The Definitive Guide to Porosity Characterization
Exploring Theory, Techniques and Practice for Industrial Application

Derived from the Greek word ‘poros’ meaning passage, porosity is a critical parameter used to describe and quantify the internal structure of many materials of industrial interest. Porosity influences permeability, the ease with which fluids can travel through a solid, the extent and accessibility of internal surface area, and relationships between strength and weight. Examples of products for which porosity is a performance-defining characteristic include: catalysts; construction materials, including concrete; packaging; ceramics; pharmaceuticals; membranes and filters; active elements of batteries and fuel cells; and the finished components produced by metal manufacturing methods such as additive manufacturing (AM) and metal injection molding. Porosity characterization is equally essential for the effective exploitation of oil and gas bearing reservoirs.

Practical techniques used to investigate porosity include pycnometry, gas adsorption, mercury porosimetry, and porometry. Mathematical manipulation of the resulting measurements enables characterization to varying degrees of detail, with alternative approaches optimal for different materials and for analysis over different length scales. This paper takes an introductory look at these techniques, the data generated, and their strengths and limitations for specific materials, covering more complex approaches such as the Reverberi method and the application of non-local density functional theory (NLDFT) to illustrate the necessity for, and potential benefits of, more advanced analysis.

Exploring the need to characterize porosity.
An examination of examples of porous materials and the ways in which porosity defines their performance is helpful when considering how to quantify porosity and when assessing the relative merits of different techniques. Examples of industrial materials for which porosity is important include 3D printed metal components, pharmaceutical tablets, membranes, oil-bearing rocks, and catalysts. Let’s look at how porosity impacts the performance of each of these materials to introduce requirements for measurement.

For metal components, porosity impacts critical mechanical properties such as strength and ductility. A more porous structure has lower density, a lower mass to volume ratio that makes it lighter weight, all other dimensions being equal. This is an advantage in many applications but not if it comes at the price of unacceptably compromised mechanical performance. AM is noted for the control it offers over porosity, with the 3D printing of replacement bone, scaffolds and implants exemplifying what can be achieved. The porosity of such parts can be controlled to promote bone regrowth and integration within the body, and to improve biomechanical performance.

While porosity also impacts the strength of pharmaceutical tablets, here there are additional motivations for its measurement and control. Porosity can be a critical quality attribute (CQA) for tablets - a parameter that defines clinical efficacy - because of its influence on how easily solvent penetrates the tablet matrix to trigger the processes of disintegration and dissolution that influence bioavailability. The porosity of the granules used to make tablets may also be critical. Granulating a tablet blend, prior to compression, can improve manufacturing efficiency and the quality of the finished tablets. Controlling porosity helps to ensure that a blend has the compaction characteristics required to produce a stable tablet under acceptable processing conditions.

In these examples, it can be argued that the basic informational requirement for porosity data can be met by an averaged value for the sample, the exception being to understand the penetration of liquid into a tablet matrix. To determine whether a material will allow the ingress of a solvent we need more detailed information than an averaged total porosity value essentially differentiating solid material from voidage. Rather, we need information on the size of individual pores. Many applications call for this level of porosity information to elucidate the ability of molecules to move within a substrate, to access internal surface area and volume, for example, or active sites within a solid matrix.

Mudrocks and catalysts exemplify materials for which the need for extensive porosity characterization is linked to development of a greater understanding of fluid motion. The porosity of mudrocks such as shale defines their capacity to hold hydrocarbons and permeability, with finer pores giving rise to low permeability resulting in a higher resistance to fluid flow.
Traditionally, tight shale, which has low porosity and permeability was not considered economically viable for hydrocarbon production but in recent years, its relative abundance, combined with an enhanced desire for energy independence, has led to the reappraisal of this view by economies across the globe. Measurements of porosity enable oil and gas engineers to estimate the capacity of tight shale reservoirs and support detailed reservoir evaluation. Pore throat size, pore volume and pore size distribution are all routinely measured to optimize extraction strategies.

Heterogeneous catalysts can be engineered with precisely controlled porosity, the development of hierarchical catalyst topologies and metal organic frameworks (MOFs) being notable steps forward in this regard, to exert precise control over mass transfer to and from the active site. Controlling the rate of diffusion of reactants and products makes it possible to tailor localized concentrations to optimize turnover and selectivity, thereby maximizing system productivity. Working at the molecular level, in this way, calls for information about the size, size distribution, and shape of pores to directly support catalyst design.

One other class of applications worth highlighting with respect to porosity measurement requirements is where there is specific interest in the movement of certain molecules through a substrate, as distinct from within it. Prime examples of this type of application include membranes, filters, battery separators, and medical equipment such as masks and gowns. In these applications, porosity is being controlled to allow or prohibit the transit of a specific molecule typically with the aim of performing a separation or preventing the spread of a substance. This creates a somewhat different focus to measurement and can directly impact the technology chosen for characterization.

This far from exhaustive exploration of the application of porosity data highlights two key points. Firstly, there is a need for different types of porosity characterization. In some instances, an averaged porosity value is sufficient, effectively a voidage figure indicative of the relative density of the sample, but often the requirement is for more detailed information. Metrics that may be of interest extend to

- Pore size distribution
- Total porosity or total pore volume
- Total pore surface area
- Median pore diameter
- Pore throat size

Secondly, the range of materials for which porosity is of interest is truly diverse creating a need for porosity measurement across multiple length scales. Structures are defined as microporous, mesoporous or macroporous (see figure 1), depending on the size of pores present, with effective technology required to cover each range. This is one of the principle criteria used to select a technique for a given application but beyond this, each has a unique profile of strengths and limitations that requires careful consideration to ensure an optimal choice.

Figure 1: Industrial materials exhibit porosity at the microporous, mesoporous and macroporous range, with pore size an important determinant of measurement technique.
Pycnometry: average porosity from density measurements.

One of the simplest ways of gaining insight into porosity is via density measurements. Pycnometry is widely recognized as one of the most accurate and reliable methods of determining volume, and by extension density. It is a displacement technique which means that sample volume is determined from measurements of the volume of a displaced medium. This medium can be a gas or a solid.

Gas pycnometry is suitable for a wide range of materials including powders, slurries and solid samples such as tablets or rock specimens. The sample is sealed in a chamber of known volume which is then charged to a specified pressure (see figure 2) with an inert gas, typically helium or nitrogen, depending on the properties of the material. Opening the sample chamber to a second cylinder, also of known volume, allows the gas to discharge, filling the reference chamber to a measurable pressure. The volume of gas and by extension that of the sample is calculated from the resulting measurements by applying the Ideal gas law.

This technique has the attraction of being relatively fast and simple, but only provides a single averaged density figure for the sample based on the total volume accessible to the gas. In the case of a sample with no accessible porosity – one that is fully dense or only has closed porosity - this is the volume of the sample or the total volume of all the individual particles in the case of a powder. On the other hand, if the sample has open or accessible porosity then the displacement gas will penetrate the internal structure and the volume of gas displaced will be lower, for particles of an equivalent size. The volume measured is determined by the structure of the sample and the displacement medium used and the associated density is variously known as the true, absolute or skeletal density, with the medium used for measurement usefully quoted alongside measured values. Since the displacement gas easily penetrates interstitial spaces in a powder these values are notably different from bulk density measurements which are based on the volume of the bulk powder sample, including interstitial volume.

The results generated by pycnometry are density values. To convert them into porosity values we need to compare them with, for example, theoretical density, an approach that works well for metal powders, or measurements of the envelope density of the sample, for a tablet, metal component or rock specimen. Measurements of envelope density can be made using solid phase pycnometry.

In solid phase pycnometry the displacement medium used is typically a free-flowing quasi-fluid as exemplified by DryFlo. This is a ceramic powder mixture composed of small rigid spheres that has a high degree of flowability. To carry out a measurement, displacement medium is placed in a precision cylinder of known volume and compressed as the cell vibrates to establish a zero-volume baseline (see figure 3). The sample is then placed in the cylinder along with the displacement medium and the compaction process is repeated. The volume of the sample is calculated from the difference in the distance that the piston travels to achieve an equivalent degree of compaction (h).
This technique determines the *envelope volume* of the sample. The displacement medium conforms closely to the surface of the sample during measurement but does not invade any pore space. Clearly this approach is not suitable for powders, only solid samples can be analysed. For these, samples *envelope density* in combination with *true, absolute or skeletal density* defines the *total porosity* of the sample.

**Mercury Porosimetry: rapid measurement across a broad dynamic range**

First developed around 1945, mercury porosimetry (otherwise known as mercury intrusion porosimetry) offers an unrivalled dynamic range for detailed investigation of the porosity of powders and solids. Providing rigorous characterization from the macroporous (pore size > 50 nm) down into the mesoporous region (pore size ~3nm) it is complementary to gas adsorption which extends to microporous materials. The underlying measurement principle is the correlation between the size of pores and the pressure required for filling.

In a mercury porosimetry measurement the sample is loaded into a sealed cup which is evacuated to remove contaminating gases and vapors, and then allowed to fill with mercury at a user-defined, but typically very low absolute pressure (see figure 4). A reservoir of mercury held in a capillary tube is subsequently forced into the sample through the application of pressure, typically up to a maximum of 60,000 psia. Changes in mercury level are determined from changes in capacitance, between the metal plating surrounding the capillary tube and the electrical contact at the base of the sample cup. This allows precise determination of the volume of mercury associated with intrusion into the pores of the sample at any given pressure; extrusion can be investigated in a strictly analogous way.

Mercury has a contact angle in the region of 130°, depending on the surface with which it is in contact, indicating that it is essentially a non-wetting liquid with respect to most solids. In practice this means that the surface tension of mercury is so high that it forms a bead when deposited on a surface only flowing into the pores of a solid when forced to do so via the application of pressure. During a porosimetry analysis, the increase of pressure from a sub-atmospheric starting point is associated with progressively forcing mercury into different areas of the sample. For example, at atmospheric pressure intrusion is limited to larger openings in the sample in the region of around 12 mm in size.

**Figure 4:** Mercury porosimetry involves measurement of the pressure required to force mercury into the pores of a sample.

**Figure 5:** The very high contact angle of mercury makes it non-wetting to the majority of solids.
As pressure increases beyond atmospheric levels, into the region where measurements are more routinely made, pores begin to fill. If the sample is a powder then interstitial volumes typically fill first, because of their larger size, with complete filling of the interstitial volume usually marked by an abrupt change in filling rate. Progressive filling then continues to the point at which all accessible porosity is filled. This generates a total pore volume which can be converted to a specific total pore volume, pore volume per unit mass, simply by dividing by the mass of sample present. The volume of sample present can also be quantified to generate a value of true, absolute, or skeletal density, as in gas phase pycnometry. Crucially though the numerical values generated via the two techniques may be different since the ability of mercury to penetrate internal structure differs from that of gas displacement media. Porosity measurements are routinely quoted along with the method used for measurement for this reason and this is good practice.

Of course, the primary value of mercury porosimetry lies in its ability to generate far more detailed porosity information than can be accessed from pycnometry data. This involves detailed analysis of the plot of pressure (using the Washburn equation to convert pressure to pore size) versus volume of mercury forced into the sample (using measurement of change in capacitance of the sample holder).

\[ D = -\frac{4\gamma \cos\theta}{P} \]

Equation 1: The Washburn equation correlates pore diameter, D, with the pressure applied to force liquid into it, P [Where \( \Theta \) is the contact angle of the fluid and \( \gamma \) is the surface tension of the liquid]

The Washburn equation defines the minimum pore diameter that will be penetrated by a liquid with known properties at a defined pressure (see equation 1). By measuring at multiple pressures and allowing the system to equilibrate as required at each point, it is possible to determine the incremental volume associated with pores of a given diameter to generate a pore size distribution for the sample, a plot of the volume of pores in any given pore size class. Associated metrics such as median pore size can then be determined from this plot. Washburn’s equation assumes that pores are cylindrical in shape, more specifically that the opening to the pore is a perfect circle. On the basis of this assumption pore wall area can also be calculated and where a figure is available for pore length such as the thickness of the sample, for example, the total pore wall area can used to calculate number of pores.

Advanced Porosimetry

Further investigation of the information that can be extracted from mercury porosimetry data has focused on exploitation of the observed differences between intrusion and extrusion behavior. While an intrusion curve is measured by progressively increasing pressure the corresponding extrusion curve is measured by reducing pressure, from the maximum value back down to a lower value. Intrusion and extrusion curves typically exhibit hysteresis, a phenomenon largely rationalized with reference to pore shape and connectivity.

For example, a large cavity connected to the surface by a small throat fills when the pressure is sufficient to force mercury through the throat. During extrusion, the throat will empty at the pressure at which it filled but the larger cavity will remain filled, with internal forces insufficient to extrude the mercury through the smaller throat at the relatively high pressure. The cavity will empty at the lower pressure associated with its radius, or potentially not at all if the path to the surface is composed of pores of inappropriate sizes.

The modelling of intrusion and extrusion behavior can therefore yield insight into, for example, the relative size of pore throats to pore cavities, permeability, pore tortuosity and tortuosity factors, a topic discussed in detail in reference 1. Tortuosity is the ratio of the actual distance travelled between two points in a sample relative to the minimum distance between the same points, while tortuosity factor is the ratio of tortuosity to constriction. Tortuosity factor characterizes the efficiency of fluid diffusion through a porous medium and is therefore particularly relevant in heterogeneous catalysis. The Reverberi method is covered here by way of illustrating the techniques that can be applied with advanced, appropriately specified instrumentation, and the information generated.

The Reverberi method [2] is based on the assumption that hysteresis in mercury intrusion/extrusion data derives exclusively from the existence of inkwell-shaped pores as oppose to the cylindrical pores assumed by the Washburn equation. Inkwell-shaped pores can be characterized by two dimensions: a cylindrical pore throat radius and the radius of the cylindrical pore cavity. The Reverberi method determines the distribution of throat sizes connected to a cavity of specific size.

From a practical perspective, a Reverberi porosimetry measurement begins with intrusion to a maximum pressure \( P_z \) to fill all accessible porosity. Pressure is then reduced to \( P_y \), a pressure that corresponds to a capillary size \( R_y \). This reduction of pressure empties pores and pore cavities with diameters between \( R_y \) and \( R_z \), and the volume of cavities
that lie in this range can be determined by comparing the cumulative intrusion/extrusion curves. Pressure is increased again back to $P_z$ but the intrusion curve will not retrace the original if there is trapped mercury in the pores.

Reducing pressure from $P_z$ to a new pressure $P_x$ where $P_y < P_x < P_z$ empties throats and cavities in the size range $R_y$ to $R_x$ and as the pressure is raised again to maximum a new intrusion path is formed which retraces none of the preceding paths. Repeating this process ultimately produces a three-dimensional data set quantifying the distribution of pore cavity volumes as a function of cavity sizes behind each of a series of pore throat sizes.

Gas Adsorption: detailed characterization into the microporous region

Physical gas adsorption, the gold standard technique for measuring the surface area of a solid, can also be used to characterize porosity. Though slower than mercury porosimetry, gas adsorption offers more benign health and safety characteristics and, crucially, the ability to measure into the microporous region, to measure pore sizes less than 0.5 nm in size. This is of particular interest for materials such as zeolites, activated carbons and MOFs. Characterizing porosity by gas adsorption involves measuring the amount of gas – typically nitrogen – adsorbed by a sample, as a function of pressure, up to the point of complete pore filling.

In a gas adsorption measurement, a manifold is charged to a specified pressure, at known temperature (see figure 6). The manifold is then opened to a degassed, decontaminated sample to allow gas adsorption into the sample. Once the pressure has equilibrated the volume of gas adsorbed is calculated by difference using the Ideal gas law, from the known volume of the apparatus and precise temperature and pressure measurements. Making measurements at progressively higher pressure produces sequential points on an adsorption isotherm, a plot of quantity of gas adsorbed versus relative pressure, equilibrium or sample pressure ($p$) divided by the reference pressure which is commonly taken as the saturated vapor pressure of the sorptive gas ($p_{sat}$).

![Figure 6: The amount of gas physically adsorbed onto a sample can be determined from precise measurements of the equilibrium pressure that develops when a charged manifold (left) is opened to the sample (right), provided that the volume and temperature of the apparatus are known.](image)

![Figure 7: At higher pressures the pores of the sample fill generating data that can be used to characterize porosity.](image)
Figure 7 shows a simplistic model of the progression of a gas adsorption measurement at the molecular level. At lower pressures gas molecules adsorb sparsely across the surface of the sample (Stage 1), progressively creating a (theoretical) monolayer as pressure is increased (Stage 2). Analysis of data generated during these stages provides surface area information, but for porosity characterization it is necessary to further increase pressure, to relative pressures in excess of 0.4 up to near saturation (p/p₀ ≈ 1). This induces multilayer coverage (Stage 3) and ultimately complete pore filling (Stage 4). Of course, pore filling and surface coverage are not discrete sequential processes, with micropores typically filling before a complete monolayer has formed. Furthermore, as multilayer coverage establishes, mesopores can fill via capillary condensation. When estimating the size of pores within the sample it is therefore critical to take account of the fact that they are filling via two mechanisms (Figure 8).

\[
\ln[p/p_0] = -\frac{2\gamma v_l}{RT (r - t)}
\]

**Equation 2:** The Kelvin model correlates relative pressure \([p/p_0]\) with the radius of a pore, \(r\) [Where \(\gamma\) is the surface tension of the condensed sorptive, \(v_l\) is the molar volume of the condensed sorptive gas, \(R\) is the gas constant and \(T\) is the analysis temperature]

As relative pressure approaches one, all the pores, to a pore size of around 300nm become filled; the sample is saturated. The total pore volume can be determined from the cumulative adsorbed volume from the y-axis of the adsorption curve, at this point, by applying the Gurvitch rule (see equation 3) which converts the quantity of gas adsorbed into a liquid volume. This liquid volume is taken as being equivalent to the total pore volume.

\[
TPV = \Phi q_{aTOTAL}
\]

**Equation 3:** The Gurvitch Rule converts the total quantity of gas adsorbed \((q_{aTOTAL})\) into a liquid volume \((V)\) [Where \(T\) is the analysis temperature, \(P\) is the pressure at the end of the analysis, and \(\Phi\) is a numerical constant dependent on the properties of the sorptive - specifically liquid molar volume at the analysis temperature.]

**The role of NLDFT modelling**

All models have their limitations and those of the BJH model have become progressively more evident in recent decades. From the 1990s onwards our ability to synthesize materials with controlled microporous structures has improved considerably and applications for these materials have increased correspondingly. Over the same period there have been marked improvements in the accuracy of gas adsorption measurements. Together these advances have revealed that the precision of popular models such as BJH reduces considerably at pore sizes below ~ 7 – 8 nm. Certain models, as exemplified by that of Horvath-Kawazoe and of Dubinin, can be successfully applied to calculate pore size distributions in the microporous region. However, the use of two models to handle materials that have structure at both the meso-macroporous and microporous level is problematic since the models are discontinuous. In contrast, NLDFT (non-local density function theory) provides a single model that is effective from the micro through to the macroporous region.
In simple terms NLDFT is a computational modelling tool used widely in solid state physics to calculate physical properties from molecular structures/sub-atomic interactions. In gas adsorption applications it is used to predict fluid density within the pore, under the conditions applied during measurement. One of the primary reasons that classical models break down is that in very fine pores the condensing phase, typically nitrogen, does not exhibit conventional liquid-like behavior, largely due to interactions between the pore surface and the molecules of the sorptive. With an NLDFT gas adsorption model, calculated fluid density values are used in combination with proposed models of pore structure to generate theoretical isotherms for a given system. The result is a ‘kernel’ or a series of theoretical isotherms describing the behavior of a material at a specific temperature, as a function of pore size. Comparing these kernels with measured data enables the selection a suitable model for the material of interest.

To generate a pore size distribution for a specific sample, measured isotherms are mathematically compared with the theoretical isotherms of a chosen kernel to determine a distribution that best fits the measured data. From a practical perspective this means that there are two important aspects to the application of NLDFT. Firstly, there is construction of the models themselves, the development of a library of different models that can effectively represent the materials routinely encountered. This is a complex, mathematical task that remains a work in progress. Recent, notable advances include the development of pore models with surface energy heterogeneity and corrugated, rather than smooth walls, that give improved results for specific activated carbons [3]. Secondly there is the rigorous mathematical process of optimally fitting the model to the experimental data. In Micromeritics systems this is achieved using the SAIEUS (Solution of Adsorption Integral Equation Using Splines) program.

While many gas adsorption users know very little about NLDFT models, their construction and solution, this brief overview highlights the potential value of investing in instrumentation that has supported capability in this area.

**Porometry: characterizing through pores**

Porometry covers a comparable pore size range to mercury porosimetry but unlike all preceding techniques it is specifically for the characterization of through porosity, for measuring the size and size distribution of pores that extend from one side of the sample to the other. In essence, porometry measures only those pores which allow for passage of a fluid through the material (see figure 9) – typically in a direction normal (z) to the surface of the solid (although complex flow patterns such as “in plane” or x,y are also measurable).

Key applications are therefore for the analysis of filters, membranes, including hollow fibres, and materials used to make medical supplies such as woven cloth for masks and gowns. For these applications, porosity measurements based on volume, and or those methods which assess closed, non-transport pores, are often invalid. It is for this reason that the filtration and separations market has been dominated by porometry, while curiously, other industries which may benefit from this method have remained largely unaware.

The underlying principle of the technique is the determination of pore characteristics by measured displacement of a fluid from a saturated sample; “traditional” gas-liquid and a more recently advanced “liquid-liquid” porometry method can be applied.

![Figure 9: In porometry gas flow rate is measured as a function of pressure to characterize pore emptying characteristics and by extension pore dimensions (Where d = dry curve, w = wet curve, d/2 = half dry curve, FBP = First Bubble Point, MFP = Mean Flow Pore and SP = Smallest Pore.](image-url)

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In gas-liquid, or capillary force porometry, pressurized gas is used to displace the wetting liquid. A good wetting liquid preferably has a zero degree contact angle (perfectly wets the solid), low surface tension, low viscosity, and low vapor pressure to ensure easy and complete impregnation of the sample as well as to avoid evaporation and other time-sensitive effects. It should also be chemically inert and not cause sample swelling. Fluorinated hydrocarbons, as exemplified by the perfluoroethers Porefil and Galpore meet these criteria. For other samples, silicone oils or even alcohols may be utilized.
During measurement the sample is initially saturated with wetting liquid which is then displaced by applying a flow of gas. Initially, no flow is registered. Once the pressure corresponding to the largest pore has been reached, the sample will allow first flow. This is also known as the “bubble point” (ASTM F316) – an important data point because often this predicts the “absolute rating” in terms of separations for a material i.e. no particle, virus, or bacterium larger than the largest pore will have passage. Progressively finer pores empty as gas pressure is increased and measuring the flow rate of gas as a function of pressure therefore generates a ‘wet curve’ for the sample (w), as shown in figure 9 and figure 10. Once all the liquid has been displaced gas flow is again measured as function of applied pressure, this time for the dry sample, to generate the ‘dry curve’ (d). The ‘half dry’ curve (d/2) is produced by dividing the flow values from the dry curve by 2.

The pressure required to expel liquid from a pore is related to its diameter via the Young LaPlace equation which is closely similar to the Washburn equation. On this basis we can scrutinize the curves generated in porometry to determine the metrics that can be determined. A key parameter is first bubble point (FBP), the point at which gas flow is first registered, as noted above. On the other hand, the wet curve meets the dry curve when the substrate is empty of liquid i.e. the smallest pore has been emptied. The pore size distribution can be determined from the pressure range defined by these two points. The mean flow pore diameter can be determined from the intersect with the half dry curve and is the pore size at which 50% of the total gas flow can be accounted for, in other words, half the flow is through pores larger than this in diameter. Other parameters that can be generated include gas permeability (if material area and thickness are known) and metrics specifically associated with filtration performance.

Liquid-liquid porometry is closely similar but enables the characterization of materials with finer porosity, with pores as small as 2nm, and/or the use of more moderate pressures to test materials with pores in the submicron size range. As the name suggests it relies on the use of two immiscible liquids, a wetting liquid, as in gas-liquid porometry, and a displacement liquid which is usually water (see figure 11). The parameters generated are exactly analogous to gas-liquid porometry but extend to total pore number and total pore area.

With both forms of porometry measurement accuracy relies on the precision of the pressure and flow rate sensors since these are the only two parameters measured. Choosing technology built with sensors that offer the highest available resolution therefore maximizes the value of the technique for sensitive porosity characterization.
In conclusion

Porosity is a performance defining physical property of many industrial materials and there are multiple, powerful techniques to characterize it. Choosing a technique that is well-matched to the structure of your sample and that robustly provides sufficient detail is the key to generating porosity data that optimally supports a given application.

References:

