

# On the universal behavior of sorption isotherms in disordered mesoporous solids

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## Abstract

Adsorption–desorption isotherms in disordered mesoporous solids, described by the Dual Site-Bond Model, are obtained through Monte Carlo simulations and their behavior is correlated to the topological properties of the porous networks and to their percolation properties, extending previous results to the general case of variable connectivity networks. A quasi-universal curve is found which may be useful in the problem of obtaining pore size distributions from the analysis of experimental Adsorption–desorption isotherms.

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## 1. Introduction

There is a great variety of physicochemical processes occurring in porous media (in general disordered porous media), which are strongly affected by the morphological and topological characteristics of the porous structure, like, for example, the invasive displacement of a fluid by another (with important applications to oil recovery), imbibition and drying processes, separation of fluid mixtures, heterogeneous catalysis and catalyst deactivation, etc. [1–6]. The study of this kind of systems is exciting due both to the need for the development of new theoretical methods for the understanding of their behavior, as well as to the potentiality of their applications to processes of practical interest. In spite of an intense activity in this field during the last two decades, there are still important aspects which need to be solved or more deeply investigated, in particular, the connection between the genesis of the porous medium and its morphological and topological properties, the way the structure of the medium, represented, for example, by spatial corre-

lation, affects the percolation properties and the behavior of critical exponents, the development of a methodology to obtain the morphological and topological properties from experimental determinations (the problem of *characterization* of the porous medium), specially for mesoporous materials, the way these properties affect the behavior of fluids in the medium and the way in which some processes are affected when the porous space reduces to nanoscopic dimensions (micropores). The present work is an effort to contribute to the understanding of the fundamental phenomena in this field and to the discussion of applications of practical interest.

Among the above problems, the characterization of mesoporous and macroporous materials, especially regarding the determination of the pore size distribution from experiments, is a subject of great practical importance involving the development of both theoretical and experimental methods. Some of the experimental methods, like NMR, SAXS, and SANS, require sophisticated instruments while others, like porosimetry and Adsorption–desorption of vapors, use much simpler apparatus, which can practically be available to any laboratory. Therefore, it is of special interest to count on a reliable theoretical background for the analysis of Adsorption–desorption or intrusion–retraction experiments. However, the development of such a theory still

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stands as an open problem presenting interesting theoretical challenges [7–11], and this is more remarkable in the case of disordered (or amorphous) porous media. In fact, the shape and extent of the Adsorption–desorption hysteresis loop (ADHL) of vapors in mesoporous materials, or the intrusion–retraction hysteresis loop (IRHL) of mercury in mesoporous and macroporous ones, are known to be influenced by several characteristics of the porous space: the geometrical shape of the pores, their size distribution, and the interconnectivity of the porous network are among those, which have been studied intensively for a long time.

The problem has two aspects: in first place, a model describing the properties of the medium must be given, and then, within that model, a procedure to determine the pore size distribution must be developed.

As for the first aspect, modeling of porous media has evolved along two different, but complementary, lines: continuum and discrete models. Continuum models considering the medium as a composite material, described by a continuous characteristic function attaining the value 0 at an empty point and 1 at a solid point, have proven to be more adequate to study the flux of fluids through the medium [12–16]. On the other hand, discrete models, representing the porous space by a network of voids (sites) connected by throats (bonds), have demonstrated to be a powerful tool to study the percolation properties of the medium and those phenomena depending on its connectivity properties [4,9,17–24]. Among the family of discrete models, the Dual Site-Bond Model (DSBM), introduced by Mayagoitia et al. [25,26] is the simplest model, which takes into account spatial correlation among pore sizes, allowing in this way to generate porous networks with different structures. The DSBM describes the porous solid as a network of sites (corresponding to the main voids of the porous space) connected by bonds (corresponding to the necks between adjacent voids). Within the framework of this model, through analytical calculations on a Cayley tree (where no closed loops are involved) [27] and through Monte Carlo simulations in two-dimensional networks [28], it has been shown that spatial correlation among pore sizes affect drastically percolation probabilities. It is then to be expected that in more realistic three-dimensional networks, spatial correlation will have similar effects on the percolation probabilities, and these, in turn, will affect the ADHL.

As for the second aspect, procedures to obtain the site and bond distributions have been determined satisfactorily so far only for non-correlated porous networks and in the extreme cases where the pore volume can be attributed entirely to the sites or entirely to the bonds [11,29,30]. We remark that real disordered mesoporous materials are hardly conceivable as completely random media and that the determination of percolation probabilities in three-dimensional correlated porous networks is a non-solved problem.

Recent Monte Carlo simulation studies of ADHL on correlated networks generated through the DSBM [31,32] have shown that, when representing the data as the mean bond

radius versus the relative pressure at the desorption branch knee, all data corresponding to different site and bond distributions collapse on a single universal curve, and that this fact can be used to determine the site and bond size distributions from experimental ADHL. These results were obtained for constant connectivity networks.

Since many disordered porous media are more realistically represented by variable connectivity networks, the purpose of the present work is to investigate how the universality behavior is affected by a variable connectivity. In Section 2, we briefly review the DSBM and the simulation method to obtain ADHL. Results about the behavior of ADHL and universality are presented in Section 3, and finally, conclusions are given in Section 4.

## 2. Dual Site-Bond Model (DSBM)

Let  $S(R)$  and  $B(R)$  be the distribution functions associated with the site and bond size  $R$ , and  $F_S(R)$  and  $F_B(R)$  the corresponding probability density functions, such that [25]

$$S(R) = \int_0^R F_S(R') dR'; \quad B(R) = \int_0^R F_B(R') dR' \quad (1)$$

and let the intervals  $s = [s_1, s_2]$  and  $b = [b_1, b_2]$  be the support of site and bond measures, i.e., the set of values of  $R$  for which  $F_S$  and  $F_B$  are positively defined. The way in which sites and bonds are connected to form the network is given by the joint probability density function,  $F(R_S, R_B)$ , of finding a site with size  $R_S \in (R_S, R_S + dR_S)$  connected to a bond with size  $R_B \in (R_B, R_B + dR_B)$ . The two basic laws describing the DSBM are [25,26]

$$B(R) - S(R) \geq 0 \quad (2)$$

$$F(R_S, R_B) = 0 \quad \text{for } R_S < R_B \quad (3)$$

The first law, Eq. (2), implies that  $b_1 \leq s_1$  and  $b_2 \leq s_2$ , while the second law, Eq. (3), called the *Construction Principle* (CP), is of a local nature and expresses the fact that the size  $R_B$  of any bond cannot be bigger than that of the two connected sites (in a porous medium the size of a throat cannot be larger than that of the two connected voids).

If the joint probability function is expressed as

$$F(R_S, R_B) = F_S(R_S) F_B(R_B) \Phi(R_S, R_B) \quad (4)$$

then the correlation function  $\Phi$  carries the information about the site-bond assignment procedure in the network. In the simplest case, where sites and bonds are assigned to each other in the most random way as allowed by the CP, called the *Self Consistent* case, then  $\Phi(R_S, R_B)$  attains the following expression [25]

$$\Phi(R_S, R_B) = \frac{\exp\left[-\int_{R_B}^{R_S} (dB/B - S)\right]}{B(R_B) - S(R_B)} \quad (5)$$

Eq. (5) is deduced by following a particular method of site and bond assignment: first, sites with smaller sizes are

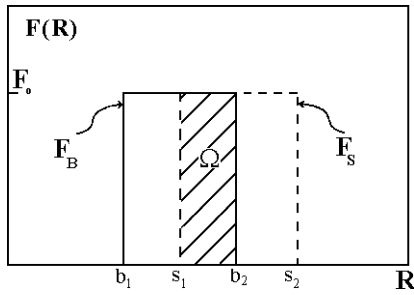


Fig. 1. Uniform site and bond size probability densities, showing schematically the overlapping between the two distributions.

assigned to a set of bonds, each of which can adopt a random size, although restricted to the condition indicated by the CP, and in accordance with the available distribution of bonds. The procedure follows in such a way as to continue the exhaustion of sites, each time larger and larger.

If we denote by  $\Omega$  the overlapping area between the site and bond probability density functions, as shown in Fig. 1 for the simple case of uniform distributions, the function  $\Phi$  has the following properties: (i)  $\Phi_{\Omega \rightarrow 0}(R_S, R_B) = 1, \forall R_S, R_B$ , sites and bonds are distributed completely at random, and (ii)  $\Phi_{\Omega \rightarrow 1}(R_S, R_B) \propto \delta(R_S - R_B), \forall R_S, R_B$ , sites and bonds group together in macroscopic patches, each having a value of  $R$ . Then, the overlapping  $\Omega$  is the fundamental parameter describing the topology of the network in this model.

This behavior also suggests that  $\Omega$  must be related to some *correlation length* (which would be a physically more meaningful parameter), characteristic of the decay of the spatial correlation function defined as:

$$C(r) = \langle R_S(\vec{r}_0) R_S(\vec{r}_0 + \vec{r}) \rangle = \langle R_B(\vec{r}_0) R_B(\vec{r}_0 + \vec{r}) \rangle \quad (6)$$

In fact, it is expected that  $C(r)$  decays approximately in an exponential form (this would be the exact behavior for a one-dimensional network generated by a Markov chain of events).

$$C(r) \approx \exp\left(\frac{-r}{l_0}\right) \quad (7)$$

where  $l_0$  is the correlation length (measured in lattice constants). Monte Carlo simulations have shown that the correlation length  $l_0$  is related to the overlapping  $\Omega$  through the relation [33]

$$l_0 \approx \frac{2\Omega^2}{(1 - \Omega)^2} \quad (8)$$

We observe that  $l_0 \rightarrow 0$  for  $\Omega \rightarrow 0$  and  $l_0 \rightarrow \infty$  for  $\Omega \rightarrow 1$ .

The problem of numerical generation of DSBM networks has been intensively investigated [4]. We employ here the method presented in [34] for the Monte Carlo generation of such networks, which can be resumed in the following very simple terms. An initial network is prepared by sampling the values of  $R_S$  and  $R_B$  from the corresponding probability density functions  $F_S$  and  $F_B$  and distributing them completely

at random on the lattice. This network will have the correct  $F_S$  and  $F_B$  but not the correct  $\Phi(R_S, R_B)$ , in particular, the CP is not obeyed everywhere. Then, a Markov chain of new states of the network is generated by choosing at random pairs of sites (or bonds) attempting to exchange them, the exchange is accepted if it does not violate the CP. It has been demonstrated [34] that this procedure leads finally to the equilibrium distribution for the network and that it does not suffer from the imperfections introduced by other methods (mainly anisotropy). In order to generate a network with locally variable connectivity (i.e. the precise number of bonds that can surround each site throughout the network) and a mean connectivity  $\bar{Z}$  (i.e. the average number of bonds that delimit a site), an appropriate fraction of bonds with  $R_B = 0$  is initially assigned at random in the network, and then the usual procedure described above is followed [35].

Once a network with the desired properties has been generated, ADHL can be simulated by following the method described in [31], which can briefly be described as follows. For the adsorption branch, at any given value of the relative pressure  $p/p_0$ , an adsorbed thickness is calculated through, for example, the Halsey equation, and the elements of the porous network (sites and bonds) are inspected, so that those who have reached the critical Kelvin radius for capillary condensation to occur are filled. For the desorption branch, at any given value of the relative pressure, a particular filled element must fulfill two conditions in order to be evaporated: (i) it must have the appropriate radius (dictated by the Kelvin equation), and (ii) it must be connected to the vapor phase. The evaporated element is then left with an adsorbed thickness corresponding to the given relative pressure.

### 3. Results and discussion

Our results were obtained for the sorption of  $N_2$  at 77 K on a cubic network under the following assumptions: (i) sites are considered to be of spherical geometry, while bonds are cylinders; and (ii) the pore volume is mainly contained in the sites. Different kinds of pore size distributions (sites and bonds) were tested, in particular: (i) Gaussian distribution, with mean values  $S_m$  and  $B_m$  for sites and bonds, respectively, and the same standard deviation  $\sigma$ . The limits for sampling radii for sites and bonds were chosen to be  $S_m \pm 2\sigma$  and  $B_m \pm 2\sigma$ , respectively. (ii) Gamma distribution, of the form  $\Gamma(R) = (R - R_0)^{n-1} e^{-(R-R_0)}$ , truncated at the upper limit at the value  $R_0 + 2n$ . The values of site and bond radius at maximum probability ( $S_m$  and  $B_m$ , respectively) are obtained in each case as  $R_0 + (n - 1)$ . For a given set of ADHL, the mean site radius was kept fixed and the overlapping  $\Omega$  was varied by changing the mean bond radius.

A reduced set of typical ADHL, where the relative adsorbed volume  $V$  is plotted as a function of the relative pressure  $p/p_0$ , is presented in Fig. 2, for site and bond Gaussian distributions with  $S_m = 11$  nm,  $\sigma = 0.8$  nm. The left column corresponds to non-correlated networks with  $B_m = 4.5$  nm,

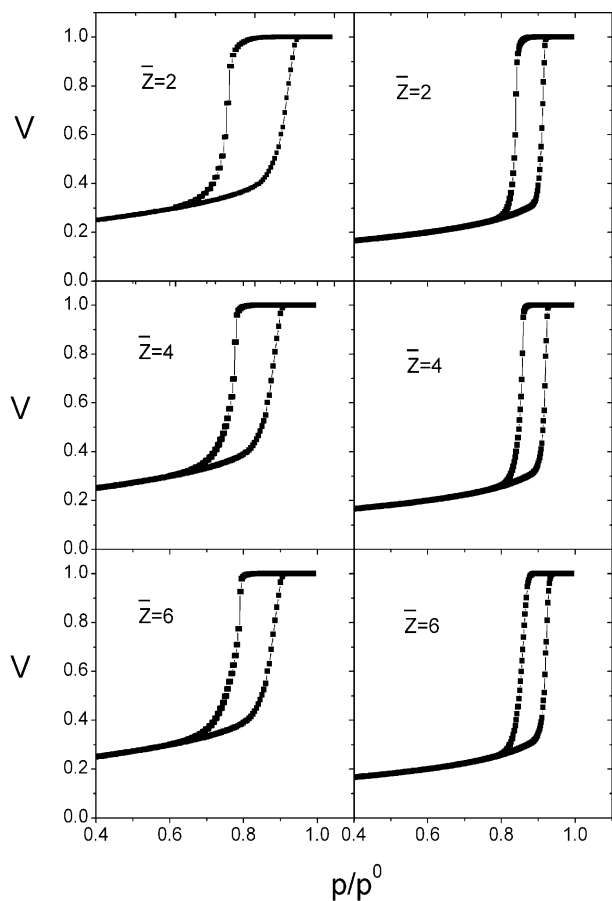


Fig. 2. Adsorption–desorption  $N_2$  isotherms at 77 K for site and bond Gaussian distributions with  $S_m = 11$  nm,  $\sigma = 0.8$  nm. Left column:  $B_m = 4.5$  nm,  $\Omega = 0$ . Right column:  $B_m = 7.1$  nm,  $\Omega = 0.5$ .

resulting in  $\Omega = 0$ , while the right column is for correlated networks with  $B_m = 7.1$  nm, resulting in  $\Omega = 0.5$ . The mean connectivity,  $\bar{Z}$ , changes from top to bottom through the values 2, 4, and 6. As it is well known, during the adsorption process the vapor phase accesses to the whole porous structure, and there are no percolation effects. On the contrary, during the desorption process, a given pore is allowed to evaporate at a given pressure only if two conditions are accomplished: first, its radius must correspond to the critical Kelvin radius for the given pressure, and second, the pore must be connected to the vapor phase. This last condition, introduces percolation effects on the desorption branch: the larger is the percolation threshold for the porous network the lower is the relative pressure needed to produce an appreciable desorption (in other words, the retarding in the desorption branch increases with the percolation threshold). In Fig. 2, we can appreciate that, for a given mean connectivity, the retarding of the desorption branch is smaller for correlated than for non-correlated networks. This is a consequence of the fact that the percolation threshold in correlated networks decreases with  $\Omega$  [27,28]. It can also be observed that the retarding effect increases when the mean connectivity decreases, as expected from normal percolation theory.

The above analysis of the effects of percolation on ADHL for correlated networks suggests that the position of the bond distribution, determined by  $B_m$ , and the relative pressure at the desorption knee,  $p^*$  are the two most relevant parameters.

When all results for the Gaussian and Gamma distributions, for constant connectivity  $Z = 6$ , are represented as  $B_m$  versus  $p^*$ , as it is shown in Fig. 3, it turns out that all data collapse on a single “quasi-universal” curve, given, by means of a least-square curve fitting, by the simple equation

$$B_m \text{ (nm)} = \frac{1}{1 - p^*} \quad (9)$$

where  $B_m$  should be measured in nanometers; for comparison reasons, Fig. 3 also includes a dotted line, that represents the result of plotting the radius  $B_m$  of a single bond against the relative vapor pressure at which capillary evaporation occurs in this lonely pore. The  $B_m$  versus  $p^*$  relationship represents a quasi-universal equation in the sense that, at least for the variations we have considered here, it does not depend on the shape of the size distribution (i.e. the same universal curve stands for any combination of  $B_m$ ,  $S_m$ , and  $\sigma$  of the twofold site-bond size distribution) or on the position of the site distribution, or even on the adsorption process considered (i.e. cooperative effects involving the coalescence of liquid–vapor menisci proceeding from  $Z-1$  bonds for condensation in an adjacent site to happen [27]). The universality is limited by the facts that we considered a network whose pore volume is essentially attributed to the sites and that we considered the weakest possible form of correlation, i.e. the one imposed by the CP and given by the DSBM. At the present time, we do not have a theoretical or approximate scaling justification of this equation and it should be taken as an empirical law.

In the more general case of variable connectivity, it is found that the value of  $\bar{Z}$  strongly affects the behavior and different variables must be used to make that all data collapse on a single curve, as shown in Fig. 4, where each symbol corresponding to a given connectivity, also correspond to several different porous networks (i.e., with different correlations and different site and bond distributions). The dotted line in Fig. 4 represents once more the relationship between  $B_m$  and  $p^*$  for a single bond. The  $B_m$  versus  $p^*$  fitting for variable site connectivity gives rise to a more general quasi-universal curve given by the empirical equation

$$B_m \text{ (nm)} + 0.22\bar{Z} = \frac{p^*}{(1 - p^*)^{1.81}} + 6.5 (p^*)^{1.7} \quad (10)$$

Eq. (9) can now be used to propose a characterization method, which we believe is a first realistic approach to obtain the site and bond size distributions from experimental ADHL of vapors for disordered mesoporous materials. The method can be described through the following steps:

- (i) Obtain the size distribution for sites from the experimental adsorption branch. This, assuming that the volume associated with bonds is negligible, can be eas-

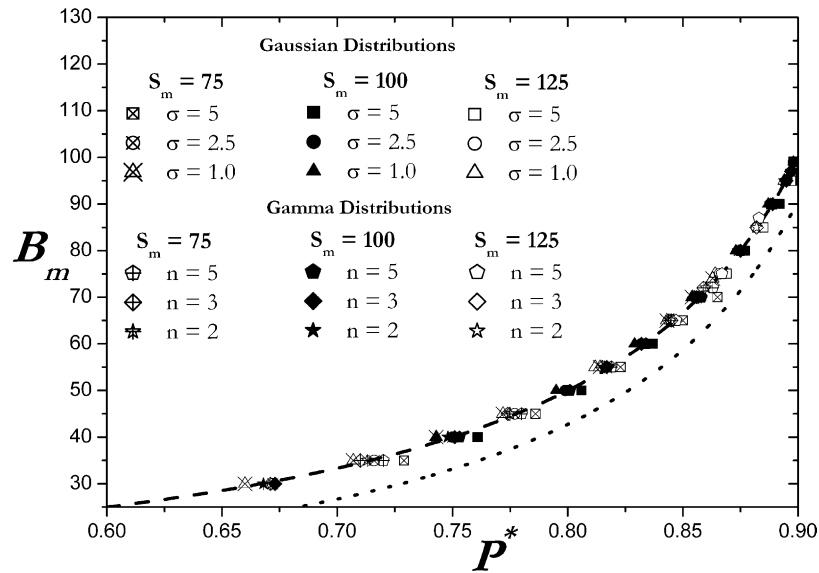


Fig. 3. Quasi-universal behavior for constant connectivity  $Z = 6$ . All pore sizes are given here in Angstroms. The dotted line represents the result of plotting the radius  $B_m$  of a single bond (assumed as a cylindrical pore open at both ends) against the relative vapor pressure at which capillary evaporation from this lonely pore occurs.

- ily achieved from a differential analysis of that branch [3,8].
- (ii) Independently of the shape of the distribution obtained for the sites, assume the same shape for the bond distribution. This is not a too strong restriction, considering that other quite stronger restrictions are necessarily made in any method, like the one regarding the pore geometry (sites and bonds).
- (iii) Obtain  $p^*$  from the experimental desorption branch and calculate  $B_m$  from Eq. (9). This will give the positioning of the bond distribution.

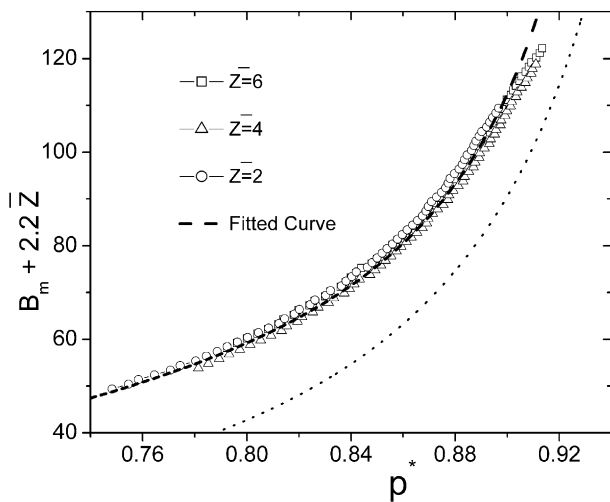


Fig. 4. Quasi-universal behavior for variable connectivity. Pore sizes are given here in Angstroms. The dotted line represents the result of plotting the radius  $B_m$  of a single bond (assumed as a cylindrical pore open at both ends) against the relative vapor pressure at which capillary evaporation from this lonely pore occurs.

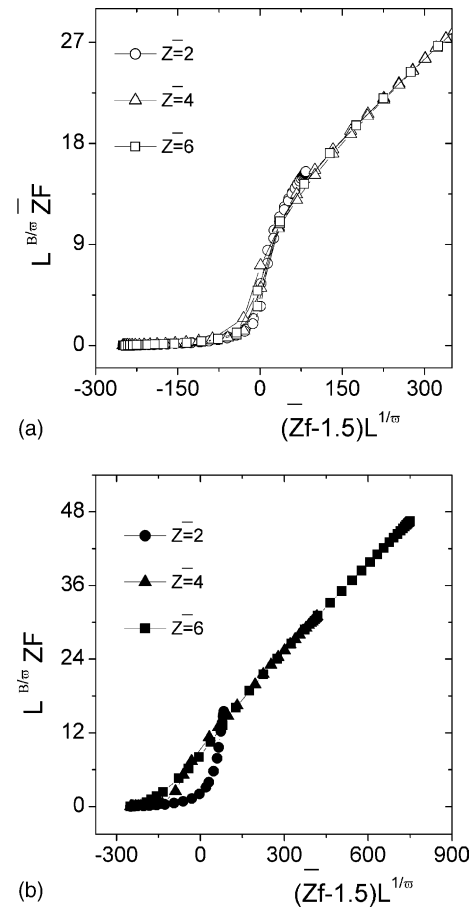


Fig. 5. Test of Seaton's scaling behavior: (a) non-correlated networks,  $\Omega = 0$ ; (b) correlated networks,  $\Omega = 0.5$ .



However, this is valid only for constant connectivity  $Z = 6$ . For the more general case of variable connectivity,  $\bar{Z}$  should be first determined by some independent method, and then Eq. (10) should be used in place of Eq. (9). One of the methods to determine the mean connectivity is the one based on lattice size scaling, due to Seaton [2,10]. In order to test such a scaling behavior, we have repeated the simulations of ADHL for different lattice sizes,  $L$ , and plotted the quantity  $L^{\beta/\nu} \bar{Z} F$  versus  $(Zf - 1.5)L^{1/\nu}$ , where  $F$  is the fraction of evaporated bonds at a given pressure,  $f$  is the fraction of bonds whose radius satisfies Kelvin equation at that pressure, and  $\beta$  and  $\nu$  are the usual critical exponents for normal percolation. Fig. 5(a) shows the results for uncorrelated networks ( $\Omega = 0$ ), where we see that all data corresponding to networks with different connectivities scale correctly collapsing on a single curve. For correlated networks ( $\Omega = 0.5$ ), however, this does not happen, as shown in Fig. 5(b). We therefore conclude that Seaton's scaling law is not valid for correlated networks, and that further theoretical developments are necessary in order to determine the mean connectivity through lattice size-scaling methods.

#### 4. Conclusions

We have studied through Monte Carlo simulation how ADHL are affected by the topological characteristics, closely related to the percolation characteristics, of correlated three-dimensional porous networks described by the DSBM for Gaussian and Gamma size distributions. We have in addition assumed that the pore volume in our networks resides mainly in the sites, whereas bonds only contribute to percolation effects. The use of the DSBM has the advantage that, once fixed the shape of site and bond size distributions, the topological characteristics of the porous network depend on a single parameter, the overlapping  $\Omega$  between those distributions, closely related to the correlation length of the network, Eq. (8).

A wide range of the parameters has been investigated and we have found out that all results can be conveniently represented in terms of two main variables:  $p^*$ , the relative pressure at the desorption knee in the ADHL, and  $B_m$ , the size of maximum probability in the bond distribution. By using these two variables, all results scatter closely around a characteristic quasi-universal curve, Eq. (9) for constant connectivity  $Z = 6$ , or Eq. (10) for general variable connectivity networks. These findings provide us with a method to determine the site and bond distributions for correlated networks from the analysis of experimental ADHL, provided that the mean connectivity can be determined independently.

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