Mercury Intrusion Porosimetry: Determination of Pore Size, Particle Size and Density of Dry and Wet Materials

Jürgen Adolphs

POROTEC GmbH, Niederhofheimer Str. 55a, D-65719 Hofheim

Mercury intrusion porosimetry is a suitable technique to determine pore size, particle size as well as the bulk and apparent density of solids. The liquid nonwetting mercury is pressed into the porous or particulate system, while the applied pressure is inverse proportional to the pore size. Thus with a pressure of up to 400 MPa pore radii of 1.8 nm are detectable. After a review of the technique and the according evaluation models for pore size (Washburn) and particle size (Mayer Stowe) some examples on pharmaceutical materials both from literature (1 – 5) and from own measurements will be discussed. In particular for compressible hollow wax spheres it will be shown that the choice of the proper range of the mercury intrusion and extrusion curves for the determination of the density coincides very well with results from high precision helium pycnometry. Another issue that will be discussed is the question of the necessity of drying samples before they are investigated with mercury intrusion porosimetry. It is evident that hydrates can be dehydrated or swelling porous materials change their porosity depending on relative humidity. It is also clear that processing, production and storage is influenced. Therefore determination of the pore size distribution e.g. of a dried superporous hydrogel (6) is hardly to correlate with swelling phenomena or water-solid contact angles. The nonlinear effects of moisture on the porosity of an inorganic hydrate - hardened cement paste - was investigated in detail by the author (7). In future as a result the sample preparation according the new ISO 15901-1 (international standard for mercury intrusion porosimetry) will consider the samples nature. In this presentation it will be shown that with an appropriate porosimetry equipment such moisture related investigations are possible. Similar porosimetry investigations of moisture preconditioned mannitol tablets and microcrystalline cellulose (8, 9) are reported, however these samples had to be dried at vacuum in order to manage mercury filling specifically for the employed porosimeter equipment. Contrary in the author's investigations (7) the degassing pressure was controlled and kept well above the highest water vapour saturation pressure of the samples – in this way drying was avoided. The humidity effects of changing contact angles, change of porosity and swelling are considered. In addition the principle for the determination of the moisture dependent density with helium pycnometry will be explained.

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