Particle Characterization: Assignment 1

1. Define a particle.

   Ans: A particle is a part of a whole. It is something that is large enough to be distinguished from a cluster of atoms, but small enough to remain suspended in its environment for an observable period of time.

2. Conventionally, what is the "nano" size-range?

   Ans : 1 to 100 nanometers is conventionally called the ''nano'' size range.

3. Identify the various categories of particle characteristics, with examples.

   Ans : The various categories of particle characteristics with examples are as follows:

   a. Morphological - shape, size
   b. Structural - Crystalline or amorphous, sectional analysis
   c. Compositional - element, compound, polymer, ceramic
   d. Interfacial – solid/solid, solid/liquid, solid/gas, liquid/solid, liquid/liquid, liquid/gas, gas/solid, gas/liquid, gas/gas.
   e. Physico-chemical - density, hardness, roughness
   f. Transport -diffusional, convective, inertial, phoretic
      Storage – adhesion, cohesion
   g. Functional – Toxicity, catalytic effectiveness

4. Outline a statistical method for shape assessment in a particulate assembly.

   Ans: We take the tabulated data and average the Martin's diameters as the function of theta and average all the Feret's diameters and take the ratio of d_{Martin} over d_{Feret} as being the average shape coefficient.

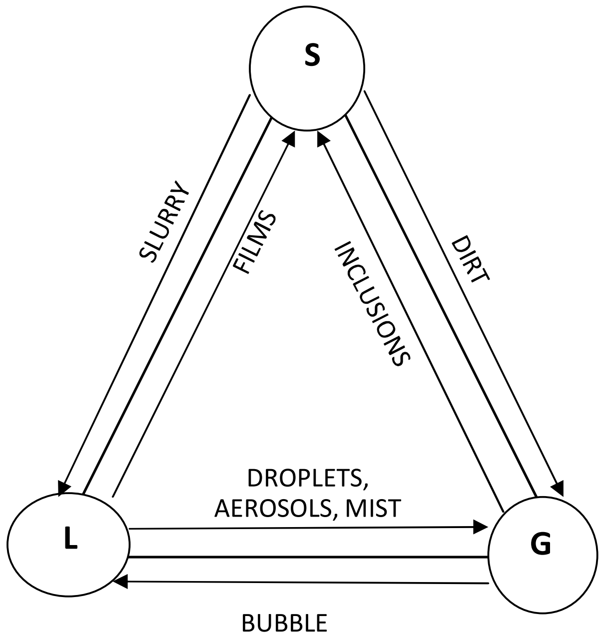
$$\frac{\overline{d_M}}{\overline{d_F}} = \text{Shape coefficient}$$

$$\frac{\sigma_M / \overline{d_M}}{\sigma_F / \overline{d_F}} = \text{Variability}$$

   Variability can be defined by taking the above sigma values and dividing them by the representative mean diameters. A higher variability coefficient indicates a highly irregularly shaped object.

5. Can you have solid particles in solids, liquid particles in liquids, gas particles in gases? Illustrate with examples.

   Ans: Yes, we can have solids in solids, liquids in liquids and gas particles in gases.

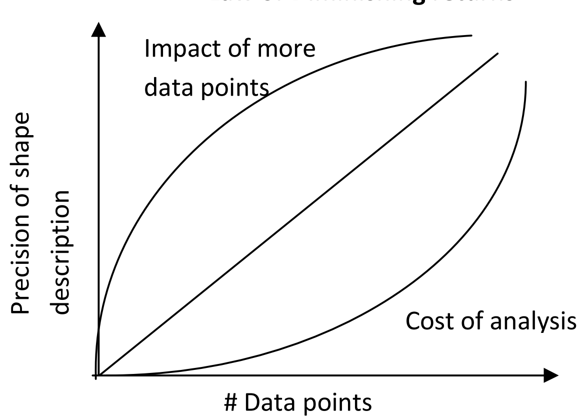


6. Explain the concept of "diminishing return" in the context of shape analysis.

   Ans: When you start with zero knowledge about an object, even looking at it or collecting one or two data points will give you an insight into its shape and will start propelling you towards a logical conclusion about the shape of the particle. Once you go beyond a certain amount, the more data points you collect, it is more for validating your judgment rather than forming your judgment. This is called the law of diminishing returns.

   It basically says that in most situations the relative impact of additional effort or investment gets reduced as you expend more and more effort or energy to make it happen.

Law of Diminishing returns



7. In the mathematical method of shape analysis, is it better to leave after discretization, or proceed to fit the data with a continuous function?

   Ans : Once we have obtained a discretized profile of the particle, we can try to fit a polymer and obtain a continuous representation. However, there is a risk associated with doing that. There is a finding that closer the fit of the polymer to the points that we take, the greater the deviation from the actual profile of the particle. As a particle scientist, we need to make a call as to how much information is sufficient and stop at that.

8. What is an essential requirement for Fourier shape analysis to be effective?

   Ans : To do Fourier shape analysis, we must have a library of reference spectra to which we can compare the Fourier spectrum of the particle. We can then compare the Fourier spectra for the particle against the ones that are in the library. By doing this comparison, we can see where the best fit is and represent the shape of the particle by the corresponding best fit shape in the library.

9. What is the relevance of "graded memberships" for shape analysis?

   Ans: Consider a set of members and non-members of a club. Let us assign members to be 0 and non-members to be 1. This type of binary membership approach can be applied to shape analysis too. A shape which belongs entirely to a reference shape can be assigned 1 and can be assigned 0 otherwise. But the binary approach can seldom be used for shape analysis as there are many shades of shape. So we adopt a graded membership approach. A life member of a club can be assigned an index of 0.9, a senior member can be assigned 0.8, a regular member and student member can be assigned indices of 0.7 and 0.5 and so on. The same can applied to shape analysis. Here we change from binary membership to fuzzy membership. A shape having a membership index of 0.7 means that it corresponds 70% to the reference shape that we are looking at. This method is known as Membership-Roster approach for shape analysis.

10. Highlight the difference between "distance function" and "common property" approaches to shape assessment.

    Ans: In the distance function approach, we take a three dimensional space in which each axis represents a relevant characteristic of the particle (ex. Bulkiness, roughness, rugosity etc.) This is called shape space. We take the object we are trying to assess and find its position in the shape space. From the library we choose two shapes that have characteristics similar to the shape we are trying to assess and find their positions in the shape space. We then find the distances between the chosen shape and the reference shapes. A comparison of the distances gives an idea of how close the candidate shape is to the reference shape.

In the common property approach, we decide the most relevant characteristics of a particle such as bulkiness, sphericity, roughness etc from a shape view point and instead of computing the distance in shape space, we look for commonality. This method looks for absolute coincidence of one or more properties.

11. Differentiate between agglomeration & aggregation.

    Ans: When two particles are loosely joined together, they are called an agglomerate. If the joining force between the particles is substantial and if a force is required to break them apart, they are called an aggregate.

12. Differentiate between impingement & impaction.

    Ans: In impingement, the particles are smaller than particles associated with impaction. Here, the particles that are flowing parallel to the surface can be collected as long as they come in contact with the substrate. It is a contact collection mechanism.

    During impaction, the particle deviates from the flow stream lines and normally impacts the surface and gets collected because of the high sticking coefficient that is associated with high Stokes numbers. So, the impingement collector is a way to collect particles in a slightly finer size range compared to what we can normally collect in a cascading impactor.

13. Contrast bright-field & dark-field microscopy.

    Ans: In bright-field microscopy, the entire surface is illuminated with white light and the defects and particles show up as dark spots.

    In Dark field microscopy, the entire surface is dark while the surface features shine. This is done to distinguish between the various defects on the surface from the particles that are present. Dark-field microscopy helps us resolve surface features with better clarity than bright-field microscopy.

14. Contrast imaging & scanning modes of surface analysis for particle size distribution.

    Ans: In the imaging mode of analysis, we capture an image of the surface by using a camera and then analyse the image using various algorithms.

Scanning mode is real time analysis of the surface. We take the optics and traverse the entire surface with it and the signals are sent back to the instrument. In this method, feedback is instant. However, this works well for smooth and polished surfaces because rough surfaces would interfere with the signal and produce more noise.

15. How does scattering intensity (I) depend on particle diameter and incident wavelength in the Rayleigh and Mie regimes?

Ans:

Rayleigh scattering regime:

I is related to particle size and wavelength in the following manner:

$I \propto 1/\lambda^4$

$I \propto dp^6$

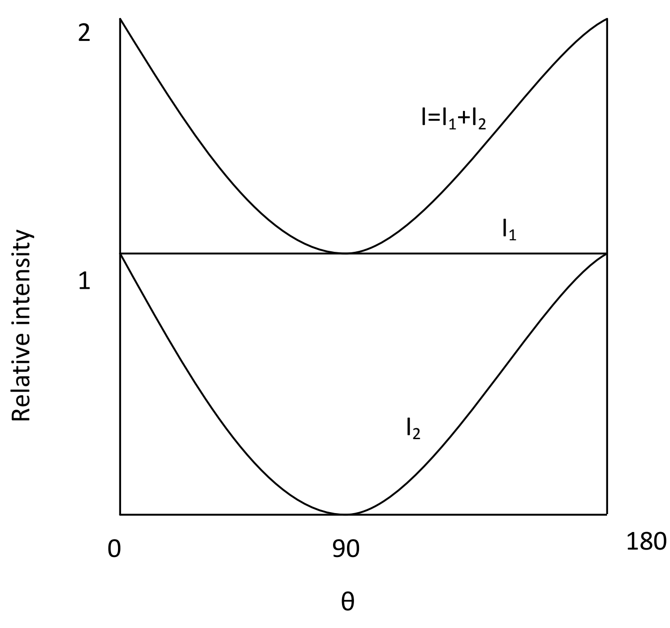
Mie scattering regime :

I is directly proportional to square of the wavelength and independent of particle size.

$I \propto \lambda^2$

16. Sketch I1, I2, I versus scattering angle in the Rayleigh regime.

Ans:



17. How does a “diffusion mobility analyser” help measure sizes in nano range?

    Ans: A diffusion mobility analyser takes advantage of the fact that as particles become smaller, diffusion becomes the primary transport mechanism and rate of diffusion scales inversely to particle size.

    In this method, particles are first sent into a diffusion battery where they are segregated into various size buckets based on their diffusion characteristics. Once this is done, they are sent into condensation nuclei counters(CNC) where the particles of specific size range can be enlarged and their size can be measured.

18. Differentiate between a particle size analyser and a particle counter.

    Ans: A particle size analyser gives the count of particles by size, in various size channels. It also reports differential counts of particles in that channel as well as cumulative counts of all particles, that are larger than a particular size. A particle counter gives the number of particles above a specific size in the chosen sample/clean room/air.

19. Define surface cleanliness “Levels”.

    Ans : Levels are reflective of how clean a surface is. For example, the definition of a level 1000 surface is that it contains 1000 particles that are half a micron or larger per square centimetre.

20. Define fluid cleanliness “Classes”.

    Ans : Just like Levels represent the cleanliness of a surface, Classes reflect the number of particles in ambient environment. For example, a class 1000 clean room will have 1000 particles per cubic foot which are half a micron or larger, and a class 10 clean room will have 10 particles per cubic foot which are half a micron or larger.

21. Give one example where differential particle counts are important.

    Ans: If a product being manufactured is sensitive to particles in a particular size range (ex. 0.5 to 1 micron) then we focus on the differential counts in that size range and differential counts become important.

22. Give one example where cumulative particle counts are important.

    Ans: If a product being manufactured is sensitive to particles that are larger than a particular size (ex. above 0.5 microns) then cumulative particle counts are important because it gives us information about the total number of particles above that size. For example in the manufacture of hard disks, all contaminants above a particular size (say 1 micron) may cause failure. So we look at the cumulative count of particles above 1 micron in size.

23. What particle property does photocorrelation spectroscopy exploit?

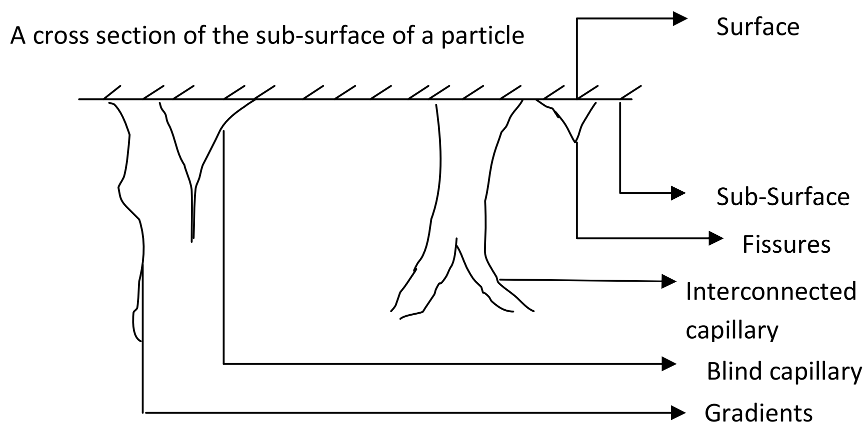
    Ans: Photon correlation spectroscopy or Dynamic light scattering technique makes use of the fact that rate of Brownian diffusion is inversely proportional to particle size. As the size of the particle increases, diffusion becomes slower.

24. Which size is relevant for flowability of a suspension?

    Ans: Agglomerate diameter is the most relevant characteristic to look at the flowability of a suspension.

25. Sketch a typical sub-surface region of a particle.

    Ans:



26. Name some typical gradients from surface to sub-surface.

    Ans: Some of the gradients observed from surface to sub-surface are physical properties, chemical properties (composition), stresses, defects.

27. Derive Langmuir model of adsorption from a dynamic equilibrium viewpoint.

    Ans: Consider a case, where we have species A that is getting adsorbed on the surface.

    Rate of adsorption = Rate of desorption (at dynamic equilibrium)

    $n_A f_s (1-F) = F r_A$

    $n_A f_s - n_A f_s F = F r_A$

$n_A f_s = F(n_A f_s + r_A)$

$F = n_A f_s / (n_A f_s + r_A)$

Where

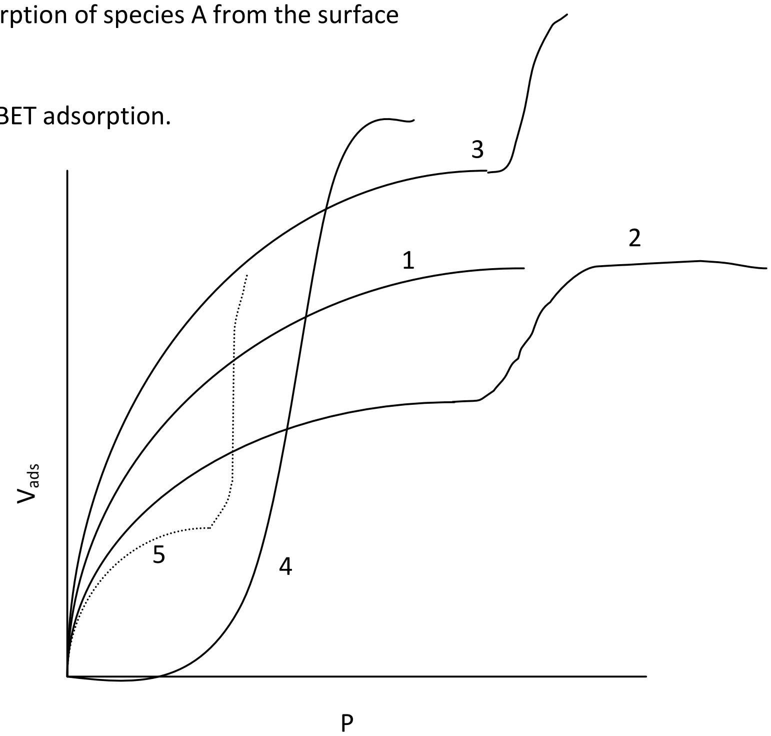
n_A = no. of particles approaching the surface

f_s = Fraction of particles that stick to the surface

F = Fraction of surface covered

r_A = rate of desorption of species A from the surface

28. Plot V_{ads} vs P in BET adsorption.



1 – Multilayer adsorption proceeds in a manner very similar to single layer adsorption

2- Sub-surface adsorption begins after top surface is saturated

3- Surface, sub-surface and core of the particle involved in adsorption

4-Chemisorption

5- Combination of physisorption and chemisorption

29. For a spherical particle interacting with a planar surface, how does F_{vdw} depend on d_p and Z_0?

Ans: For a spherical particle, F_{vdw} is directly proportional to the diameter of the particle and inversely proportional to the square of the distance of separation between the particle and surface.

$F_{vdw} \ \alpha \ dp$

$F_{vdw} \ \alpha \ 1/z_o^2$

30. How is F_{vdw} affected by intervening fluid, material type, surface roughness, time?

Ans:

Intervening fluid: Hamaker constant is much lesser when the intervening fluid is a liquid like water and higher for air. Therefore Van der waals forces are also reduced in water compared to air.

Material type: Hamaker constant is highest for metals followed by semi-conductors, plastics and elastomers in decreasing order. Therefore Van der waals forces are also highest for metals.

Roughness: When we have two smooth surfaces, the Van der waals forces is very high. When one of the surfaces is rough, the Van der waals force decreases substantially because of reduced contact area. However when both the surfaces are rough, depending on how the asperities are placed and the interlocking mechanism between the two surfaces, the Van der waals forces may increase or decrease.

Time: If the surface is soft and a particle approaches it, over a period of time the particle may start deforming the surface and ultimately get encapsulated within the surface itself. This will lead to an increase in Van der waals force as the distance of separation Z_o is greatly reduced and there is a higher level of contact between the particle and the surface.

31. How does the ionic double layer force depend on double layer thickness, d_p, Z_0?

Ans: Ionic double layer force is directly proportional to the diameter of particle (d_p). It depends on K and Z_o according to the following relationship:

$F_{idl} \propto K [e^{-Kzo}/1- e^{-Kzo}]$

32. How does surface tension force depend on d_p, γ?

Ans : Surface tension force is directly proportional to both d_p and γ.

$F_{surface\text{-}tension} \propto d_p , \gamma$

33. What are some methods to assess particle removal from surfaces?

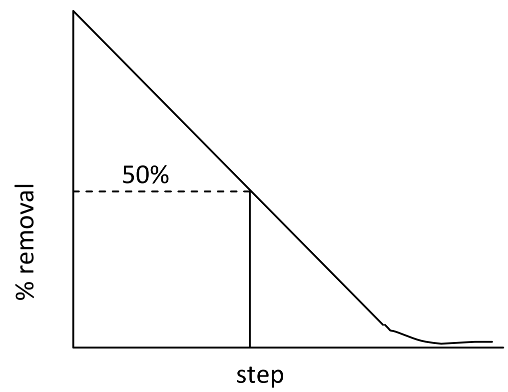
Ans:

a. Optical microscopes – for large surfaces with coarse particles.
b. Scanning electron microscopes – to look at how individual particles get dislodged from a surface. Works well for particles in 1 micron size range.
c. Image capture- capture an image of the surface before and after the application of removal force and compare the images.

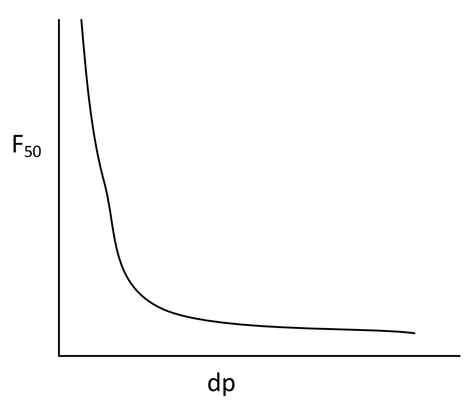
d. Laser scanning- Looks at light scattering from a surface before and after the removal step. Works well for highly polished and smooth surfaces where the surface does not interfere with the measurements.

34. How do extractive methods work for adhesion force measurement?

Ans:



To measure the adhesion force, we apply a removal force starting from time t=0 and measure the number of particles left on the surface at each step. The number of particles removed will exhibit asymptotic behaviour as shown above. Corresponding to each of these stages we can estimate the force required to remove the particles and the force required to remove 50% of the particles in conventionally taken as the adhesion force.



35. What are advantages & disadvantages of wet vs dry removal of particles?

Ans:

Dry removal	**Wet removal**
Advantages: 1. One step process, can be automated. 2. Effective for loose dirt accumulated on the surface. 3. Brushing/scrubbing can be done on both top and bottom surfaces at the same time. Quick and easy method to remove impurities.	**Advantages:** 1. Done in the presence of a liquid. Prevents contact between the surface and the bristles of the brush and thereby reduces any risk of damage to the surface. 2. Effective for oily layer/hydrophobic layer/film type of impurities on the surface. 3. Can reach particles that are hard to reach using a dry wipe.
Disadvantages: 1. Line of sight process. Cannot remove contaminants in the hidden recesses of the surface. 2. Aggressive method. Chances of causing damage to the surface.	**Disadvantages:** 1. Requires a drying step. The drying step may be an intrusion to the manufacturing process itself (ex. silicon wafers) if it is a predominantly dry process. 2. Adds to cost. Drying is also energy intensive. 3. The chemical/solvent used to remove impurities from the surface must be checked for purity and can be a potential source of contaminants.

36. Why is a finer particle more difficult to remove?

Ans: A particle is removed from a surface when the drag force exceeds the adhesive force provided the surface is smooth. Probability of removal is given by the ratio of Drag force to Force of adhesion.

$$P_{removal} = \frac{F\ drag}{F\ adhesion}$$

$F_{drag} \;\alpha\; dp^2$

$F_{adhesion} \;\alpha\; dp$

Therefore, $P_{removal}$ scales as particle size and that is why it is easier to remove bigger particles and finer particles are difficult to remove from a surface.

37. What is the mechanism of ultrasonic cleaning? How does ultrasonic force depend on f, A?

Ans : In ultrasonic cleaning, an oscillating pressure field is set up within the liquid by generating an acoustic field. As the liquid oscillates, it keeps going through alternating phases of compression and rarefaction and breaks into bubbles. The energy present in the bubble volume before it collapsed must be conserved and this is done by transmitting the energy in the form of a shock wave that travels through the liquid. The formation and collapse of bubbles is known as cavitation. The bigger the size of the bubble before it collapses, the greater will be the cavitation energy. Cavitation forces are much higher in low frequency Ultrasonics compared to high frequency Ultrasonics.

Ultrasonic force is inversely proportional to the square of frequency

($F_{ultrasonic} \alpha 1/f^2$) and independent of amplitude (A)

38. What is the mechanism of megasonic cleaning? How does megasonic force depend on f, A?

Ans : When we have an ultrasonic field and operate it at a very high frequency, it is called megasonic cleaning. Acoustic streaming essentially refers to the unidirectional flow induced in the liquid when we couple a high frequency field to it in the megasonic range. Megasonic field has a strong streaming characteristic whereas the ultrasonic field has a strong cavitational characteristic. Acoustic streaming is essentially a shearing mechanism and results in high velocity flow of liquid over the surface.

Megasonic force is directly proportional to the square of frequency and Amplitude.

($F_{megasonic} \alpha f^2$,A)

39. What effect do particle size, temperature and fluid viscosity have on cohesion?

Ans : Particle size and fluid viscosity are inversely proportional to cohesion. As particle size decreases, agglomeration increases. As fluid viscosity decreases, agglomeration increases.

Temperature is directly proportional to cohesion. So by lowering the temperature, we can keep the particles uniformly dispersed.

40. Name 2 ways in which particle size distribution can indicate extent of cohesion.

Ans : The rate of reduction of total number of particles in the system can be detected and expressed in two ways. When we plot the number of particles N(t) as a function of time t, a decrease in the number of particles over time is indicative of cohesive phenomena in the suspension. Similarly when we plot particle size dp as a function of time t, an increase in the particle size with time indicates agglomeration and presence of cohesion between particles in the system.