HELIUM PYCNOMETRY AND POROSIMETRY

DETERMINATION OF BULK AND BULK TAPPED DENSITY

LASER DIFFRACTION

COMPARISON OF TWO TYPES OF MICROCRYSTALLINE CELLULOSE

1. Determination of pycnometric density

Density is the ratio of the weight to volume of the powder. The density measured with a gas pycnometer is determined by measuring the volume occupied by a powder of known weight. This volume corresponds to the volume of gas replaced by powder. When measuring density with a gas pycnometer, the volume occupied by open pores is not included, but the volume occupied by closed pores or pores inaccessible to the gas is included.

Helium is usually used as a measuring gas mainly because of its high ability to penetrate small open pores. If gases other than helium are used, different values are obtained, since the gas penetration depends on the pore size, as well as on the effective cross-section of the gas molecules.

Materials: MCC 102, MCC 200

Aids: gloves

Equipments: Helium pycnometer and measuring vessel with frit, Analytical balance

Procedure:

1. Wear gloves whenever handling cups!
2. Place the empty sample cup without frit on the analytical balance and press TARE.
3. Place the sample in the cup. It should be approx. 80% full.
4. Weigh the cup and sample and record the mass.
5. Remove the chamber lid.
6. Insert the cup into the chamber. Place the frit on top of the cup and close the chamber.
7. Press **Alt + 4** (analysis) and set parameters (Sample ID, Description Line 1 and 2, Sample mass). Press **ENTER** to move between fields. Insert none (press **ENTER** to confirm)
8. Press **ENTER** to start analysis. In case of incorrect data, press **ESCAPE**.
9. When the analysis is complete, the pycnometer beeps three times, and the indicator light (Active) goes out.

10. **Press Alt + 5** to display results (average density and the deviation from the mean)

2. Determination of bulk and tapped bulk density

The bulk density of a powder can be determined by measuring the volume of a powder sample of known weight using a volumetric cylinder. The tapped bulk density can be determined by the same procedure, but it is calculated from the tapped bulk volume of the powder that is obtained after its tapping, usually after 1250 taps.

Materials: MCC 102, MCC 200

Aids**:** measuring cylinder 50 ml

Equipment: Erweka SVM 102 device, analytical balances

Procedure:

1. Weigh the empty graduated cylinder and then pour 50 ml of powder into it without tapping (= bulk volume V_0). Weigh the powder cylinder again and determine the weight of the powder.
2. Attach the cylinder to the Erweka SVM 102 device and enter 1250 tapping, confirm (Enter).
3. Press Start to begin the motion of the cylinder and determine the tapped bulk volume V_{1250}
4. Repeat the measurement for each sample three times, calculate the bulk and tapped bulk density values from each measurement, and then their average value.

3. Calculation of interparticular porosity

External (interparticular, extraparticular) porosity is defined as the ratio of the volume of gas (i.e. the volume of air filling the free spaces between particles in their tightest arrangement) to the total volume of particles. External porosity can be determined using the equation:

$$interparticular\ porosity = (1 - \frac{\rho_{1250}}{\rho_{pycn.}}) \cdot 100$$

Where ρ_{1250} denotes the tapped bulk density after 1250 taps and ρ_{pycn} indicates the pycnometric density.

Procedure:

Calculate the interparticular porosity for MCC 102 and MCC 200 from the average values of pycnometric density and bulk tapped density.

4. Measurement of particle size volume distribution using laser diffraction

The laser diffraction method is based on the analysis of the diffraction pattern formed by exposing particles to a beam of monochromatic light. The principle of the method is therefore the scattering of monochromatic light after passing through the cell with the sample. The intensity of the scattered light is directly proportional to the size of the particle. The magnitude of the scattering angle and the particle size are inversely proportional.

Materials: MCC 102, MCC 200

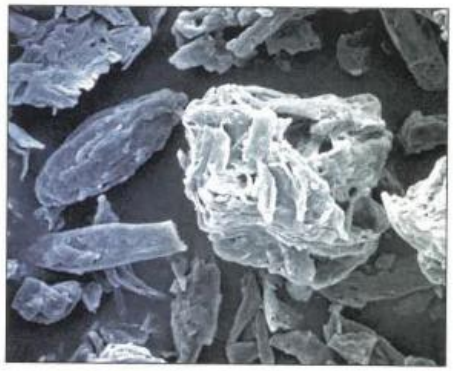
Aids**:** spoon

Equipment: Mastersizer 3000, dry cell

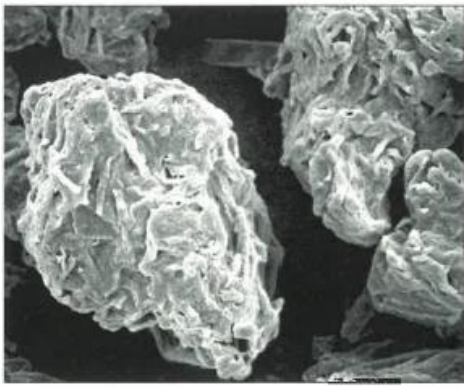
Procedure:

Using laser diffraction with the dry cell method, determine the values of d_{10}, d_{50}, d_{90} for both types of MCC (refractive index MCC = 1.5, absorption index = 0.1). Repeat the measurement three times and calculate the average value.

Conclusion: Compare the tested Avicels in terms of measured characteristics.



Avicel PH 102



Avicel PH 200