Laboratory and Computing Methods

CHARACTERIZATION OF POROUS GLASSES BY MEANS OF POROSIMETRY

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Methods for the determination of the textural characteristics of microporous (mean pore diameter $\bar{d} < 7.5$ nm) and macroporous ($\bar{d} > 7.5$ nm) glasses by nitrogen adsorption and mercury porosimetry were examined. The results obtained were compared and the practical scope and limitations of the two methods determined. With macroporous glasses, the results were significantly affected by the input material constants employed, and also in the case of very fine particles.

INTRODUCTION

Porous glass is a special material prepared by leaching the chemically less resistant phase from a phase-separated glass (usually derived from the system Na_2O-B_2O_3-SiO_2) following suitable heat treatment [1, 2, 3]. The range of pore sizes in the porous glasses is very wide (from 0.2 to 1000 nm) [1]. According to the final pore size, which depends on the chemical composition of the initial glass, on its heat treatment and on the way of leaching, porous glasses are divided into macroporous glasses (mean pore size more than 7.5 nm, which is the smallest one measurable with standard mercury porosimeters) and the microporous ones (mean pore size less than 7.5 nm). However, this classification does not merely reflect the differences in pore size, because the two groups also differ substantially in the way they are prepared, in some of their properties, and in particular in their fields of application.

Macroporous glass under the trade name Controlled Pore Glass (CPG) [e.g. 4, 5] is of practical interest for biotechnology, chromatography and technical chemistry [1, 2], whereas the properties of macroporous glass approach those of silica gel and find similar applications (adsorbents, catalyst carriers, etc. [1, 2]).

Porous glass is mostly characterized [1] by three textural characteristics: mean pore size, pore volume and specific surface area. With macroporous glasses, there is an additional characteristic associated with the width of the pore size distribution curve. The textural characteristics are almost without exception established by nitrogen adsorption and mercury porosimetry [1].

Determination of the specific surface area according to the BET theory is a standard method that will not be discussed in more detail here. On the other hand, no available prospectus nor publication describes a detailed procedure for evaluating the textural characteristics from the results obtained by mercury porosimetry. According to Haller [7], the mean pore size is defined as the pore size corresponding to penetration of one half of the total intruded mercury volume. The volume of the pores is given directly by the volume of the mercury intruded.* The width of the pore size distribution curve is a rather inadequately defined textural characteristic. For example, the firm Fluka AG [4] defines it only as the range of pore sizes (in relative percent of the mean pore size) into which 80% of the total mercury volume penetrates during mercury porosimetry.

Another problem arising in the evaluation of mercury porosimetry results is the effect of the input material constants. The relation between pore diameter d and pressure p for mercury penetration is described by Washburn's equation [8]:

$$d = \frac{4\sigma \cos\theta}{p} \qquad (1)$$

where σ is the surface tension of mercury and θ the wetting angle of mercury on the material in question. Two material constants have to be substituted into the formula: surface tension σ and wetting angle θ. The frequently employed value for the surface tension of mercury is 0.480 Nm^{-1}. Liabastre and Orr

*The term "mean pore size", as used by Haller, is in fact the "median of pore diameter" and not a mean e.g. in the statistical sense of the word.

[9], on measuring the porosity of macroporous glasses, used the value of 0.474 Nm^{-1}, which corresponds to a deviation of 1.25% in mean pore size determination. No greater deviations in the σ values employed are customary and the resulting deviation is negligible when compared to other effects. A much greater dispersion is that of the values of the wetting angle employed, with the corresponding effect on the final values reported. Most porosimetric laboratories make standard use of angles $\theta = 140°$ or 142.34° which, however, are too high for the present system (in fact mercury - silica glass). Liabastre and Orr calculated with $\theta = 130°$, while according to Haller [7] and experimental results of measuring the wetting angle on quartz and flat glass by Kloubek [10], the value of $\theta = 135°$ is more suitable. This value is also approached by the experimental determination of the wetting angle on SiO_2 pellets, $\theta = 137.7°$, by Smith et al. [11]. The respective differences are negligible for the absolute majority of porosimetric measurements on other materials. However, a change in the θ value from 130° to 142.34° would change the mean pore size by 23%, which is considerably more than the width of the pore-size distribution for most macroporous glasses.

Determination of the textural characteristics of microporous glasses is mostly based on detailed evaluation of nitrogen adsorption curves. Specific surface area determination according to the BET theory as well as the pore size determination are standard methods [6]. In contrast to this, several theories yielding somewhat different results are employed in the pore-size determination [1, 6, 12]. The "mean pore size" (here designated d_{VA}) is defined by calculation according to the equation:

$$d_{VA} = \frac{4V_H}{A_p} \qquad (2)$$

where V_H is the volume of pores and A_p is the specific surface area. This relation holds on the assumption of a system of parallel cylindrical pores with a constant diameter, which is usually not met.

For the purposes of developing the technology of porous glasses at the Institute, it was deemed necessary to introduce methods for more detailed characterization of porous glasses. The paragraphs below summarize the respective findings which may also be useful for the characterization of other porous materials of similar types (silica gel, xerogels, etc.).

EXPERIMENTAL

Materials and methods employed

The experiments were carried out on samples of porous glasses developed at SVÚS Hradec Králové. Glass A 04/49505A in microporous form was prepared by leaching heat-treated (495°C, 5 h) and ground basic glass of type A (the system Na_2O-B_2O_3-SiO_2 [3, 13] with acid only (3 M HCl, 50°C). The macroporous form of this glass (A 04/49505B) was obtained by subsequent leaching of ground glass A 04/49505A in a basic solution (0.5 M NaOH, 25°C) [3, 13].

Glass C 08/66372B was prepared similarly to glass A 04/49505B from the type C initial glass (the system Na_2O-B_2O_3-SiO_2, using additions of P_2O_5 and fluorides) [3, 13] after heat treatment (663°C, 72 h).

Prior the measurements, the samples were dried at 200°C for 16 hours in a drying oven. In view of their hygroscopicity, they were weighed only after evacuation in the measuring apparatus.

The nitrogen adsorption-desorption curves were measured by means of the ASAP 2000+ instrument (Micromeritics Instruments Corporation, USA) and processed by the firm's own programs on the basis of the BET and the Barrett-Joyner-Halenda (BJH) theories. The pore-size distribution curves were calculated from both the adsorption and the desorption branches of the isotherm.

The mercury porosimetric measurements were carried out in the laboratory of the Mining Research and Safety Institute at Paskov (with Mr. P. Beneš as operator) on the Porosimeter 225 (Carlo Erba Strumentazione, Italy) and the Pore-Sizer 9310 (Micromeritics Instruments Corporation, USA) apparatus. The individual pressures were chosen so as to cover the maximum intrusion region uniformly and with the optimum number of points (15 to 40). The total number of points was about 55, the final pressure being 100 MPa.

The processing of experimental values from mercury porosimetry

From the values of the dependence of intruded mercury volume on pore diameter (for $\sigma = 0.480$ Nm^{-1}, $\theta = 135°$) obtained, the first value for mercury pressure lower than 3 MPa (or 7.5 MPa for pore sizes above 100nm) was chosen as 0%, the final value as 100%, and the remaining values were recalculated proportionally, thus eliminating the effect of interparticle spaces in the ground glass which tended to distort the results. The pore volume V_H was also determined for this pressure range.

The converted values of the intruded mercury volume in percent were plotted into a probability chart vs. the pore diameter, with emphasis on the course between 10% and 90% of the value. The interpolated straight line was used to determine the values of pore sizes corresponding to 10% of the intruded mercury volume, $d_{10\%}$, and to 90% of the intruded mercury volume, $d_{90\%}$. Their arithmetic average was further taken as the mean pore size, $\bar{d}$, of the respective sample. The evaluation of the results is shown comprehensively in Fig. 1.

Table 1

A comparison of textural characteristics of microporous glass A 04/49505A and macroporous glass A 04/49505B obtained by different methods

Sample	Method	$\bar{d}$ [nm]	V_H [mm^3g^{-1}]	H_d [%]	A_p [m^2g^{-1}]	d_m [nm]	d_{VA} [nm]
A 04/49505A	1	<7.5	12		3.6		
	2		146		272		2.1
	3		96		143	2.3	2.7
	4		99		158	2.3	2.5
A 04/49505B	1	11.6	392	18.6	151	11.5	10.4
	2		427		159		10.8 11.8
	3	19.3	426	29.0	108	26.3	**15.8**
	4	13.0	442	19.2	142	13.1	**12.4**

Explanatory notes :
Method 1 – mercury porosimetry.
Method 2 – nitrogen adsorption:
A_p according to the BET theory, V_H according to the single-point method for $p/p_0 = 0.986$ (the volume of pores less than 140 nm in size).
Method 3 – nitrogen adsorption: Evaluated by the BJH method (pores more than 2 nm in size).
Method 4 – nitrogen desorption: Evaluated by the BJH method (pores more than 2 nm in size).
The individual textural characteristics are defined in the text.

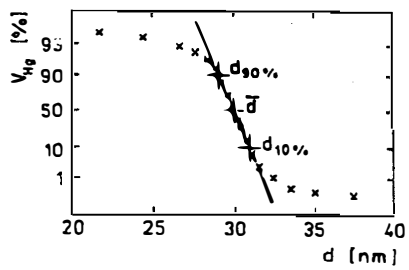

Fig. 1. The method of graphic evaluation of mercury porosimetric results

The width of the pore size distribution curve, H_d, was calculated from the equation:

$$H_d = \frac{d_{10\%} - d_{90\%}}{d_{10\%} + d_{90\%}} 100 \qquad (3)$$

and expressed in percent.

The conversion of the experimentally established pore volume V_H (in m^3g^{-1}) to percent of pore volume V_p can be effected according to the equation:

$$V_p = \frac{V_H}{V_H + \frac{1}{\varrho}} 100 \qquad (4)$$

where ϱ is the specific gravity of the macroporous glass skeleton. In practical calculations, use was made of the value $\varrho = 2\,200$ kg m^{-3}, which corresponds to silica glass [14]. The value is in a satisfactory agreement with the results of our measurements of the specific density of macroporous glass by means of the helium pycnometer Autopycnometer 1320 (Micromeritics Instruments Corporation, USA), $\varrho = 2\,217$ kg m^{-3}, as well as with those of similar measurements carried out by Tomanová and Schneider [15] on commercial macroporous glass CPG-10, $\varrho = 2\,189$ kg m^{-3}.

Mutual comparison of nitrogen adsorption and mercury porosimetry measurements

The results of determining the pore-size distribution curves by various methods were compared on ground porous glasses A 04/49505A and A 04/49505B (particle size 125 – 200 μm). The frequency curves obtained are shown in Fig. 2. With glass A 04/49505A, mercury porosimetry did not allow the pore size distribution curve to be determined, because an absolute majority of the pores were less than 7.5 nm in size. The resultant pore-size distribution curve for glass A 04/49505A from the adsorption branch was virtually identical with the curve from the desorption

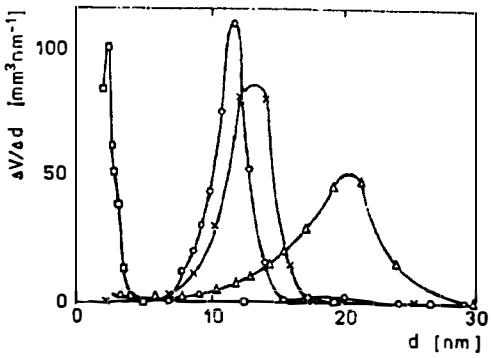

Fig. 2. Pore-size distribution curves of porous glasses.
A 04/49505A: □ nitrogen desorption.
A 04/49505B: ○ mercury porosimetry, △ nitrogen adsorption, × nitrogen desorption.

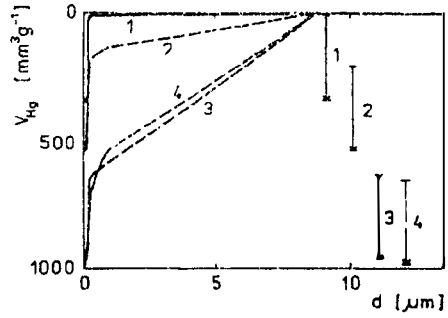

Fig. 3. Total volume V_{Hg} of intruded mercury in terms of pressure (recalculated to pore diameter) for glass C 08/66372B with various grain sizes (samples 1 – 4)

branch, and is therefore not shown separately. Table I summarizes the textural characteristics of the two porous glasses. For the purpose of comparison, also the modal diameter d_m, established from the pore-size distribution curve, and the "mean pore size" d_{VA} according to equation (2) are also listed.

The effect of particle size

The procedure for evaluation of the results of mercury porosimetry for macroporous glasses was successfully employed not only on ground glass with particles greater than 80 μm in size, but also on massive samples (plates, tubes, rods) with absolutely identical results. However, the procedure was not found suitable for measuring very fine-grained powders of macroporous glasses.

The effect of the sizes of very fine particles on the textural characteristics, calculated from mercury porosimetry, was studied on macroporous glass C 08/66372B with an initial particle size of 200 – 315 μm. This glass was ground in a ball mill in an aqueous medium and after drying divided into three fractions by means of the Alpine classifier. The particle size distribution curves were determined by the SediGraph 5100 analyzer (Micromeritics Instruments Corporation, USA). The input value of apparent density was calculated from the specific gravity of the macroporous glass skeleton (ϱ = 2200 kg m^{-3}) and of water, and from the volume of pores established by mercury porosimetry. The results obtained are summarized in Table II. In Fig. 3 the curves of the dependence of the volume of intruded mercury on pressure (following recalculation to pore diameter) for the individual samples are shown. Table II then lists the textural characteristics based on these values, on the assumption that the actual pore volume has not been changed by grinding (sample 1: V_H = 331 mm^{3}g^{-1}). For the determination of $\bar{d}$ and H_d, the initial value of the intruded mercury volume corresponding to 0% was therefore established by subtracting 331 mm^{3}g^{-1} from the total volume of intruded mercury (cf. the line segments in Fig. 3). The procedure described above was then followed.

Table II

The effect of particle size on textural characteristics calculated from the results of mercury porosimetry for glass C 08/66372B (V_H = 331 mm^{3}g^{-1})

Sample	Particle size		$\bar{d}$ [nm]	H_d [%]
	Median diameter [μm]	Range [μm]		
1		200–315[a]	188.7	7.2
2	30.6	18–120	183.3	12.4
3	14.4	9–23	182.3	11.5
4	4.5	0.1–13	182.5	13.6

a – screen mesh range

DISCUSSION

The applicability range of nitrogen adsorption and mercury porosimetry

The analysis of the technical possibilities of the two main porosimetric methods, as well as the results obtained showed that the two methods are conveniently complementary. Nitrogen adsorption and mercury porosimetry are based on quite different physical principles and their results are mutually independent. An additional advantage is that the possibilities of measuring by nitrogen adsorption increase with decreasing pore size where the accuracy and informative value of mercury porosimetry decreases.

The nitrogen adsorption method (and the processing of results of determining the nitrogen absorption-desorption isotherm on the basis of various theories) is applicable even for extremely small pores, but involves a number of limitations or equivocations. The most widely accepted values are those of specific surface area A_p on the basis of the BET theory. The procedure is applicable virtually all over the range of pore sizes in porous glasses. Determination of pore volume V_H by the single-point method for p/p_0 approaching unity is already restricted (e.g. according to the prospectus for ASAP+) for pores of up to 100 – 150 nm in size, and the pore size distribution curve according to the BJH theory can be theoretically determined over the range of 1.7 – 300 nm; however, both characteristics involve a considerable error from the pore size of about 50 nm upwards.

Direct determination of the mean pore size is rather difficult, as it depends strongly on the theory employed and usually fails to take into account very small pores which are virtually always present in microporous glass. As has been demonstrated by a comparison of the various methods (Table I), equation (2) is adequate as the first approach in the case of microporous glasses. With the use of the procedure based on the BJH theory, lower values of V_H and A_p were obtained in consequence of not including the very small pores (Fig. 2). Depending on the theory employed in the evaluation of the experimental values, even substantially differing textural characteristics were sometimes obtained [12]. One has to be therefore careful in their interpretation and compare only the textural characteristics obtained by the same procedure. In the case of microporous glasses, however, the detailed analysis of the adsorption-desorption isotherm of nitrogen is the fundamental (and only) resource of data for the determination of textural characteristics.

Mercury porosimetry is suitable for the determination of the mean pore size $\bar{d}$, the volume of pores V_H and the width of the pore-size distribution curve H_d for macroporous glasses. It can even be employed to determine the specific surface area A_p; however, in view of the lower accuracy of the procedure (calculation from the pore size distribution curve on the assumption of parallel cylindrical pores), evaluation of the nitrogen adsorption data according to the BET theory is more suitable. Under optimum conditions, both results of A_p determination are very close (cf. Table I). The applicability of mercury porosimetry to pore sizes exceeding 7.5 nm (3.8 nm for special porosimeters), and thus virtually only to macroporous glasses, is the main limitation of the method.

The relation of the characteristics determined to the actual texture of porous glasses

Of interest is a comparison of the pore-size distribution curves obtained from nitrogen adsorption and mercury porosimetry for the pore-size region where the applicabilities of the two methods overlap. Fig. 2 and Table I present the results for glass A 04/49505B which indicate a satisfactory agreement of the pore-size distribution curves provided by mercury porosimetry and the desorption branch of the nitrogen adsorption-desorption isotherm (evaluated according to the BJH theory). In contrast to this, a similar calculation from the adsorption branch yielded distinctly higher values. This difference can be explained by the texture of macroporous glasses, which is assumed to contain bottle-shaped pores.

Macroporous glass has a characteristic texture composed of channels interconnecting cavities having diameters larger by a factor of two or three [1, 3, 16]. The channels have a relatively circular cross section, formed by surface tension when the primary texture is created by separation. This is well discernible in electron micrographs [3, 9, 16]. These systems typically involve capillary condensation during adsorption of nitrogen, revealed by a characteristic course of the isotherm [12]. In contrast to this, microporous glasses exhibit adsorption properties close to those of silica gels, and in most instances (with the basic glasses derived from the system Na_2O-B_2O_3-SiO_2) comprise major cavities interconnected by channels that are at least partially filled with silica gel, precipitated in the course of leaching the chemically less resistant separated phase with acid [1, 17].

The results of mercury porosimetry and those of the desorption branch of the nitrogen isotherm on macroporous glass describe the diameter of the channel necks between the cavities, whereas the values from the adsorption branch characterize the cavity diameters. The results obtained correspond to those obtained in studying the initial, separated as well as leached glasses by transmission and scanning electron microscopy [3, 9, 12, 16].

The effects taking part in the determination of textural characteristics

In the case of macroporous glass, most textural characteristics ($\bar{d}$, V_H, H_d) are determined from the results of mercury porosimetry. Under the conditions of mercury porosimetry measurement the structure is stable (the compressive strength of silica glass, what is practically the skeleton of these glasses, being at least 490 MPa [14]), and as a result of low compressibility (a linear shrinkage of 0.018% at 200 MPa [14]), under a pressure of 200 MPa a pore diameter of 7.5 nm will theoretically change by the negligible value of 0.0014 nm.

The width H_d of the pore-size distribution curve is a statistical expression of the smallest dimensions of

the channels; however, the relationship will be more complex in consequence of the texture with interconnected channels described above. The actual value is associated with the way the glass has been prepared, and with high-grade products attains 5 to 20 relative percent in dependence on $\overline{d}$. The volume V_p of pores in macroporous glasses corresponds well to the volume of the alkali borate phase which is dissolved by the acid during the preparation of macroporous glasses [13].

As already mentioned above, nitrogen adsorption evaluated according to the BET theory is more suitable only for the determination of the specific surface area, A_p. The value of A_p is related to $\overline{d}$ and V_p (cf. equation (2)), and a comparison may be used as a check of the absence of micropores which may arise as a result of incorrect preparation and are undesirable for some applications.

Another problem arises in connection with very small particles. As shown by Fig. 3, incorrect values would be obtained if the entire volume V_{Hg} of the intruded mercury were taken as being equal to the total volume V_H of the pores. The results in Table II indicate that the texture of macroporous glass remains unchanged by grinding, so that it is only necessary to eliminate the effect of interparticle spaces whose dimensions, in the case of very fine particles, begin to approach those of the texture proper. This fact has to be taken into consideration when checking the properties of very fine powders (with grain sizes less than about 40 μm).

CONCLUSION

A comparison of the results obtained by nitrogen adsorption and mercury porosimetry showed that different methods have to be employed in characterizing the properties of macroporous and microporous glasses. Microporous glasses can be generally characterized by analyzing the adsorption-desorption isotherms of nitrogen. These allow the specific surface area A_p, the pore volume V_H and on certain theoretical assumptions also the pore-size distribution curve, and from this also the mean pore size $\overline{d}$, to be determined. However, the mean pore size, given by the d_{VA} value (according to equation (2)) is an adequate characterization for most practical purposes.

Satisfactory results of characterization of macroporous glasses were achieved with the procedure for the evaluation of mercury porosimetric data which is described above and which allows the mean pore size $\overline{d}$, the volume of pores V_H and the pore size distribution curve H_d to be determined. The set of textural characteristics is suitably supplemented with specific surface area A_p obtained by nitrogen adsorption (according to the BET theory). These characteristics are well interpretable and are directly associated with the actual texture of macroporous glasses.

The above procedures are well suited for particle sizes greater than 40 μm. It has been proved that the texture of porous glasses is retained by even finer particle sizes, but in consequence of the dimensions of interparticle spaces approaching those of the pores, an individual approach has to be taken in the interpretation of mercury porosimetric results.

Experience from porosimetric practice has shown that because of its properties, macroporous glass is a suitable standard material for mutual comparison of the results of different porosimetric methods, and even of those obtained in different laboratories. Its texture is likewise very interesting theoretically, and in measuring its properties one can detect certain measurable effects which are virtually negligible with other porous materials.

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CHARAKTERIZACE PORÉZNÍCH SKEL POMOCÍ POROMETRIE

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V článku jsou diskutovány metodiky stanovení texturních charakteristik mikroporézních (průměrná velikost pórů $\bar{d}$ pod 7.5 nm) a makroporézních ($\bar{d}$ nad 7.5 nm) skel pomocí adsorpce dusíku a rtuťové porometrie a jejich vztah ke skutečné textuře porézních skel. Vedle tří obecných texturních charakteristik: průměrné velikosti pórů $\bar{d}$, objemu pórů V_H a měrného povrchu A_p je u makroporézních skel doplněna ještě šířka frekvenční křivky velikosti pórů H_d. Podrobně je uvedena metodika vyhodnocení texturních charakteristik makroporézních skel z experimentálních výsledků rtuťové porometrie (obr. 1).

Z experimentálního ověření metodik na porézních sklech připravených v SVÚS vyplynulo, že adsorpce dusíku a rtuťová porometrie se dobře doplňují a v oblasti velikostí pórů, kde se možnosti obou metod překrývají, poskytují srovnatelné výsledky (obr. 2 a tab. I.).

Texturní charakteristiky mikroporézních skel je možné stanovit prakticky pouze podrobným zpracováním adsorpčně desorpčních izoterem dusíku podle různých teorií. Naproti tomu pro stanovení $\bar{d}$, V_H a H_d makroporézních skel je nutné dát přednost rtuťové porometrii a pouze měrný povrch A_p je výhodnější stanovit pomocí adsorpce dusíku (podle teorie BET). Dobře reprodukovatelné a interpretovatelné výsledky rtuťové porometrie jsou výrazně ovlivněny použitým úhlem smáčení θ (jako nejvhodnější se zdá hodnota $\theta = 135°$). Další problémy nastávají i při vyhodnocení texturních charakteristik velmi jemných prášků makroporézních skel (obr. 3 a tab. II).

Obr. 1.: Způsob grafického vyhodnocení výsledků ze rtuťového porozimetru.

Obr. 2.: Frekvenční křivky velikosti pórů porézních skel.
A 04/49505A: □ desorpce dusíku.
A 04/49505B: ○ rtuťová porometrie; △ adsorpce dusíku: × desorpce dusíku.

Obr. 3.: Celkový objem vtlačené rtuti V_{Hg} v závislosti na tlaku (po přepočtu na průměr pórů) pro sklo C 08/66372B s různou velikostí částic (vzorky 1 – 4).