

The Effect of Air Humidity on the Flow Properties of a Powder of Glass Beads

G. Landi, D. Barletta, G. Donsi, G. Ferrari, M. Poletto

Abstract – In this work the effect of air humidity on powder flow properties was studied by means of a ring shear tester. Powder sample moisture content was conditioned by humid air in a fluidization column. In spite of the very low moisture contents in the powder (<0.2%) obtained by this technique, a significant change in the powder cohesion was observed. The Kelvin equation was used to infer on the pressure increase inside the capillary bridge and the resulting typical interparticle force. This result was used to derive the powder tensile strength following the Rumpf approach and compare it with values derived from shear experiments. A single fitted value of the unknown capillary bridge gap is able to provide quantitative agreement for experiments with highly humid air. Model estimation indicates that water mainly condenses on rough surfaces of the particles and only a small portion of this condensed humidity contributes to change the powder flow properties in interparticle capillary bridges. Copyright © 2009 Praise Worthy Prize S.r.l. - All rights reserved.

Keywords: Cohesive powder behavior, Flow properties, Humidity

Nomenclature

a	bridge gap, m
C	powder cohesion, Pa
d	average particle diameter, m
d_{10}	particle diameter corresponding to the 10% in the cumulative psd, m
d_{50}	median of the psd, m
d_{90}	particle diameter corresponding to the 90% in the cumulative psd, m
F_c	tensile force of the bridge, m
P_o	vapour pressure over a planar surface, Pa
P'_o	effective vapour pressure over a curved surface, Pa
R	universal constant of gases, $J\ kmol^{-1}\ K^{-1}$
r_1	radius of the bridge axial section, m
r_2	radius of the bridge smaller cross section, m
r_a	asperity radius, m
r_m	average radius of convex curvature, m
RH	Relative humidity,
V_{bridge}	bridge volume, m^3
$V_{liq-bed}$	volume of the water condensed over a particle, m^3
X_w	water content of powders
β	angle defined in Fig. 2,
ΔP	total pressure difference between gas and liquid, Pa
ε	bulk solids porosity,
ϕ	angle of static internal friction, deg
ρ_l	liquid density, $kg\ m^{-3}$
ρ_l	molar density, $kmol\ m^{-3}$
ρ_s	particle density, $kg\ m^{-3}$
σ	normal stress, Pa

σ_1	consolidation stress, Pa
σ_c	unconfined yield stress, Pa
σ_t	tensile strength, Pa
σ_s	surface tension, Pa
τ	shear stress, Pa

I. Introduction

In powder processing industry one important issue is the handling of fine and cohesive powders, that is strongly dependent on operating conditions such as the temperature, the presence of liquids as well as on chemical and physical characteristics such as shape, roughness and particle size distribution. In fact, both operating conditions and chemical and physical characteristics may affect the flowability of granular materials and compromise the reliability of many operations related to storage and transportation as well as to powder processing, such as drying, dosing and fluidization. It is well known that among these properties humidity plays a significant role in affecting powder flowability. In fact, humidity can cause the formation of liquid bridges between particles giving rise to interparticle capillary forces. These contribute to increase the powder cohesion and negatively affect its propensity to flow. Moreover, capillary forces modify also powder bulk properties such as voidage, aggregative structure and permeability. These properties and the relative importance of capillary forces with respect to fluid dynamic forces seriously affect the fluidization behaviour of fine powders [1]-[2]. D'Amore *et al.* [3] studied the effect of powder humidity on

fluidization properties and found very different results for porous and non porous materials.

The effect of liquid bridges on powders cohesive behaviour has been studied both at the particle level and at the bulk level. At the bulk level experimental measurements of the tensile strength or of the unconfined yield strength of wet powders were carried out by split-cells and shear cells [4]-[5]. Investigated water contents were between few points percent and 30% mainly corresponding to liquid bridges in the pendular state. More recently, Johanson *et al.* [6] demonstrated that also oil contents below 0.2% can significantly affect glass beads unconfined yield strength.

The first model relating the tensile strength of moist powder agglomerates to capillary forces at the particle scale was proposed by Rumpf [7] by applying a force balance at interparticle contacts. The same approach was then followed by other authors [4]-[5] to predict the strength of wet granular materials. In particular, Schumann *et al.* [8] focused the attention on the particle contact model of two spherical particles in contact with humidity adsorption layers. According to Schumann's work, the flowability of bulk solids decreases when relative humidity increases. This behaviour is accounted for by the behaviour of a single particle in wet environment. Johanson *et al.* [6] proposed a different model to link capillary forces and the powder strength by calculating the work necessary to separate adjacent particles. In general, all the above described models depend on powder bulk properties such as voidage but also on geometrical parameters of liquid bridges and interparticle contacts, which are often more difficult to measure. Studies on surfaces with roughness has been taken. Rabinovich *et al.* [9] found that the presence of nanoscale roughness reduces the capillary adhesion between particles and in the same while it increases the critical relative humidity.

This paper presents results of the effect of air humidity on the strength of a non porous powder assessed by preliminary shear experiments. The original idea was to measure the powder shear properties in an aerated shear cell developed at the University of Salerno and to use a humid air flow for conditioning the powder. Unfortunately this approach was not feasible due to poor air distribution in the powder sample determined by the formation of a first humid layer at the cell bottom. For this reason the powder sample was moisturized in a fluidizing column with humid air at low fluidization velocities. The flow properties of moisturized powder were measured in a Schulze ring shear tester.

II. Experimental Setup

II.1. Apparatus

The fluidization column is made of glass (50 mm ID)

and is provided with a porous distributor about 5 mm thick made of sintered glass particles of about 100 μm diameter. Humidity in the air fed to the apparatus was regulated by mixing two air streams, one made of dry air and the other made of water saturated air. Dry air was obtained from the technical desiccated air provided by the departmental compressor. Water saturated air was obtained by forcing air flow through the water contained in a temperature controlled bubble trap. Air flow rates of the two streams were separately adjusted with two thermal mass flow controllers. Humidity in the mixed air stream was monitored with a relative humidity and dry bulb temperature sensor (Delta Ohm).

A Schulze ring shear tester RST-01.01 equipped with a small cell of 96 mL was used. A software application to acquire, visualize and record the main measured data was developed in LabVIEW environment (National Instrument).

Water content in powders, X_w , was measured with a thermogravimetric moisture analyzer (OHAUS MB 45).

II.2. Materials

In this work fine glass beads ($d_{10}=36\ \mu\text{m}$, $d_{50}=48\ \mu\text{m}$ and $d_{90}=65\ \mu\text{m}$, $\rho_s=2450\ \text{kg m}^{-3}$) were used in the experiments. A SEM microphotograph of this material is reported in Fig. 1, from which it appears that, in spite of the solvent washing procedure performed before the experiments, beads show a number of superficial asperities that are probably due to adhering impurities.

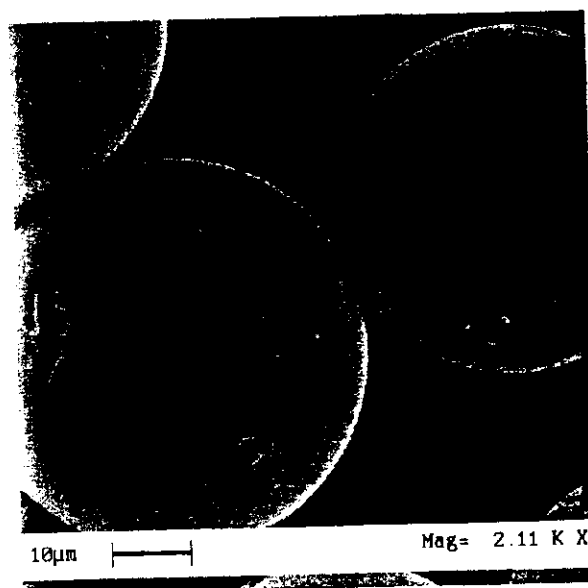


Fig. 1. SEM microphotograph of glass beads

II.3. Procedure

Batches of about 200 g of glass beads were conditioned by holding them in the fluidization column in contact with air at a certain relative humidity. The air flow was kept at values just above the minimum for

fluidization, in order to avoid the formation of preferential flow channels with water condensation. The contact time was always several hours long and generally longer than 12 hours. The sample was then fully fluidized with air at a similar humidity to that used for homogenization. Few grams of the humidified batch were used in the powder moisture analyzer to evaluate the water content and about 130 g were used to perform shear experiments. At the end of the shear experiment, the moisture of the tested powder was measured again in order to verify that moisture had not changed during the shear experiment.

A moisturized powder sample was used to evaluate a single yield locus. The procedure adopted is the standard procedure to be used with the Schulze shear tester (ASTM D6673-02). Two consolidation loads of 300 and 600g were adopted. A simplified Coulomb approach was used and each yield locus was characterized by the cohesion, C , and the angle of static internal friction, ϕ . According to one of the mostly consolidated approaches, the (σ, τ) point relative to steady state flow under the consolidation load was used to draw the Mohr circle tangent to the yield locus and to determine the major principal stress at consolidation, σ_1 . Coherently, the unconfined yield strength, σ_c , was obtained from the Mohr circle passing through the origin and tangent to the yield locus. Values of powder cohesion and angle of internal friction were used to estimate the powder tensile strength, σ_t that according to the Mohr-Coulomb approach is given by:

$$\sigma_t = -\frac{2C \cos \phi}{1 + \sin \phi} \quad (1)$$

III. Results and Discussion

Shear experiments with moisturized powder were carried out with two consolidation loads of 300 and 600g. Resulting yield loci for the 600g consolidation load are reported in Fig. 2, which shows that the moisture content causes an increase of the powder cohesion, C , while its effect on the angle of internal friction, ϕ , is weak.

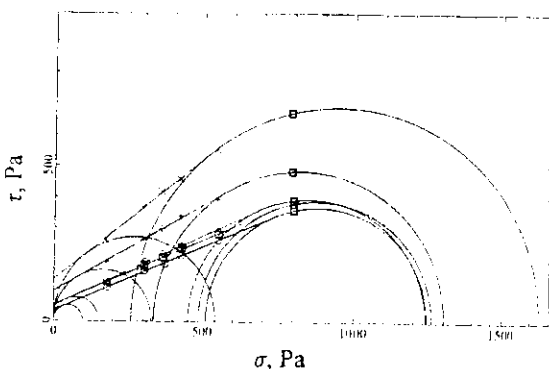
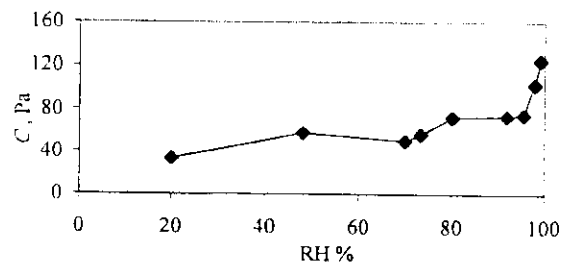


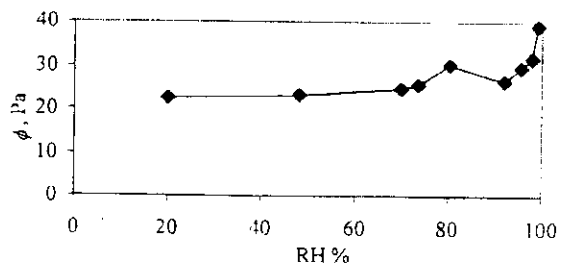
Fig. 2 Moisturized powder yield loci at 600 g normal load: ○, RH=20%; □, RH=50%; ◇, RH=70%; +, RH=91%; ×, RH=95%

These effects are made clear in Figs. 3(a) and 3(b), which respectively plot C and ϕ as a function of the air moisture content. These results are coherent with the so called theory of shift, by Pierrat *et al.* [10]. According to this theory, the effect of humidity is that of changing the powder cohesion, that is the yield locus intercept, without affecting significantly the angle of internal friction, that is the yield locus slope. A consequence of this theory is that, once a powder yield locus is known at a certain humidity, powder yield loci at different moisture contents can be obtained by measuring only one physical parameter related to the intercept such as the tensile strength, σ_t , or the cohesion C . The angle of internal friction in fact is not expected to change significantly. It is remarkable that, in spite of the fact that the theory of shift was originally verified at higher moisture contents ($1\% < X_w < 25\%$) [10], it also applies to the much lower powder moisture contents investigated in this work. In particular, direct inspection of Fig. 3 indicates that this kind of behaviour is found for $RH < 90\%$.

In fact, in this range of RH the angle of internal friction ϕ is only limitedly affected by this parameter, changing between 23° and 27° . Instead, in the same range of RH , the cohesion increases considerably rising from 33 up to 74 Pa.



(a)



(b)

Fig. 3. Powder flow properties vs air humidity for a consolidation load of 600g: (a), cohesion C ; (b), angle of static internal friction ϕ

For air RH higher than 90% both C and ϕ strongly increase with RH and the theory of shift is no more applicable. This behaviour is further confirmed by plots of the unconfined yield strength as a function of the major principal stress, σ_1 , reported in Fig. 4 and derived from shear experiments with consolidation loads of 300 and 600 g.

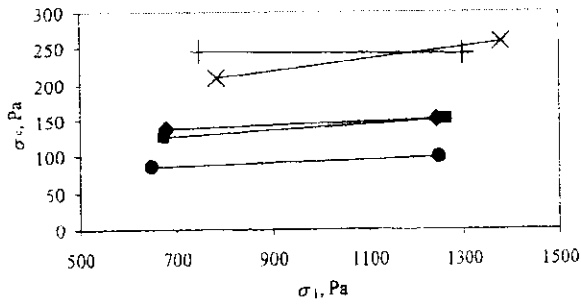


Fig. 4. Powder flow functions of at different moisture contents: ●, RH=20%; ■, RH= 50%; ◆, RH=70%; +, RH=91%; ×, RH=95%

The effect of humidity on the powder shear properties is due to a partial condensation of water under concave air-water interfaces or, more in general, interfaces which, with their shape, determine some pressure reduction within the liquid as a consequence of the action of the surface tension. When this happens the liquid vapour pressure P'_0 is reduced with respect to that measured over a planar surface P_0 . The Kelvin equation relates the reduced vapour pressure of the liquid behind the curved surface with the total pressure difference between gas and liquid, ΔP :

$$\frac{P'_0}{P_0} = \exp\left(\frac{-\Delta P}{\bar{\rho}_l RT}\right) \quad (2)$$

In Eq. (2) R is the ideal gas constant, T is the absolute temperature and $\bar{\rho}_l$ is the molar density of the liquid. At equilibrium between humid air and water we have that the water partial pressure in air is equal to the water vapour pressure within the condensed water and, therefore:

$$RH = \frac{P'_v}{P_0} \quad (3)$$

Equation (3) explains why in presence of a rough surface, also with humid air below saturation ($RH < 100\%$), we can have water condensation in all asperities or contact points which are able to produce interfacies that, with their curvature, produce an internal pressure reduction ΔP sufficient to produce, according to Eq. (2) a ratio P'_v/P_0 equal to air RH .

It was mentioned above that the total pressure difference, ΔP , between gas and liquid is determined by the action of the surface tension, σ_s , over a concave interface. This value is a function of the local average radius, r_m :

$$\Delta P = \frac{2\sigma_s}{r_m} \quad (4)$$

The combination of Eqs (2) to (4) can be used to evaluate the maximum average radius r_m that a liquid

surface can have to be stable with a certain air relative humidity. For RH of the order of 90% and for typical water surface tensions $\sigma_s = 0.073 \text{ Pa m}$, this procedure provides values of r_m of the order of 10^{-8} m and ΔP values of the order of 10^8 Pa . This value of r_m is so small that it means that any liquid bridge between particles cannot include the surface asperities shown by the glass particle on the micro-photograph in Fig. 1, that, in fact, are approximately 1 or 2 order of magnitude larger than the liquid bridge itself. This also implies that if liquid bridges form they are likely to form between asperities of adjacent particles. Therefore, in the following we will assume that liquid bridges between particles are connected to spherical objects having the asperities radius, r_a , rather than to spherical objects characterized by the particle radius. In the lack of a more precise determination we will assume r_a to be 100 times smaller than the particle diameter and, therefore it was set to $0.5 \mu\text{m}$.

The theory of capillary bridges, reported by Pierrat and Caram [11], corrects Eq. (4) for the saddle shaped surface of the typical liquid bridge as represented in Fig. 5.

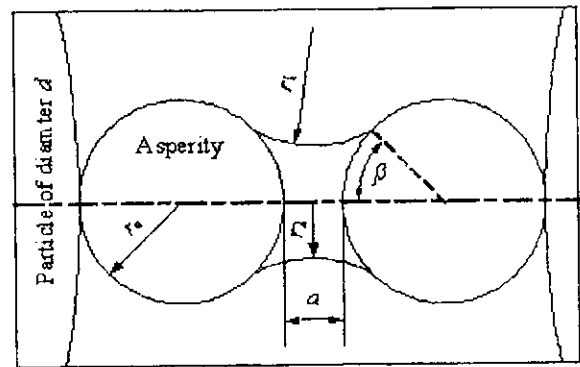


Fig. 5. Scheme and nomenclature in a the capillary bridge

This theory accounts for the radius of the bridge smaller cross section, r_2 , which has a negative effect because it is the convex curvature around the bridge axis which tends to increase the internal bridge pressure. The theory also accounts for the radius of the bridge axial section r_1 which, instead, has a positive effect because it refer to a concave curvature which tends to reduce the pressure within the bridge:

$$\Delta P = \sigma_s \left(\frac{1}{r_1} - \frac{1}{r_2} \right) \quad (5)$$

Both radii appearing in Eq. (5) can be expressed as a function of the radius of the spherical object to which the bridge is connected. As previously explained in our case the object is the particle asperity and the appropriate radius is r_a in our case. The other parameters which determine r_1 and r_2 are the angle β as defined in Fig. 5, and the gap a between the bridged objects. By

applying geometrical considerations it is possible to write the following relationships between r_1 and r_2 and the contact parameters:

$$r_1 = r_a \left[\left(1 + \frac{a}{2r_a} \right) \sec \beta - 1 \right] \quad (6)$$

and

$$r_2 = r_a \left[1 + \left(1 + \frac{a}{2r_a} \right) \tan \beta - \left(1 + \frac{a}{2r_a} \right) \sec \beta \right] \quad (7)$$

The gap a filled by the bridge is a parameter of the model and a value for it has to be assumed. Given the low moisture contents measured in the powders and the fact that below air saturation small curvature radius are required, reasonable values of a are of the same order of r_a or smaller.

Assumed a certain value for a and provided the above determined value of r_a , Eqs (2), (3) and (5) to (7) can be combined to evaluate for each air relative humidity an angle β and the bridge radii r_1 and r_2 . In turn, all these values can be used to evaluate the bridge volume, V_{bridge} , and the tensile force of the bridge, F_c , given by the sum of the effects of surface tension and pressure difference:

$$V_{bridge} = 2\pi \left\{ \frac{\left[r_1^2 + (r_1 + r_2)^2 \right] r_1 \cos \beta + \frac{r_1^3 \cos^3 \beta}{3} - r_1^2 (r_1 + r_2) \left[\cos \beta \sin \beta \left(\frac{\pi}{2} - \beta \right) \right]}{2\pi r_a} \right\} + \frac{2\pi r_a}{3(2 + \cos \beta)(1 - \cos \beta)^2} \quad (8)$$

$$F_c = 2\pi r_2 \sigma_s + \pi r_2^2 \sigma_s \left(\frac{1}{r_1} - \frac{1}{r_2} \right) \quad (9)$$

In order to relate the bridge tensile force to the powder tensile strength, we assumed that a single bridge is responsible for each contact of the particle with its neighbours also called the particle "coordination number".

According to Rumpf [7] the particle coordination number depends on the powder porosity, ε , and is of the order of π/ε . Therefore, the relationship between the force F_c with the powder tensile strength is a function of the particle diameter, d , and follows the equation proposed by Rumpf [7]:

$$\sigma_t = \frac{1 - \varepsilon}{\varepsilon} \frac{F_c}{d^2} \quad (10)$$

Given the above set of equations, the procedure to estimate the powder tensile strength starting from air relative humidity can be summarized as follows. Given RH , Eqs (2) and (3) are used to evaluate the absolute pressure reduction within the bridge ΔP . This value together with the asperity radius r_a and the assumed value for the bridge gap a are used to evaluate from the set of Eqs (5), (6) and (7) the values for the angle β and the bridge radii r_1 and r_2 . In turn these values are used to evaluate the bridge volume, according to Eq. (8), the interparticle contact force F_c , according to Eq. (9), and the powder tensile strength, according to Eq. (10). For each of the air humidity tested, Table I reports the results of the averaged values of the measured tensile strength according to Eq. (1) and of the values of the tensile strength estimated according to the above procedure starting from the air RH . In the same table are reported the bridge radii r_1 and r_2 , and the β angle. All the results depend on the bridge gap a . As indicated in the table, for all the experiments was assumed the same a value and was used as a sort of fitting parameter. The assumed for a was $0.01 \mu\text{m}$. As it is possible to observe from Table I, this single value of a is able to provide with model tensile strength values of the same order of magnitude of the experimental values. In particular, at low air humidity values ($RH < 90\%$) the model tends to overestimate the experimental tensile strength, but with a correct trend. For air $RH > 90\%$ the model prediction becomes lower than the experimental and it is not able to provide the correct trend. Both these discrepancies might be explained with a deviation of the effective number of particle contacts with respect to the Rumpf theory due to the change of volume of the condensed bridges. In particular the estimated number of contacts might be larger than real at low relative humidity and viceversa. Also the effective radius of the contact may change at high air humidity values when bridges may develop at scales larger than the asperity.

Table I reports also the liquid bridge volume calculated according to Eq. (8) compared with the water volume that condenses over each bead. According to water mass balance, this latter value is:

$$V_{liq-bead} = \frac{\pi d^3}{6} X_w \frac{\rho_s}{\rho_l} \quad (11)$$

where ρ_s and ρ_l are the solid and liquid densities, respectively. Inspection of Table I reveals that the bridge volume is significantly smaller than the liquid volume condensed over a single particle. The ratio between these two values decreases with humidity, but, in all experimental conditions tested, the ratio is always larger than 1000, suggesting that interparticle capillary bridges only represent a small portion of the total condensed water. This implies that only a small amount of this water contributes to the change of the powder shear properties. It is likely, therefore, that most of the condensed water is located within the particle surface roughness determined by asperities.

TABLE I
POWDER PROPERTIES: MEASURED (RH, X_w , σ , eq.(1)) AND ESTIMATED (ALL THE OTHERS). THE ASPERITY RADIUS WAS SET TO $r_s=0.5\mu\text{m}$

RH %	X_w %	σ , Eq.(1) Pa	ΔP 10^6 Pa	r_1 nm	r_1 nm	B deg	a μm	σ , Eq.(10) Pa	V_{bridge} 10^{-3} μm^3	$V_{\text{in-bed}}$ μm^3
20	0.04	44	222	0.3	14	1.72	0.01	79	1.26	61
48	0.04	66	101	0.7	23	2.85	0.01	93	7.70	60
70	0.10	63	49	1.4	34	4.22	0.01	99	7.70	151
73	0.07	70	42	1.6	37	4.56	0.01	101	8.88	98
80	0.14	83	31	2.2	43	5.38	0.01	101	12.38	212
92	0.11	89	12	4.5	60	8.62	0.01	91	31.95	159
96	0.09	87	6.5	10.2	88	11.53	0.01	90	57.42	129
98	0.12	109	3.0	21.5	122	16.61	0.01	81	120.70	181
99	0.16	133	3.5	17.0	113	15.20	0.01	72	100.00	242

IV. Conclusion

Experiments indicate a significant effect of air humidity on powder flow properties, even if, at equilibrium the powder moisture content was generally smaller than 0.2%.

For $RH < 90\%$ the main effect on powder flow properties is an increase of cohesion.

Significant increase of the angle of internal friction with respect to the dry powder are found only for higher values of RH . We approached theoretically the relationship between air relative humidity and powder tensile strength and set up a calculation procedure which made use of: 1) the Kelvin equation which relates liquid underpressure due to liquid surface curvature to the effective liquid vapour pressure; 2) the theory relating the liquid under pressure to the capillary bridge geometry and to the interparticle capillary force; 3) the Rumpf approach relating the interparticle forces to the powder tensile strength.

With this calculation procedure it was possible to satisfactorily estimate the powder tensile strength and compare it with the value derived from the powder flow properties.

A further finding of the application of this theoretical approach is that only a very small portion of the condensed water is active in the formation of capillary bridges which affect the powder shear properties.

This result indicates that the adopted procedure which starts from air humidity is more likely to consistently derive the powder flow properties rather than similar approaches assuming a relationship between the powder moisture content and the liquid contained within interparticle bridges.

It has to be recalled, however, that very different effects of powder moisture can be found between porous and non porous materials [3] and that, therefore the drawn conclusion may not apply to porous materials which deserve further investigation.

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