

## EXPERIMENTAL STUDY OF GAS – SOLID MASS TRANSFER IN FIXED BED OF POROUS PARTICLES

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An experimental study of gas-solid mass transfer at dynamic adsorption in fixed bed ( $H/D = 0,8$  l) is presented in this paper. The distribution of adsorbate concentration in time and in the axial direction was experimentally measured. On this basis the variation of adsorption rate and mass transfer coefficient on the fixed bed height were established. Silica gel with mean diameter of grains of  $3.57 \cdot 10^{-3}$ ,  $4.5 \cdot 10^{-3}$ ,  $5.65 \cdot 10^{-3}$  m was used as adsorbent, and air at two values of moisture content ( $10.4 \cdot 10^{-3}$ ,  $12.24 \cdot 10^{-3}$ ,  $15.72$  kg water/  $m^3$  air), and four values of flow rate ( $2.22 \cdot 10^{-3}$ ,  $3.33 \cdot 10^{-3}$ ,  $4.44 \cdot 10^{-3}$ ,  $5.55 \cdot 10^{-3}$   $m^3/s$ ) was used as gas phase. Investigations were performed at atmospheric pressure and temperature of 294 K. The results obtained show how the flow rate and moisture content of air; time and size influence the profile of water concentration in silica gel, adsorption rate and mass transfer coefficient.

### INTRODUCTION

The separation processes by adsorption of the components of a gas or liquid mixture to the purpose of recovery or purification are often met in the chemical industry or other industries. Because of the multiple industrial applications of adsorption (separating, ionic change, catalytic processes) the literature contains a great number of papers<sup>1-17</sup> that deal with this kind of processes. A range of papers treats of the gas mixture separating by static adsorption, but most of the papers refer to the separation by dynamic adsorption. Other papers present theoretical and experimental studies of mass transfer in dynamic adsorption in fixed, fluidised and mobile bed or at individual particle level.

Based on the ideal and non-ideal adsorption theories, Le Ven and others<sup>9</sup> approach the mass transfer in the case of gas mixture adsorption of components with high concentrations. Kooney<sup>11</sup> describes a simple method for the calculation of concentration profiles of adsorbent in the bed, when the adsorption takes place in fixed bed and dynamic state. Nishio and others<sup>14</sup> present the results of the theoretical and experimental researches related to the mass transfer at adsorption in fluidised bed. The authors of the paper<sup>16</sup> undertake a theoretical study of the mass transfer in adsorption at individual particle level. They developed two mathematical models considering that the profile of concentration from the interior of a particle can be represented by a parabolic expression.

In paper<sup>17</sup> the authors present an experimental study on gas drying kinetics through adsorption in a fixed bed of small height. Continuing the researches in this paper, the gas-solid mass transfer during dynamic adsorption in fixed adsorbent bed is experimentally studied. By using silica gel particles of different diameters as adsorbent and wet air with various flow rates the distribution of concentration of water vapours in the silica gel bed, in axial direction (on the bed height), and at different periods of time was experimentally determined.

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Also the variation of adsorption rate of water vapours, and the mass transfer coefficient along the fixed bed height, as a time function, was determined at different values of gaseous phase flow rate, particle size and moisture content of air.

### DETERMINATION METHOD OF THE $r_{a,i}$ AND $K_i$

This method is based on the experimental establishment of the distribution of adsorbate concentration in adsorption bed on axial direction and as a time function. Fig. 1 presents the significance of some physical quantities.

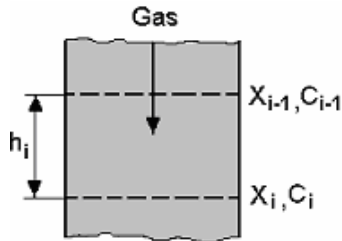


Fig. 1 – Significance of some physical quantities.

The local adsorption rate  $r_{a,i}$  is calculated as mean rate on the height,  $h_i$ , of a zone from the adsorbent bed, and it is given by:

$$r_{a,i} = \rho_v \cdot \frac{\Delta \bar{X}_i}{\Delta t} = \rho_v \cdot \frac{\bar{X}_{i|t+\Delta t} - \bar{X}_{i|t}}{\Delta t} \quad (1)$$

The local mass transfer coefficient is calculated with the equation:

$$K_i = \frac{r_{a,i}}{(\Delta C_i)_{med}} \quad (2)$$

The mean driving force can be determined as follows:

$$(\Delta C_i)_{med} = \frac{(C_{i-1} - C_{i-1}^*) - (C_i - C_i^*)}{\ln \frac{C_{i-1} - C_{i-1}^*}{C_i - C_i^*}} \quad (3)$$

The adsorbate concentration at the exit of “i” zone of the adsorption bed is given by the relation:

$$C_i = C_{i-1} - \frac{\rho_v \cdot \Delta X_{m,i} \cdot S_c \cdot h_i}{M_V \cdot \Delta t} \quad (4)$$

### EXPERIMENTAL

To accomplish the experiments a laboratory installation was used and its scheme is shown in Figure 2.

The laboratory installation consists of two cylindrical vertical columns (1), (2), a fan, and measure and control apparatuses of temperature and air flow rate. The column (1) represents the adsorption room, and it is made of stainless steel and equipped at the endings with two cone lids. At the bottom of the column (1) it is a screen of stainless steel (1a) that has the job to support the vessel with the adsorbent bed (1b). The vessel with adsorbent is introduced into the column through the inlet from its base, above the supporting screen. For the sealing of the space between the vessel (1b) and column (1) a ring garniture is used (1c). It is mentioned that the vessel (1b) is composed of a shell plate of brass having at base a screen of stainless steel. The column (2) represents the air moistening room and it is made of two cylindrical pipe sections of stainless steel joined by flanges. At frontal sides the column (2) has lids fixed with flanges. Between the pipe sections is fixed a screen of stainless steel (2) to support a packed bed of Raschig ceramic rings. The function of this bed is to retain the eventual drops carried by the air. Since at high flow rates of air the ceramic rings bed can be expanded, at the superior side is fixed another metallic screen (2c), which stops the expanding.

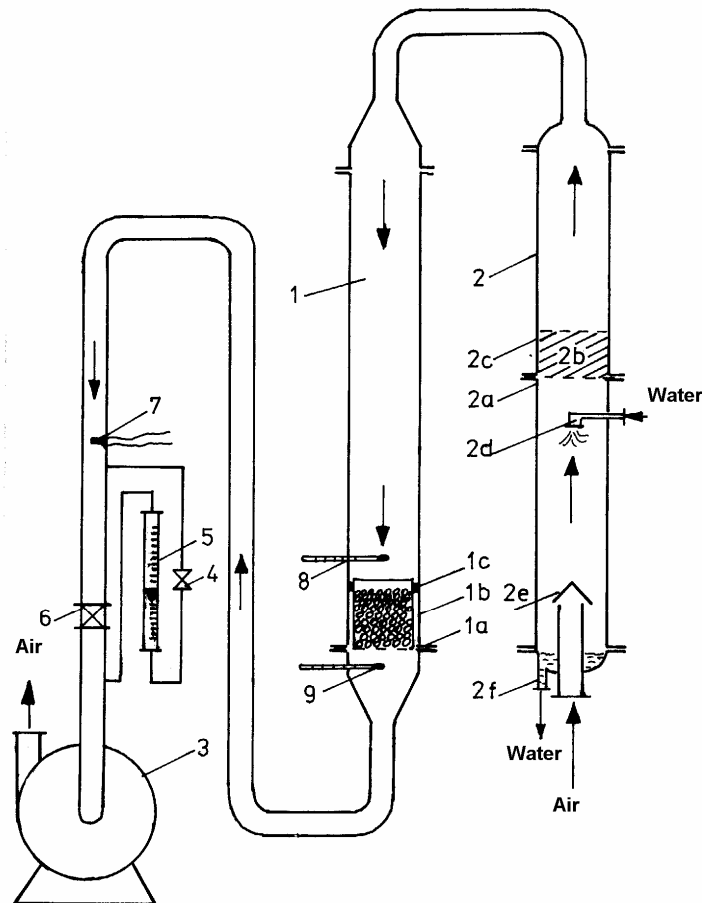


Fig. 2 – Experimental set-up. 1 – adsorption column; 2 – wetting air column; 3 – fan; 4,6 – valves; 5 – flowmeter; 7 – Pitot Prandtl tube; 8, 9 – thermometers.

To moistening the air, into the column is fixed a nozzle (2d) that provides a fine pulverization of water. At the inferior side of the column (2) is fixed a cone of stainless steel sheet (2e). The water excess of the moistening air process is evacuated through an outlet (2f) at the bottom of the column (2). Air flow rate control is realized at low values by means of the valve (4) and flow meter (5), and at high values are used the valve (6) and the Pitot-Prandtl tube (7), the last being placed in connection with a differential inclined-tube pressure gauge, which is not shown in figure 2. To measure the temperatures of the dry bulb and wet bulb thermometer there are two thermometers (8) above the adsorbent bed and other two thermometers (9) below the adsorbent bed. Both in the space above and in the space below the adsorbent bed, one of the two thermometers has the bulb covered with a cotton cloth covering, and the temperature of the wet bulb thermometer is measured.

For the experiments, silica gel particles with indefinite shape and mean diameter of  $3.57 \cdot 10^{-3}$ ,  $4.5 \cdot 10^{-3}$ ,  $5.65 \cdot 10^{-3}$  m and wet air were used. The experiments were conducted at atmospheric pressure and 294 K, at three values of the air moisture content of  $10.4 \cdot 10^{-3}$ ,  $12.24 \cdot 10^{-3}$ ,  $15.72 \cdot 10^{-3}$  kg water/ m<sup>3</sup> air and at four values of the air flow rate:  $2.22 \cdot 10^{-3}$ ,  $3.33 \cdot 10^{-3}$ ,  $4.44 \cdot 10^{-3}$  and  $5.55 \cdot 10^{-3}$  m<sup>3</sup>/s.

The adsorption process was realized in fixed bed of silica gel, in dynamic state. The geometrical parameters of the bed are:  $D = 0.086$  m and  $H = 0.07$  m.

For the purpose of determining the distribution of water vapours concentrations on the height of silica gel bed were used coloured and colourless silica gel particles but with the same size. The coloured silica gel particles were disposed in the vessel with the fixed bed and the particles with indicator were disposed at different heights of the bed (0; 0.015; 0.035; 0.055 m). The colourless particles were weighed with an analytical balance before they were introduced in the vessel. The vessel with the fixed bed coloured and colourless silica gel was weighed with a technical balance before each test. After each test were weighed the vessel with silica gel and the colourless particles after they were separated. Each time was determined the water content from the silica gel reused for adsorption.

## RESULTS AND DISCUSSION

On the base of the data obtained at weighing of the vessel with silica gel and colourless the silica gel particles and utilizing the relation:

$$X = \frac{\Delta m + m_0 \cdot x_0}{m_0(1 - x_0)} \quad (5)$$

the concentration of water from silica gel at different heights of the fixed bed can be determined. The values obtained for concentrations are represented as charts in figures 3÷6.

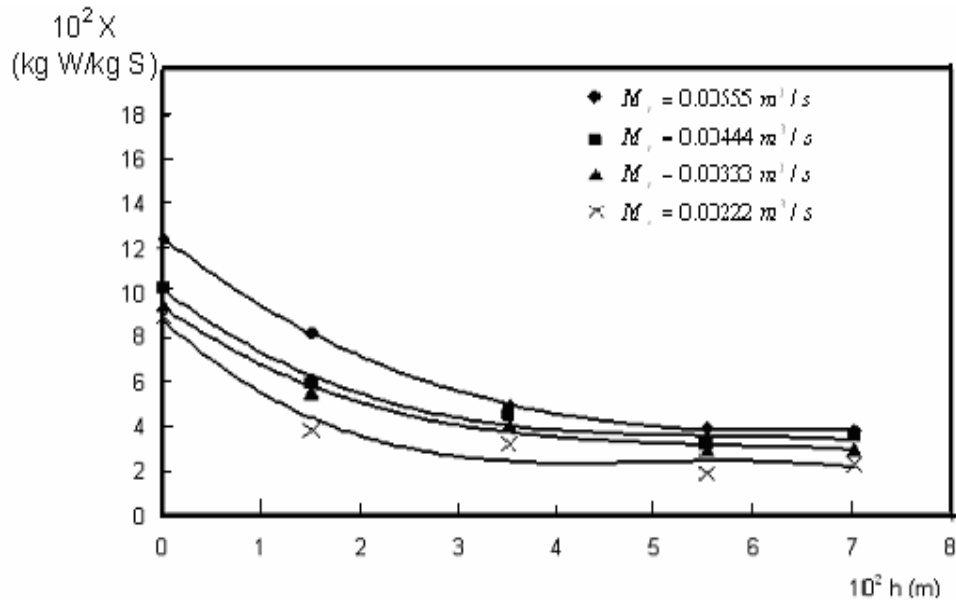


Fig. 3 – Variation of water concentration in solid phase on the height of silica gel bed,  $d=3.57 \cdot 10^{-3}$  m,  $t=300$ s,  $C_0=15.72 \cdot 10^{-3}$  kgW/m<sup>3</sup>A.

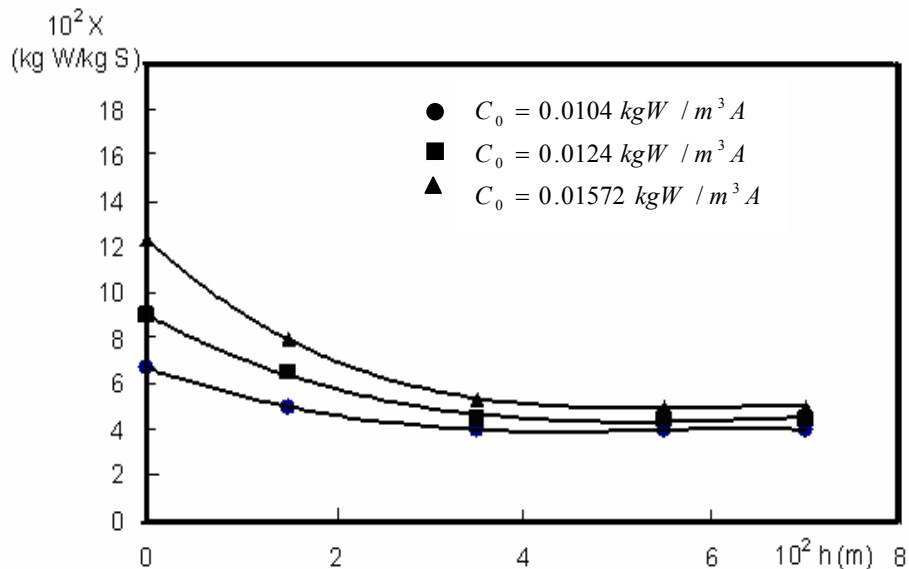


Fig. 4 – Variation of water concentration in solid phase on the height of silica gel bed,  $d = 3.57 \cdot 10^{-3}$  m,  $t=300$ s,  $M_v=5.55 \cdot 10^{-3}$  m<sup>3</sup>/s.

The data in figures 3–6 reveal a decreasing of the water concentration from silica gel with increasing of the distance given by the superior side of the fixed bed at different of the values of air flow rate, particle size, air moisture or time. The variation of water concentration,  $X$ , is more significant at the superior side of the bed.

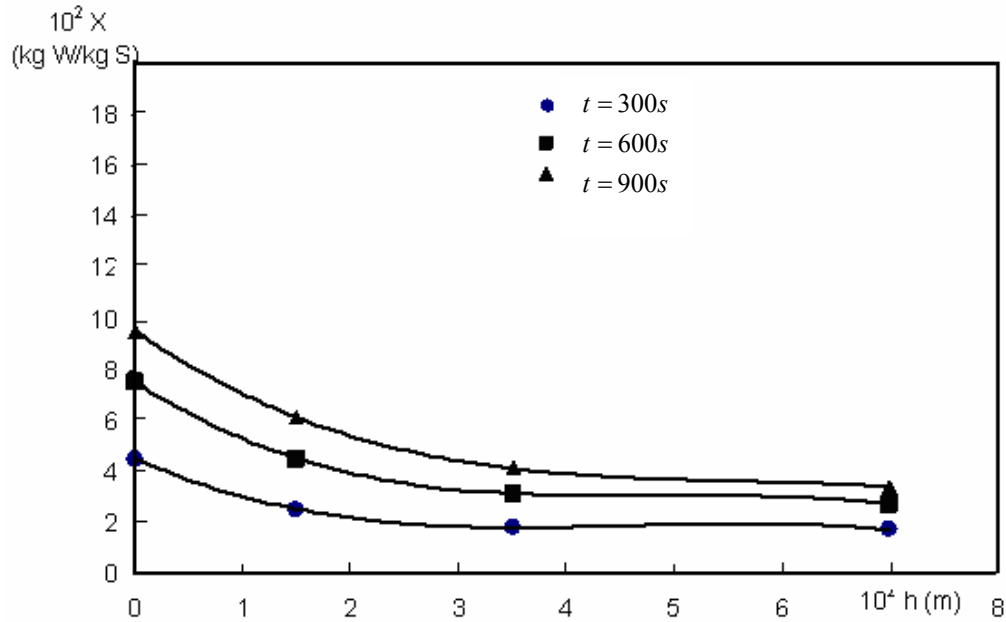


Fig. 5 – Variation of water concentration in solid phase on the height of silica gel bed,  $M_v = 3.33 \cdot 10^{-3} \text{ m}^3/\text{s}$ ,  $d = 5.65 \cdot 10^{-3} \text{ m}$ ,  $C_0 = 15.72 \cdot 10^{-3} \text{ kgW/m}^3\text{A}$ .

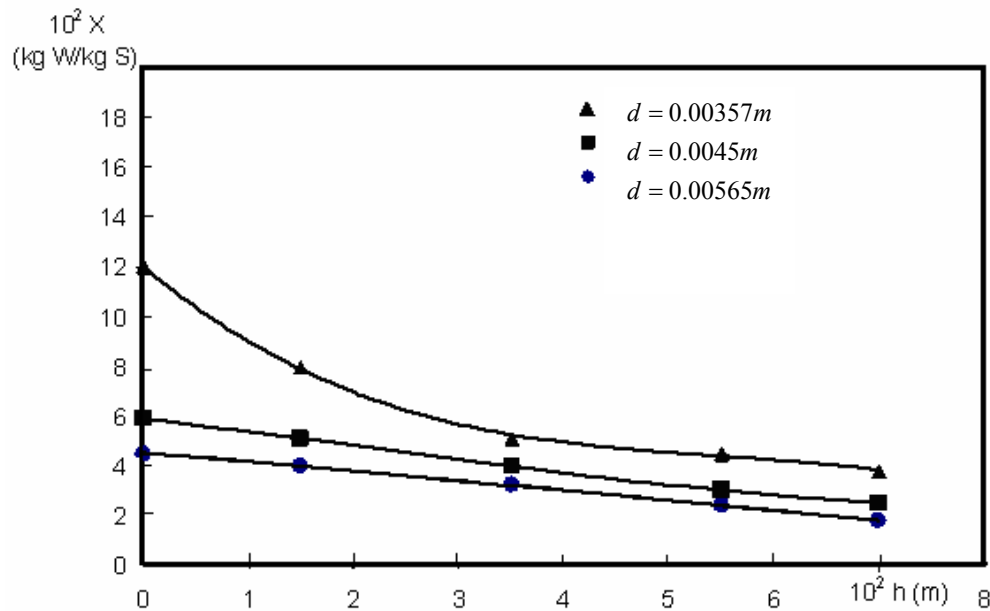
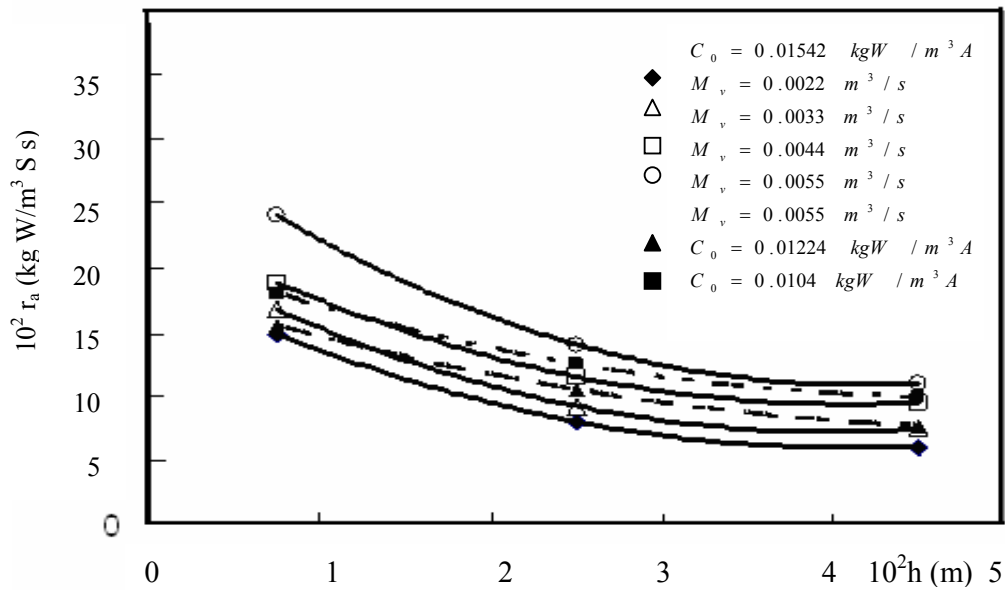
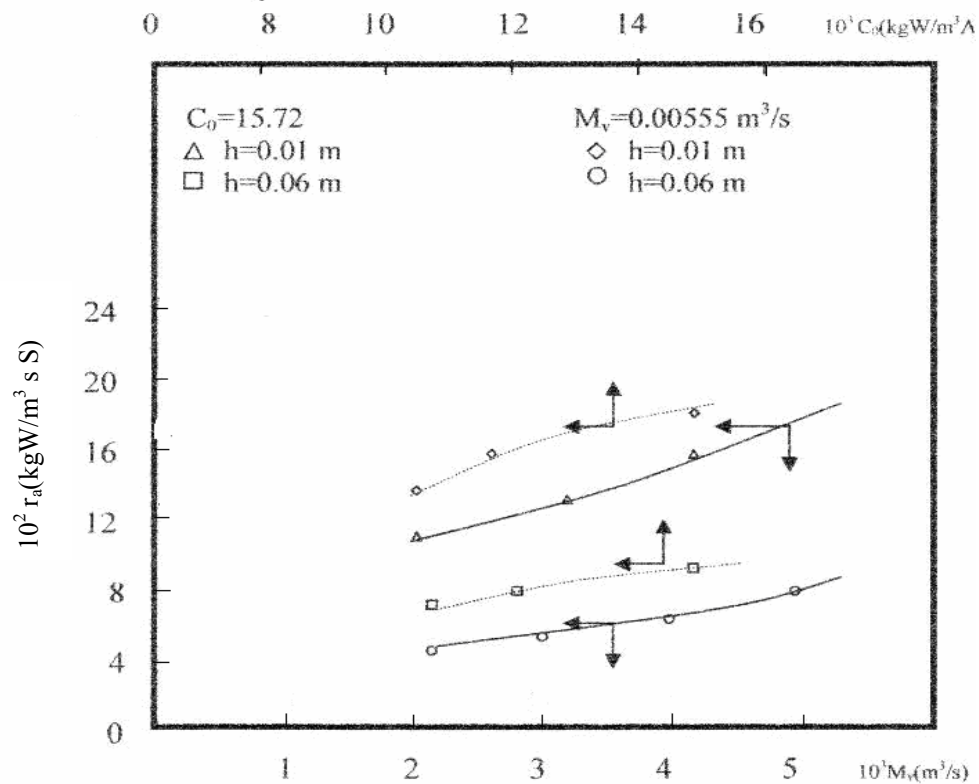


Fig. 6 – Variation of water concentration in solid phase on the height of silica gel bed,  $M_v = 5.55 \cdot 10^{-3} \text{ m}^3/\text{s}$ ,  $t = 300 \text{ s}$ ,  $C_0 = 15.72 \cdot 10^{-3} \text{ kgW/m}^3\text{A}$ .

According to figure 3, 4 the increasing of air flow rate and air moisture content leads to an increasing of the water concentration in silica gel especially at superior zone of the bed. Figure 5 shows that at bigger periods of time the water concentration in silica gel is higher than at small periods of time on the entire height of the fixed bed.

According to figure 6, the decreasing of mean diameter of silica gel particles leads to an increasing of the water concentration in silica gel on the entire height of the bed. Then, using the relation (1) it was calculated the adsorption rate. The results obtained are shown in figures 7–10.

Fig. 7 – Dependence  $r_a = f(h)$ ,  $d = 3.57 \cdot 10^{-3} \text{ m}$ ,  $t = 300 \text{ s}$ .Fig. 8 – Dependences  $r_a = f(M_v)$  and  $r_a = f(C_0)$ ,  $d = 3.57 \cdot 10^{-3} \text{ m}$ ,  $t = 300 \text{ s}$ .

As it can be seen, the profiles of adsorption rate on the fixed bed are similar to the profiles of water concentration in solid phase. Therefore, the adsorption rate of water vapours from air decreases on the height of the silica gel bed, the decreasing being more emphasized at the superior side of the fixed bed. This problem can be explained by the fact that adsorption takes place on the entire height of the silica gel bed, the

height of silica gel bed being small with a bigger intensity at the superior side of the bed. The adsorption rate is smaller and smaller in axial direction since the driving force decreases along the height of fixed bed.

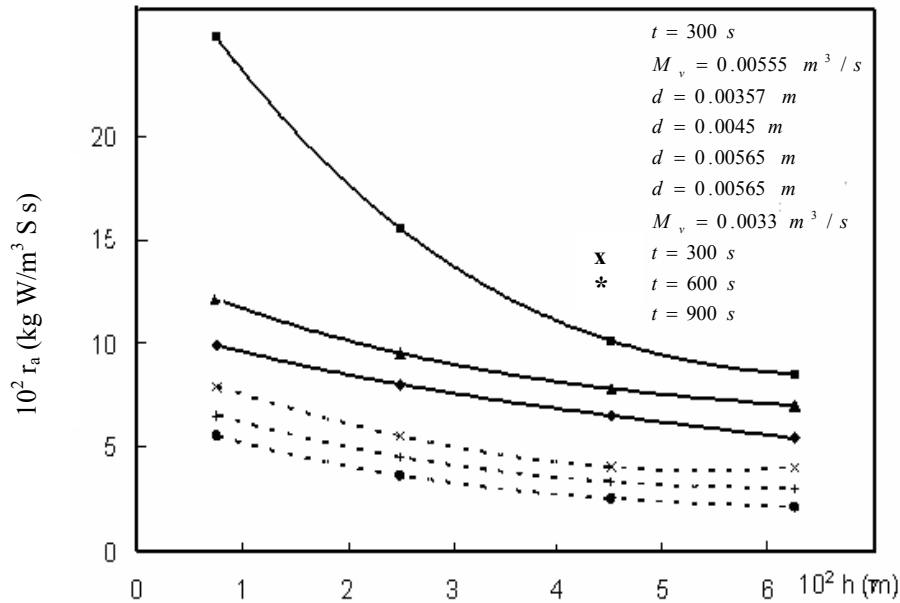


Fig. 9 – Dependence  $r_a=f(h)$ ,  $C_0=15.72 \cdot 10^{-3}$  kgW/m<sup>3</sup>A.

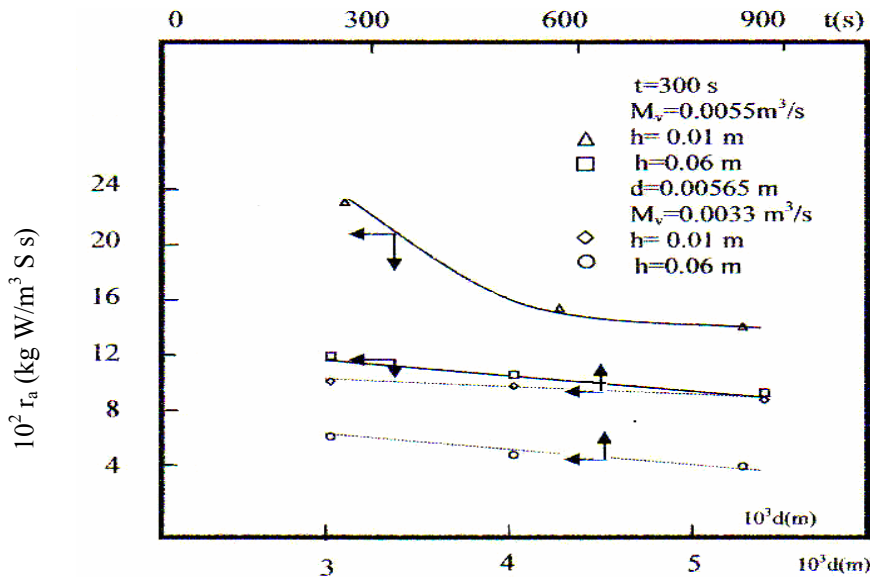


Fig. 10 – Dependence  $r_a=f(d)$  and  $r_a=f(t)$ ,  $C_0=15.72 \cdot 10^{-3}$  kgW/m<sup>3</sup>A.

The Figures 7 and 8 emphasized an increasing of the adsorption rate with air flow rate and air moisture content. The increasing of air velocity determines a decreasing of the resistance at the mass transfer and therefore an intensification of the adsorption rate.

In Figures 9, 10 the manner in which the time influences the adsorption rate is revealed. It can be established that with the increasing of time decreases the adsorption rate. This fact can be explained by the decreasing of driving force of the mass transfer as a result of increasing of the water content in silica gel.

The sizes of silica gel particles influence the adsorption rate, Figures 9, 10 in the direction of its (adsorption rate) increasing with the decreasing of the mean diameter of particles. The decreasing of particle sizes goes to the decreasing of the fixed bed porosity and increasing of the real air velocity in bed, and on the

other hand the smaller particles have short pores and this provides a greater access to the internal surface of the water vapours. These two aspects compete with each other to the decreasing of resistance at the mass transfer and to intensify the adsorption rate.

The data regarding the adsorption rate were stood at the base of determining of mass transfer coefficient. In this way it was used the relation (2).

The results obtained are shown as charts in figures 11–14.

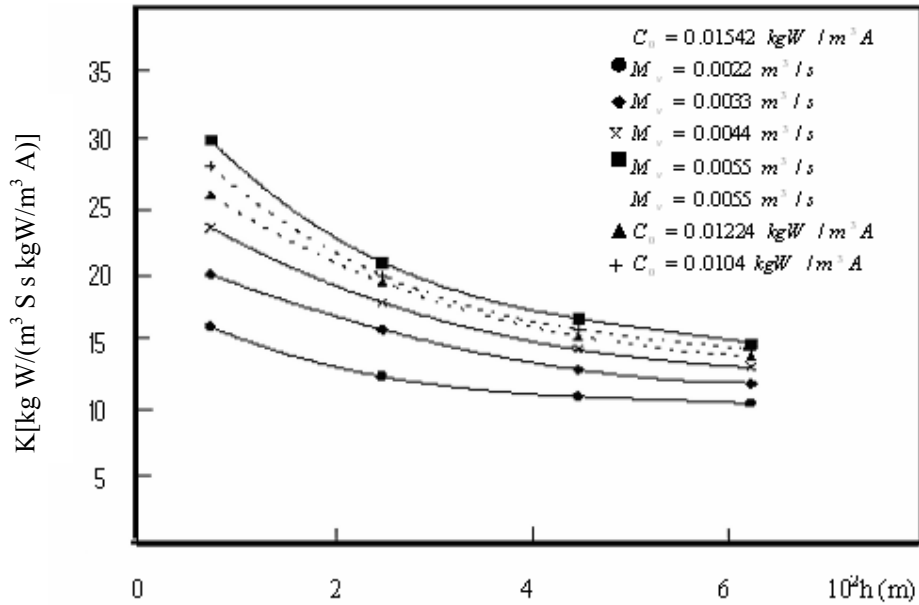


Fig. 11 – Mass transfer coefficient versus h,  $d = 3.57 \cdot 10^{-3}$  m,  $t = 300$  s.

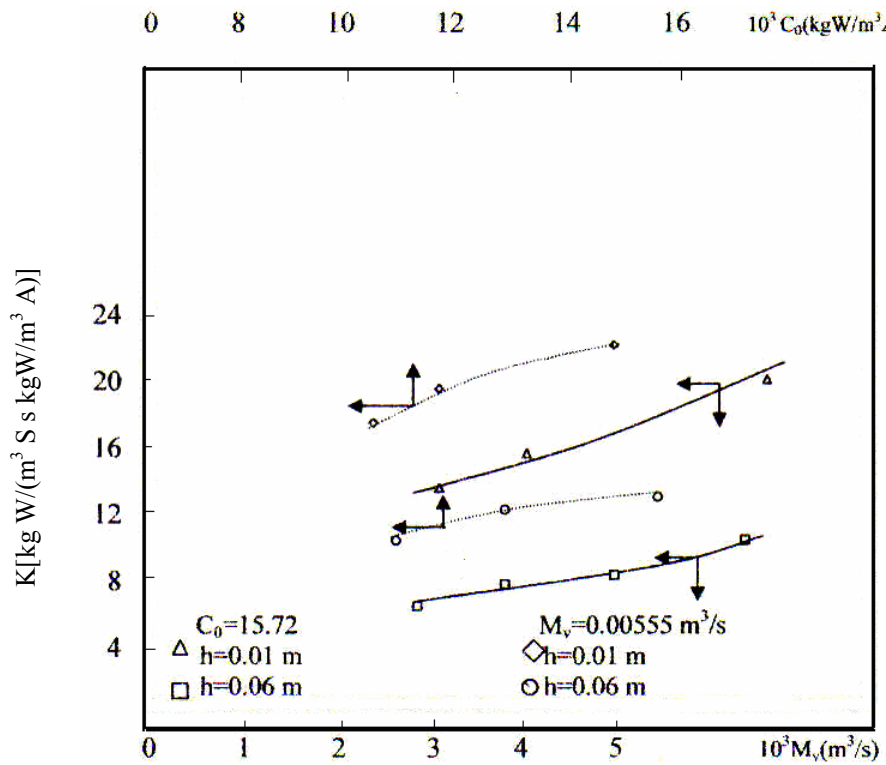


Fig. 12 – Mass transfer coefficient versus  $M_v$  and  $C_0$ ,  $d = 3.57 \cdot 10^{-3}$  m,  $t = 300$  s.



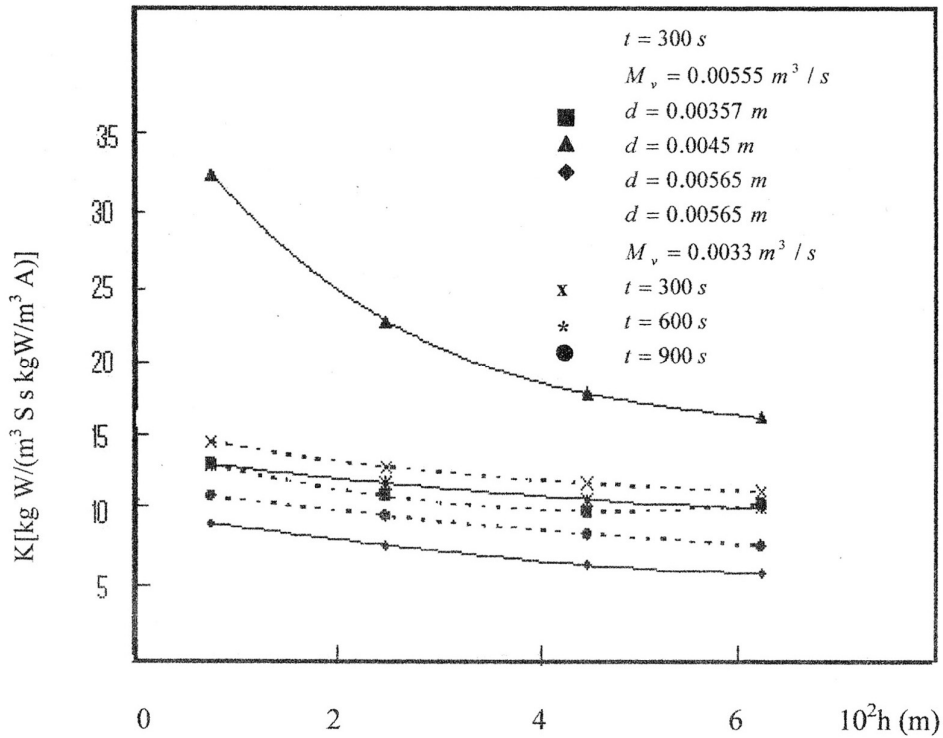


Fig. 13 – Mass transfer coefficient versus  $h$ ,  $C_0=15.72 \cdot 10^{-3}$  kgW/m<sup>3</sup>A.

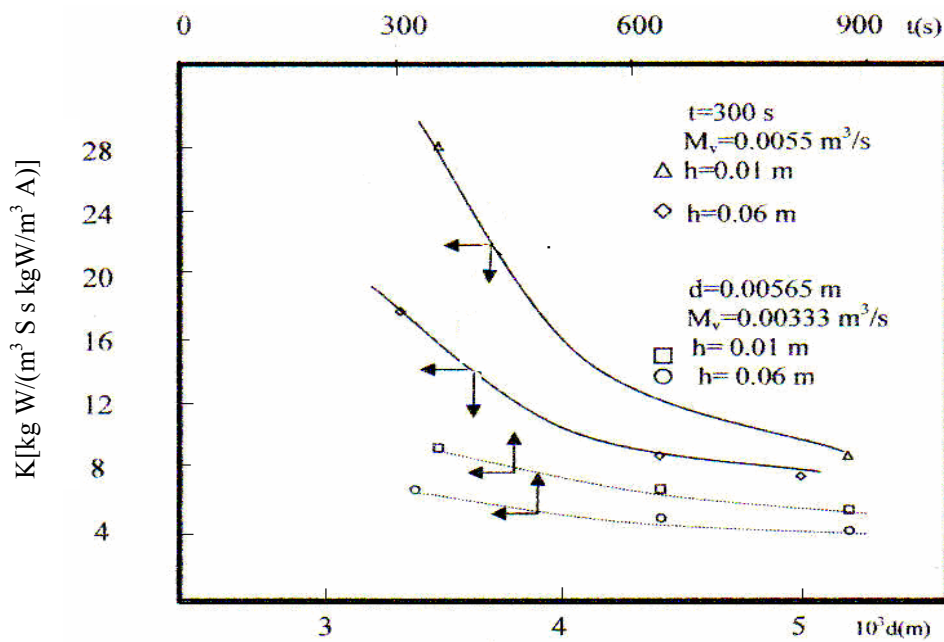


Fig. 14 – Mass transfer coefficient versus  $d$  and  $t$ ,  $C_0=15.72 \cdot 10^{-3}$  kgW/m<sup>3</sup>A.

The diagrams of variation of mass transfer coefficient on the height of the silica gel fixed bed shows a positive influence of the air flow rate and a decreasing of the value of this coefficient with the increasing of the time and particles size.

In order to verify the results of the investigation performed mean values of water concentration in solid phase on the fixed bed height were calculated using the relationship:

$$\bar{X} = \frac{1}{H} \cdot \int_0^H X(h) dh \quad (6)$$

The values obtained with equation (6) were compared to literature values<sup>17</sup>. As can be seen in Table 1 there is a good agreement between the literature values and those obtained by us.

The physical proprieties of gas and solid phases were taken from literature<sup>18, 19</sup>.

Table 1

Experimental data validation

$10^3 M_V (m^3/s)$	3.33						4.44		5.55	
t(s)	300		600		900		300		300	
	ec.(6)	ref. <sup>17</sup>	ec.(6)	ref. <sup>17</sup>	ec.(6)	ref. <sup>17</sup>	ec.(6)	ref. <sup>17</sup>	ec.(6)	ref. <sup>17</sup>
$\bar{X}$ (kg/kg)	0.0346	0.0332	0.0561	0.0570	0.0737	0.0751	0.0447	0.0421	0.0580	0.0534

## CONCLUSIONS

In this paper the gas-solid mass transfer at adsorption was experimentally studied.

Silica gel particles were used as adsorbent, and wet air was used as gaseous phase.

The profile of water vapour concentration from silica gel on the height of the bed was determined at different values of time, mean diameters of particles, air flow rate and air moisture content.

Also the variation of adsorption rate and mass transfer coefficient on the height of silica gel bed were determined.

The results obtained show the manner in which the flow rate and moisture content of air, time and size influence the profile of water concentration in silica gel, adsorption rate and mass transfer coefficient.

## Nomenclature

$m_0$  – mass of silica gel particles without indicator before adsorption, kg;

$M_V$  – gaseous phase volumetric flow rate,  $m^3/s$ ;

$C_i$  – water concentration in air at the exit of “i” zone of the bed, kg water/ $m^3$  air;

$C_{i-1}$  – water concentration in air at the entrance of “i” zone of the bed, kg water/ $m^3$  air;

$C_i^*$  – adsorbate concentration in the gaseous phase at equilibrium at the exit of “i” zone of the bed,  $kg/m^3$ ,

$C_i^* = f(X_i)$ ;

$C_{i-1}^*$  – adsorbate concentration in the gaseous phase at equilibrium at the entrance of “i” zone of the bed,  $kg/m^3$ ;

$x_0$  – mass fraction of water in silica gel before adsorption;

$X$  – water concentration in silica gel, kg water/kg silica gel;

$\bar{X}_{i|t} \left( = \frac{X_{i-1|t} + X_{i|t}}{2} \right)$  – mean adsorbate concentration in the “i” zone of the bed at  $t = t$ , kg/kg;

$\bar{X}_{i|t+\Delta t} \left( = \frac{X_{i-1|t+\Delta t} + X_{i|t+\Delta t}}{2} \right)$  – mean adsorbate concentration in the “i” zone of the bed at  $t = t + \Delta t$ ,

kg/kg;

$h_i$  – height of “i” zone in the bed;

$H$  – height of the bed, m;

$K$  – mass transfer coefficient,  $\frac{\text{kg water}}{\text{m}^3 \text{ silica gel} \cdot \text{s} \frac{\text{kg water}}{\text{m}^3 \text{ air}}}$ ;

$r_a$  – adsorption rate,  $\text{kg water}/\text{m}^3 \text{ silica gel} \cdot \text{s}$ ;

$S$  – cross section area of the fixed adsorbent bed column,  $\text{m}^2$ ;

$t$  – time,  $\text{s}$ ;

$V_{st}$  – volume of silica gel bed,  $\text{m}^3$ ;

$\Delta m$  – mass variation of silica gel particles without indicator,  $\text{kg}$ ;

$\Delta t$  – time range,  $\text{s}$ ;

$\rho_v$  – the apparent density of adsorbent,  $\text{Kg}/\text{m}^3$ ;

$A$  – air;

$W$  – water;

$S$  – silica gel, solid.

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