

Pore size distributions of complex systems

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An approach based on the sum of integral adsorption isotherm equations including independent distribution functions for each component of complex adsorbents and solved by using self-consistent regularization was developed and tested for mechanical mixtures of carbon and silica adsorbents. For individual adsorbents, e.g. carbons or silica gels, the developed procedure gives the pore size distributions close to those calculated using the DT method.

1. INTRODUCTION

Determination of structural and adsorption characteristics of any adsorbent is a non-trivial problem [1-4], not to mention complex or hybrid adsorbents because the topology of pores of different components of, e.g., carbon-mineral adsorbents or composites with polymers and metal oxides is strongly different, as well as the surface potentials [5-8]. The use of the same adsorption potential and the same pore model for different components of hybrid adsorbents leads clearly to significant errors in the determined parameters. Previously we used the sum of integral equations solved to obtain the pore size distributions for complex adsorbents using the sum of the integral adsorption equations with one distribution function [5-7]. The aim of this paper is to show an improved pathway to describe complex adsorbents using independent distribution functions of the pore size for each phase and the solution of the sum of integral adsorption equations using self-consistent regularization with the control of the correspondence of the pore model to the real pore topology using a criterion linked to deformation of the pore shape compared with the model [6].

2. COMPUTATIONAL PROCEDURE

The pore size distributions (PSDs) $f_V(x)$ (differential distribution function $f_V(x) \sim dV_V/dx$) of carbon adsorbents can be calculated with overall equation in the form proposed by Nguyen and Do (ND method) [9, 10]:

$$a = \int_{r_{\min}}^{r_k(p)} f_V(x) dx + \int_{r_k(p)}^{r_{\max}} \frac{w}{x - \sigma_{sf}/2} t(p, x) f_V(x) dx \quad (1)$$

where r_{\min} and r_{\max} are the minimal and maximal half-widths of pores respectively; $w = 1$ for slitlike pores; $r_k(p)$ is determined by the modified Kelvin equation:

$$r_k(p) = \frac{\sigma_{sf}}{2} + t(p, x) + \frac{w\gamma\mathcal{W}_m \cos\theta}{R_g T \ln(p_0/p)}, \quad (2)$$

and $t(p, x)$ can be computed using the modified BET equation:

$$t(p, x) = t_m \frac{cz}{(1-z)} \frac{[1 + (nb/2 - n/2)z^{n-1} - (nb+1)z^n + (nb/2 + n/2)z^{n+1}]}{[1 + (c-1)z + (cb/2 - c/2)z^n - (cb/2 + c/2)z^{n+1}]}, \quad (3)$$

where

$$t_m = a_m/S_{BET}, \quad (4)$$

$$b = \exp(\Delta\mathcal{E}/R_g T), \quad (5)$$

$$c = c_s \exp((Q_p - Q_s)/R_g T), \quad (6)$$

$$c_s = \gamma e^{\frac{E-Q_L}{R_g T}}, \quad (7)$$

$\Delta\mathcal{E}$ is the excess of the evaporation heat due to the interference of the layering on the opposite pore wall ($\Delta\mathcal{E} \approx 2.2$ kJ/mol [9, 11]); $t(p, x)$ is the statistical thickness of the adsorbed layer; a_m is the BET monolayer capacity; c_s is the BET adsorption coefficient on flat surface, Q_L is the liquefaction heat, E is the adsorption energy, γ is a constant; Q_s and Q_p are the adsorption heat on flat

surface and in pores respectively; $z = p/p_0$; n is the number (non-integer) of statistical monolayers of adsorbate molecules, and its maximal value for a given pore half-width x is equal to $(x - \sigma_{sf}/2)/t_m$; and $\sigma_{sf} = (\sigma_s + \sigma_f)/2$ is the average collision diameter of surface (carbon) and fluid (nitrogen) atoms. These equations can be modified to be used for adsorbents characterized by, for instance, cylindrical pores (silica gels) or gaps between spherical particles (fumed oxides, carbon black). Different surface potentials were used on calculations of Q_s and Q_p for nitrogen in slitlike pores (Steele potential) [9, 10], gaps between spherical particles [6, 7], and cylindrical pores (Lennard–Jones potential) [12–15]. Steele potential was used for the calculations of Q_s and Q_p for nitrogen molecule in slitlike pores [9, 10]

$$U(x, y) = \varphi(y) + \varphi(x - y), \quad (8)$$

with

$$\varphi(y) = 4\pi\rho_s\sigma_{sf}^2\varepsilon_{sf}\Delta\left[0.2\left(\frac{\sigma_{sf}}{y}\right)^{10} - 0.5\left(\frac{\sigma_{sf}}{y}\right)^4 - \frac{\sigma_{sf}^4}{6\Delta(y + 0.61\Delta)^3}\right], \quad (9)$$

$\Delta = 0.3354$ nm is the thickness of a nitrogen monolayer, and y is the distance from the central plane of the outermost atom layer of one pore wall. The solid–fluid interaction in cylindrical pores can be determined by [12]

$$U(r, R) = \pi^2\rho_s\varepsilon_{sf}\sigma_{sf}^2\left[\frac{63}{32}\left[\frac{r}{\sigma_{sf}}\left(2 - \frac{r}{R}\right)\right]^{-10}F\left[-4.5, -4.5, 1, \left(1 - \frac{r}{R}\right)^2\right] - \left[3\left[\frac{r}{\sigma_{sf}}\left(2 - \frac{r}{R}\right)\right]^{-4}F\left[-1.5, -1.5, 1, \left(1 - \frac{r}{R}\right)^2\right]\right] \quad (10)$$

where $F[\alpha, \beta, \gamma, \chi]$ is the hypergeometric series, r is the radial coordinate, ε_{sf} is the surface–fluid parameter in the LD potential, and ρ_s is the density of surface atoms. In the case of pores as gaps between spherical particles, eq 2 should be used in the form

$$\ln \frac{p_0}{p} = \frac{\mathcal{W}_m}{R_g T} \left[\frac{1}{r_k} - \frac{2}{\sqrt{(R + t' + r_k)^2 - R^2} - r_k + R + t'} \right], \quad (11)$$

where R is the radius of nanoparticles, and $t' = t + \sigma_{sf}/2$.

The nitrogen desorption or adsorption data can be utilized to compute $f_V(x)$ distributions with eq 1 using regularization procedure [16] CONTIN [17, 18] modified to the mentioned equations (i.e. modified ND-CONTIN (MNDC) method) under non-negativity condition ($f_V(x) \geq 0$ at any x) with a fixed or non-fixed regularization parameter α .

To consider different types of porosity (slitlike and cylindrical pores or gaps between spherical nanoparticles) simultaneously, integral equation 1 can be rewritten as follows

$$a_\Sigma = \sum_i c_i a_i = \sum_i c_i \left[\int_{r_{\min}}^{r_{k,i}(p)} f_{V,i}(x) dx + \int_{r_{k,i}(p)}^{r_{\max,i}} \frac{w}{x - \sigma_{sf}/2} t_i(p, x) f_{V,i}(x) dx \right], \quad (12)$$

where $c_i = c_{slit}$, c_{cyl} and c_{sph} are weight constants determining contributions of slitlike and cylindrical pores or gaps between spherical particles to the total adsorption (i.e. porosity), using the corresponding modified Kelvin equations (eqs 2 or 11). Eq 12 could be solved using two approaches: (i) $f_{V,slit}(x) = f_{V,sph}(x) = f_{V,cyl}(x) = f_V(x)$ (i.e. monoregularization with respect to overall $f_V(x)$ using the MNDC method); and (ii) $f_{V,slit}(x) \neq f_{V,sph}(x) \neq f_{V,cyl}(x)$ with binary or ternary self-consistent (subsequent for $f_{V,i}(x)$ at $i = slit, cyl, and sph$) regularization with respect to different types of pores (initial $f_V(x)$ could be calculated with the monoregularization) [5, 16].

The $f_V(x)$ distributions determined with eqs 1 or 12 and linked to the pore volume can be transformed to the distributions $f_S(x)$ with respect to the specific surface area using the corresponding models of pores

$$f_S(x) = \frac{w}{x} \left(f_V(x) - \frac{V_p}{x} \right), \quad (13)$$

where $w = 1, 2,$ and 3 for slitlike, cylindrical, and spherical pores respectively. However, the relationship for $f_S(x)$ and $f_V(x)$ is more complex for pores as gaps between spherical particles, since the inner volume of aggregates of primary particle plays the role of pores but both outer and inner surfaces of these aggregates contribute the specific surface area. For a cubic lattice with spherical nanoparticles $w \approx 1.36$; however, this value increases for a denser hexagonal lattice. For estimation of deviation of the pore shape from slitlike one $\Delta w_{slit} = S_{BET}/S_{sum,slit} - 1$, eq 13 could be used at $w = 1$ for calculations of $f_S(x)$ for the model of slitlike pores with [6]

$$S_{sum,slit} = \int_{x_{\min}}^{x_{\max}} f_S(x) dx = \int_{x_{\min}}^{x_{\max}} \frac{w}{x} \left(f_V(x) - \frac{V_p}{x} \right) dx, \quad (14)$$

In the case of the mixture of pores

$$\Delta w_{total} = \frac{S_{BET}}{\sum_i \int_{R_{min}}^{R_{max}} \frac{w_i}{R} (f_{V,i}(R) - \frac{V_p}{R}) dR} - 1, \quad (15)$$

Different versions of the described approach were used for investigations of various individual (carbons, silica gels, fumed silicas) and complex adsorbents such as carbon-mineral and polymer-mineral composites [5-8, 18-37]. In this paper we used two complex systems with mechanically mixed (I) fumed silica A-300 (Pilot plant of the Institute of Surface Chemistry, Kalush, Ukraine; $S_{BET} = 232 \text{ m}^2/\text{g}$, $V_p = 0.557 \text{ cm}^3/\text{g}$) and activated carbon PS1 (PSO MASKPOL, Poland, $S_{BET} = 877 \text{ m}^2/\text{g}$, $V_p = 0.445 \text{ cm}^3/\text{g}$) as 1 : 1 (Fig. 1); and (II) fumed silica A-300, silica gel Si-60 (Merck, $S_{BET} = 447 \text{ m}^2/\text{g}$, $V_p = 0.8 \text{ cm}^3/\text{g}$), and graphitized carbon black Envicarb (Supelco, USA, $S_{BET} = 98 \text{ m}^2/\text{g}$, $V_p = 0.447 \text{ cm}^3/\text{g}$) as 1 : 1 : 1 (Fig. 2).

Individual carbon adsorbents WVA (wood based activated carbon, Westvaco) [19, 30] and Carboxen 569 (carbon sieve, Supelco) [6] were used to compare results of calculations using DFT [38] and MNDC methods.

3. RESULTS AND DISCUSSION

Pore size distributions (PSD) were calculated using the sum of integral equations (12) corresponding to each component using a complex model of slitshaped (labeled Slit) pores for activated carbon and carbon black; cylindrical pores (labeled Cyl) for silica gel, and gaps between spherical particles (labeled Sph) for fumed silica. Additionally, for the mixture I, self-consistent regularization (SCR) was used. The comparison of the PSDs of individual adsorbents and their mixtures (Fig. 2) shows that application of a simple model of pores can give inappropriate distribution functions; for instance, the model of slitshaped pores gives the PSD whose peaks are displaced in comparison with the corresponding peaks of individual adsorbents (we assume that the individuality of these adsorbents remains because their mechanical mixing was careful). On the other hand, application of the complex model including pore models corresponding to all the components of the mixture gives the PSD while maintaining shapes of the corresponding components (Figs 1 and 2).

However, the PSD of the mixture and the corresponding PSD of individual components are not identical because mixing and pre-treating (degassing at 200°C) samples can slightly change their complex texture and morphology.

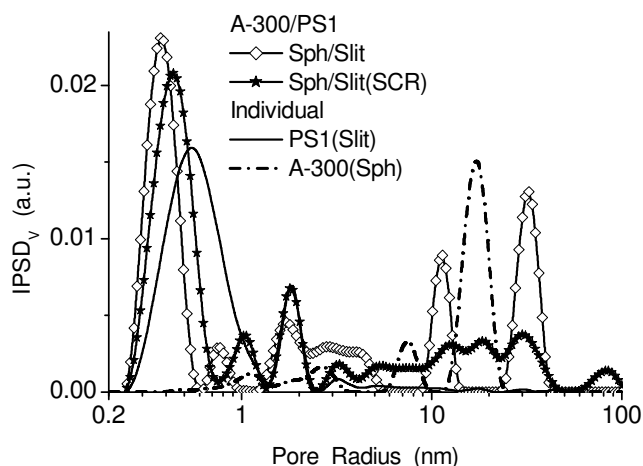


Fig. 1. Incremental PSDs for mechanical mixture of fumed silica A-300 and activated carbon PS1 (1 : 1) calculated using a complex model of pores with the contribution of gaps between spherical particles (A-300) and slitshaped pores (PS1) with SCR or standard regularization without self-consistency; and for individual adsorbents using the model of slitshaped pores for PS1 and the model of gaps between spherical particles (cubic lattice) for fumed silica.

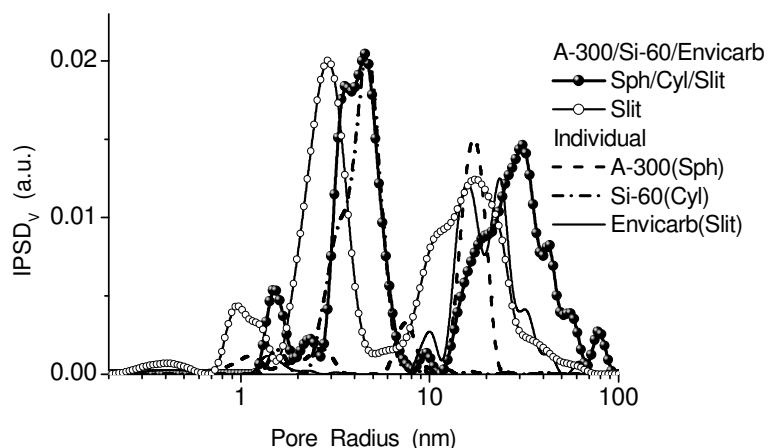


Fig. 2. Incremental PSDs for mechanical mixture of fumed silica A-300, silica gel Si-60 and carbon black Envicarb (1 : 1 : 1) calculated using a complex model of pores with contribution of gaps between spherical particles (A-300), cylindrical pores (Si-60) and slitshaped pores (Envicarb); and for individual adsorbents using the model of: gaps between spherical particles (cubic lattice) for fumed silica; cylindrical pores for Si-60, and slitshaped pores for Envicarb. Additionally, the model of slitshaped pores was used for the mixture.

Additionally, the accessibility of the surfaces of different components can differ from their percentage. The SCR method can improve the PSD for complex systems because it gives a better fitting of the isotherm. In this approach, contributions of pores of different shapes are varied for the best correspondence of the theoretical isotherm to experimental one. The developed procedure was used [5] to describe such adsorption characteristics as adsorption energy distributions [11, 39-42]. Consideration for the difference in the nature of components of complex adsorbents is very important for an accurate description of the energetic characteristics of adsorbents.

Notice that the developed MNDC procedure brings the PSD close to that computed one using the DFT method (Figs 3 and 4).

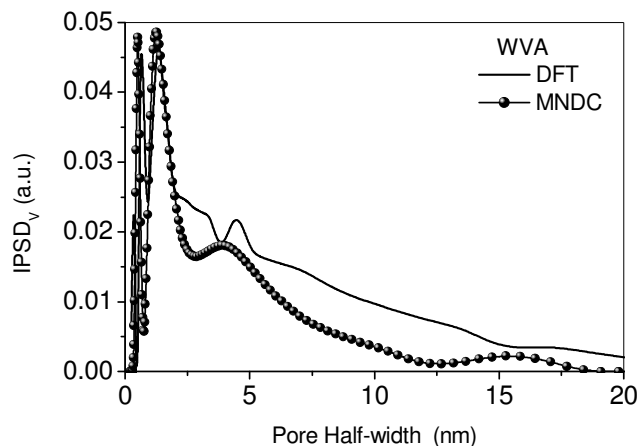


Fig. 3. PSDs for activated carbon WVA [17] calculated using the DFT and MNDC methods.

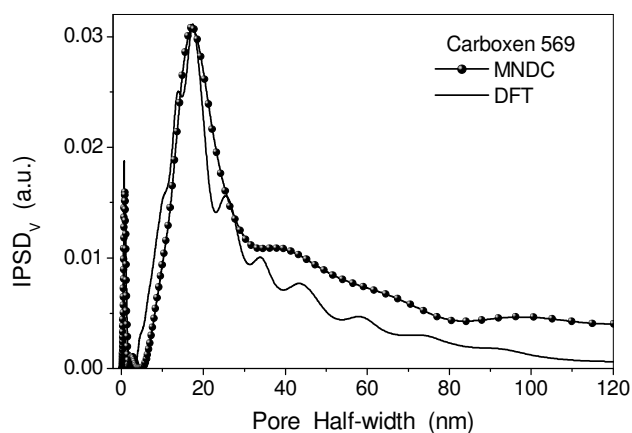


Fig. 4. PSDs for activated carbon Carboxen 569 (Supelco) calculated using the DFT and MNDC methods.

The deviation of the pore shape from the model is small $\Delta w_{total} = 0.043$ for the mixed model with slitshaped pores and gaps between spherical particles applied to the mixture with A-300/PS1 (Fig. 1) and $\Delta w_{slit} = 0.507$ in the case of the use of slitlike pore model for this complex adsorbent.

4. CONCLUSIONS

The developed procedure with SCR gives more reliable pore size distributions for complex adsorbents than standard methods. The later are based on the integral isotherm equation including certain adsorption potential for a given pore model developed for individual adsorbents and, therefore, inappropriate for hybrid adsorbents including texturally and chemically different components.

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