

# Exam Particulate Products

11 November 2010

## Instructions

- Don't forget to put your name and student number on every sheet that you hand in.
- Work out answers in formulas and fill in the numbers only at the end.
- At the final answer also give the units.
- The total number of points you can get is 96.
- You are allowed to use the attached sheet with equations and a  $C_D$ -Re graph.

## Particle size, shape and distribution (11)

1. A particle is shaped like a rectangular block of 0.4 x 0.5 x 0.6 mm. Calculate:
  - a. the volume equivalent sphere diameter, (2)
  - b. the surface area equivalent sphere diameter and (2)
  - c. the surface volume equivalent sphere diameter. (2)

answers:

$$A = 2 * (0.4*0.5 + 0.4*0.6 + 0.5*0.6) = 1.48 \text{ mm}^2$$

$$V = 0.4*0.5*0.6 = 0.12 \text{ mm}^3$$

$$\text{a) } V = (\pi/6) d^3 = 0.12 \Rightarrow d = 0.612 \text{ mm}$$

$$\text{b) } A = \pi d^2 = 1.48 \Rightarrow d = 0.686 \text{ mm}$$

$$\text{c) } V/A = d/6 = 0.12/1.48 \Rightarrow d = 0.486 \text{ mm}$$

2. Given the mass distribution of a powder in the table below, calculate the arithmetic average of the surface distribution. (5)

$d_{max}$ [mm]	$d_{min}$ [mm]	mass [%]
2.0	1.0	20
1.0	0.5	60
0.5	0.2	20

## Answer

$d_{av}$ ( $d_i$ )	$m_i/d_i$
1.5	13.33333
0.75	80
0.35	57.14286

$$d_{32} = (M_3/M_2)^{-1} = \text{sum } m_i / \text{sum } (m_i/d_i) = \mathbf{0.66 \text{ mm}}$$

## Particle size measurement (3)

3. Give two techniques with which the total surface area of a powder can be measured. (3)  
Answer: Gas adsorption (or BET), permeation

## Forces between particles (7)

4. A powder consists of particles with a density of  $1500 \text{ kg/m}^3$ , an average particle diameter of 50 micrometer, a surface roughness of 50 nanometer (i.e. the average radii of surface roughnesses) and a Hamaker constant of  $15 \cdot 10^{-21} \text{ J}$ . The contact distance between particles is 0.4 nanometer.
  - a. Calculate the Van der Waals force between two particles of this powder. (3)
  - b. Do you expect this powder to be good flowing? Explain. (4)

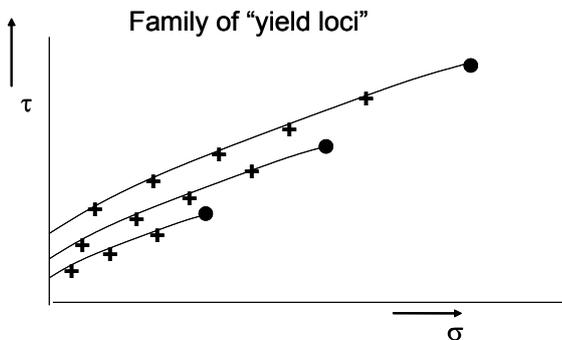
Answers:

a)  $F_{vdW} = 3.9 \cdot 10^{-10} \text{ N}$

b)  $F_g = 9.6 \cdot 10^{-10} \text{ N}$  Since gravity is higher than the cohesive force between two particles the powder is probably good flowing, but since the difference is only small it will not be very good flowing, so the flowing properties of the powder are probably moderate to good.

**Powder flow & hoppers (6)**

5. The measured yield loci of a powder are given in the picture below.  
 a. Is this the result of a test with a cohesive or a cohesionless powder? Explain. (3)  
 b. Which of the yield loci is measured at the highest porosity? Explain. (3)



**Answers**

For a cohesionless powder the yield loci would coincide and be straight lines through the origin. This test was performed with a cohesive powder.

The lowest yield locus represents the highest porosity because then the smallest friction between the particles is expected.

**Mixing and segregation (4)**

6. What are the advantages and disadvantages of mixers with rotating equipment compared to tumbling mixers? (4)

**Answer:**

They are relatively expensive compared to tumbling mixers. On the other hand they are less sensitive to (shear) segregation and they are more suited for cohesive powders.

**Sedimentation of single particles (9)**

7. A steel ball of 1.50 cm diameter and density 6.00 kg/dm<sup>3</sup> settles in an oil of density 1.20 kg/dm<sup>3</sup> and viscosity 20 mPa s. Calculate the stationary settling velocity (9)

**Answer:**

Set up a force balance with gravity, bouyancy and friction. First assume the turbulent regime:”

$$\frac{1}{6} \pi d^3 (\rho_{steel} - \rho_{oil}) g = 0.44 \frac{\pi}{4} d^2 \frac{1}{2} \rho_{oil} u_0^2 \Rightarrow u = 1.74 \sqrt{\frac{(\rho_{steel} - \rho_{oil}) g d}{\rho_{oil}}} = 1.34 \text{ m/s}$$

Then check Reynolds:  $Re = 1200$ . This is o.k.

**Sedimentation of suspensions (4)**

8. The sedimentation rate of a fine suspension can be increased by coagulation. In which two ways can coagulation be promoted? Explain. (4)

**Answer:**

By increasing the ionic strength, e.g. by adding multivalent ions like  $Al^{3+}$  and  $Ca^{2+}$  (these decrease electrical repulsion between particles) or by adding coagulant or flocculant (charged polymers that can create bonds between small particles)

**Fluidisation (16)**

9. 200 kg of spherical particles with a diameter of 100  $\mu m$  and particle density 1500  $kg/m^3$  are fluidised by water (density 1000  $kg/m^3$ , viscosity 0.001 Pa s) in a circular bed of cross-sectional area 0.25  $m^2$ . The single particle terminal settling velocity of the particles is 2.7 mm/s and the voidage at incipient fluidisation is known to be 0.45. The flow regime is laminar (so you can use the Carman-Kozeny equation).
- calculate the bed height at incipient fluidisation, i.e. at the liquid flow rate at which fluidisation starts (4)
  - calculate  $(1-\epsilon)/\epsilon^3$  when the liquid flow rate is  $1 \times 10^{-4} m^3/s$ . (8)

**Answer:**

a) The volume of the particles is  $200kg / 1500 kg/m^3 = 0.133 m^3$ , The bed volume is this volume divided by  $(1-\epsilon)$ . This is equal to area\*height, so  $H_{bed} = V_{particles} / A (1-\epsilon) = 0.97 m$

b) The dynamic pressure drop is then from:  $(m g)_{eff} = m g (\rho_{particles}-\rho) / \rho = \Delta p A \Rightarrow \Delta p = 7850 Pa$   
This should be equal to the dynamic pressure drop according to Carman Kozeny (laminar flow,  $Re_{single\ particle} = 0.27$ ). All data are known except the porosity and the bed height ( $L$  in the Carman Kozeny eq.), but the bed height is related to the porosity by  $L = L_0/(1-\epsilon)$  with  $L_0 = m/\rho_{particles}$ . So actually only the porosity is unknown. The equations can be solved to give:  $(1-\epsilon)/\epsilon^3 = 2.04$ . From this the porosity can be calculated to be 0.587

10. An A-type powder has a minimum fluidisation velocity of 1 cm/s. What can you say about its minimum bubbling velocity? (4)

**Answer:**

An A-type powder has a minimum bubbling velocity above its minimum fluidisation velocity, therefore  $u_{mb} > 1 cm/s$

**Grinding (6)**

11. A crusher is used to reduce dolime (crushing strength 100  $MN/m^2$ , particle density 2300  $kg/m^3$ ) from 10.0 to 0.3 mm average particle size. This process requires 19 kJ per kg of crushed dolime. Use Kick's equation to estimate the energy requirement for crushing of limestone (crushing strength 70  $MN/m^2$ , particle density 1600  $kg/m^3$ ) from 15.0 to 0.3 mm average particle size with the same machine. Is the Kick equation the right equation? (6)

**Answer:**

The Kick equation is the right one, because the product is still quite coarse.

Using  $E_{coarse} = K_K f_c \ln(S_2/S_1)$  we can calculate  $K_K$ .  $S$  is the surface area per kg, i.e.  $S = 6/\rho_p d_p$  and the proportion of the  $S$ 's is:  $S_2/S_1 = d_{p,1}/d_{p,2}$ . So it follows that  $K_K = ???$ . Finally for limestone we find:  $E = ??? kJ/kg$ .

**Other particle properties (7)**

12. Which characteristic of the surface energy of a crystalline substance is not shown by an amorphous substance? (3)

**Answer:**

Orientation-dependence: the various surfaces of a crystal can have different energies.

13. Describe the main differences between chemisorption and physisorption. (4)

Answer: chemisorption is due to chemical bonds with a high energy (vs. physical bonds with a low energy), chemisorption is always only in monolayers while physisorption can be in multilayers

**Production of particles & crystallization (7)**

14. Which steps can be rate limiting for crystal growth? (3)

Answer: crystal growth occurs in three steps, 1) diffusion of a molecule from the solution to the crystal surface, 2) diffusion of a molecule on the crystal surface to a binding site and 3) incorporation of a molecule in the crystal. Each of these steps can be rate limiting.

15. In a certain crystallisation process supersaturation is achieved by cooling. What is the expected effect of increasing the cooling rate on the crystal size? Explain. (4)

Answer:

One would expect more and smaller crystals, because the supersaturation will be higher, and therefore the nucleation rate will increase more than the growth rate.

**Powders and products (8)**

16. Explain why lactose which is partly amorphous can be made by spray drying, but not by vacuum crystallisation (in which supersaturation is achieved by simultaneous evaporation and adiabatic cooling)? (4)

Answer:

Spray drying is a much faster process, in which an amorphous structure can be frozen. Vacuum crystallisation is slower, and the molecules have time to arrange themselves in a crystal lattice.

17. Give two ways in which the dissolution properties of a powder be improved? (4)

**Answer:**

Granulation and lecithination.

**Tabletting (12)**

18. Three different substances are compacted to tablets with a load of 150 Mpa after mixing with 1 % magnesium stearate. Mixing time and compaction time are varied. The strengths of all tablets are determined. The results are in the table below. Give for each of the three substances the dominating behaviour during compaction and explain your answers. (8)

Mixing time	5 min	5 min	30 min
Compaction time	2 s	30 s	2 s
Asperidine nitrate	75 N	83 N	42 N
Asperidine sulfate	72 N	73 N	70 N
Asperidine stearate	capping	20 N	capping

Answer:

Asperidine nitrate is probably plastic because there is a considerable effect of mixing the magnesium stearate longer. During longer mixing more surface of the original particles is covered with Mg-St and this prevents good binding. Since the tablets strength hardly depends on the compaction time the behavior is not elastic

Asperidine sulfate is brittle because the parameters have almost no effect on tablet strength; this happens with brittle substances because during compaction the particles break and a lot of new surface area for binding is created and this new surface area determines the strength of the tablet. Asperidine stearate is elastic because the tablets are either weak, or very weak (capping) and the tablet strength depends on the compaction time.

19. Which parameter in the tableting cycle can be varied in order to produce strong tablets from visco-elastic powder mixtures? Explain. (4)

**Answer:**

Tableting speed. By decreasing the speed stronger tablets can be obtained.

Plastic behavior gives stronger tablets than elastic behavior. A viscoelastic powder shows both plastic and elastic behavior. At short time scales the behavior is primarily elastic, at large time scales it is primarily plastic. Increasing the time scale increases the importance of plastic behavior and therefore gives stronger tablets.