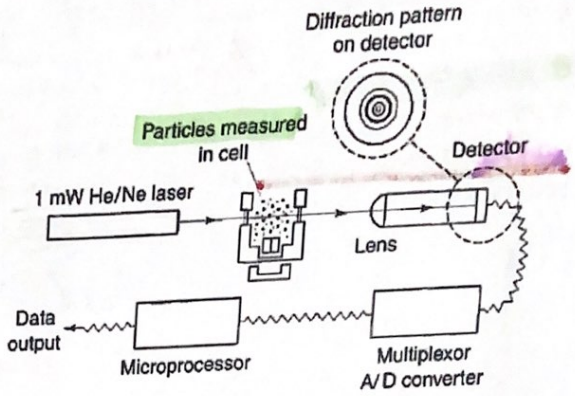


Fig. 10.17 Schematic diagram of laser diffraction pattern particle sizer.

Particle size analysis methods

Laser light scattering methods

2) Photon correlation spectroscopy (PCS)

- It is termed also Dynamic light scattering (DLS)
- This is based on the Brownian movement (random motion of small particles or macromolecules caused by the collisions with the smaller molecules of the suspending fluids) .
- Range of analysis ~ 0.001- 1 μm
- PCS analyses the constantly changing patterns of laser light scattered or diffracted by particles in Brownian movement and monitors the rate
- Calculation of size is based on Stokes-Einstein equation:

$$D = \frac{1.38\times10^{-12}T}{3\pi\eta d} m^2 s^{-1}$$

- T = absolute temperature, d = diameter, η= viscosity of liquid,
- D = Brownian diffusion.

Selection of particle size analysis method

Factors to be taken into consideration:

- Size range of powder
- Amount of sample
  - If sample is very small we can use microscopy but we can not use sieving
- Speed of analysis
- Accuracy of results
- Cost
- Physical nature of material (like Agglomeration and cohesiveness)

Influence of particle shape

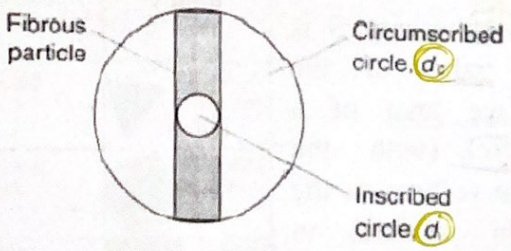


Fig. 10.6 A simple shape factor is shown which can be used to quantify circularity. The ratio of two different diameters (d_i/d_c) is unity for a circle and falls for acicular particles.

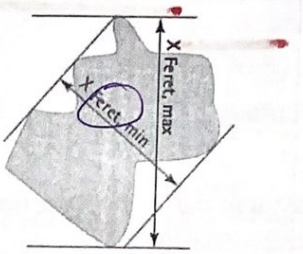
d_i / d_c

تعريف الاستقامة كلما كانت أقرب للواحد
needle تتكون

Particle shape descriptors

Aspect ratio

- The ratio of the minimum to the maximum Feret diameter is another measure for the particle shape.



Particle shape descriptors

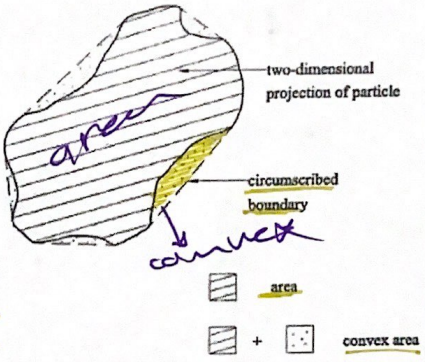
Sphericity

- The sphericity S 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.

Shape	Spherecity
Tetrahedron	0.671
Cube	0.806
Dodeca-hedron	0.910

Particle shape descriptors

Convexity and fullness ratio



$$\text{convexity ratio} = \frac{\text{area}}{\text{convex area}}$$

$$\text{fullness ratio} = \sqrt{\frac{\text{area}}{\text{convex area}}}$$