Adsorbents for the natural gas drying at CNG stations

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Humidity present in the natural gas causes problems by its compression at CNG filling stations. Therefore, the natural gas has to be dried before compression. For this reason, the water adsorption from the gas is mostly used as a suitable adsorbent. A device for drying compressed natural gas at high pressure is, therefore, an important part of the CNG filling station and affects the quality of produced CNG. For the correct operation of the drying equipment is decisive the selection of the suitable material for the adsorption of water vapor from the gas. As adsorbents for the removal of water from the gas, molecular sieves 4A, 5A, and 13X are primarily used. Molecular sieve 4A has a high sorption capacity for water vapor, which is, however, significantly reduced by co-adsorption of methane at high pressures. Other molecular sieves have not so high sorption capacity for water, but the influence of co-adsorption of methane at high pressures is by these adsorbents not so significant. The article is focused on selection of commercially available adsorbents having a high capacity for water steam and high efficiency of its removal from the gas. The results of laboratory tests of methane drying have been used in the selection of best adsorbent for natural gas drying at CNG stations.

Key words: *adsorbent; natural gas; CNG station; drying; methane*

1. Introduction

The natural gas drying technologies are applied in operational practice, especially after wet gas extraction from the underground storage or deposits before gas transportation in long-distance or gas distribution pipelines. For this purpose, so-called absorption drying technologies, applying as a drying agent glycols, mostly triethylene-glycol (TEG), are used in the operational practice. Furthermore, it is necessary to dry the natural gas also before its compression at high pressure to produce compressed natural gas (CNG). In these cases, there are typically a relatively small gas flow rates, which are dried at very low residual moisture content. The water condensation in the compressed gas after the gas compression to high pressure (up to 30 MPa) can so not occur. The technologies operating on the principle of the water adsorption on an appropriate adsorbent are used to this deep gas drying in operational practice. This article is focused on the testing of suitable adsorbents for deep natural gas drying in laboratory conditions and selection of the best adsorbent for operational practice.

2. Water content in natural gas

The higher water content in the natural gas causes varies problem for a number of reasons; the most significant ones are corrosion and formation of hydrates. The main danger of higher water content in the gas is the possibility of water condensation due to the decreasing of gas temperature (Løkken, 2013). For example, when the wet gas is passing through the depressurizing station, due to a drop in temperature, the water may be separated from the gas in the liquid or solid phase and causes trouble in the regulator (freezing, erosion, etc.). For high-pressure gas pipelines, the possibility of hydrates formation as the consequence of water condensation is the main problem with high pressure and low temperature (Kim et. al., 2010; Caroll, 2014).

According to the order of the operator of the gas transportation system in the Czech Republic, the "dew point" is defined as the temperature in degrees Celsius at which water vapor contained in the gas at operating pressure begins to condense. The value of the dew point of water related to the pressure of 4 MPa shall not be higher than -7 °C. That is when the maximum water vapor concentration in the gas 104 mg/m^3 is calculated under normal conditions (15 °C, 101.325 kPa) (Netusil and Ditl, 2011; TPG 902 02, 2006). Since 2012, there is a maximum dew point value of water in the natural gas -8 °C at the pressure of 7 MPa. The partial water vapor pressure at this dew point is 334 Pa. That is when converting to normal conditions (15 ° C, 101.325 kPa) is the maximum water vapor concentration in the gas 68 mg/m^3 (DVGW G 260: 2013-03, 2013; CBP 2005-001/02, 2008).

The water content in gas distribution systems in the Czech Republic should have a moisture content expressed as the dew point 2 °C below the temperature of the soil by the operating pressure (TPG 902 02, 2006).

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3. Technologies used to the natural gas drying

For the natural gas drying, three different technologies are used in operational practice. The first one is the absorption of water vapor from the gas by using triethylene-glycol (TEG). The second technology is based on the adsorption of moisture at a suitable adsorbent; the last one works on the principle of water condensation from the gas cooled due to the Joule-Thomson's effect of its expansion. Methods differ in principle and suitability for using by individual specific cases (Mokhatab et. al, 2006).

For the small and medium flows of natural gas promote in the operational practice the adsorption drying technology for its simplicity, high efficiency and minimum demands on the service. Polar inorganic materials are used as the adsorbent, mostly on the basis of zeolite molecular sieves, which reach a high adsorption capacity for water even at low concentrations of water vapor in the gas and allow a high degree of the gas drying (Farag et. al., 2011).

Both systems are enforced into operational practices without regeneration of water saturated adsorbent, so systems with integrated regeneration of water saturated adsorbent directly in the adsorption equipment. Gas drying systems with regeneration of the adsorbent typically use more adsorbers connected parallel, where one of the adsorber is being used to dry the gas and the other is regenerated by heating to a high temperature and subsequently cooled after desorption of water captured in the adsorbent. Systems without regeneration of water saturated adsorbent are usually used only for drying of small flows of gas, where the frequent exchange of water saturated adsorbent is not necessary.

4. Adsorbents used for natural gas drying and their significant properties

Polar adsorbent on the basis of inorganic compounds are used to the water adsorption of natural gas. The most important characteristics of adsorption materials are adsorbent polarity, its specific surface size, pore size distribution and the adsorption pore volume.

The adsorbent polarity is characterized by its ability to preferential adsorption of polar or non-polar substances. Polar substances are adsorbed better on the polar adsorbents and non-polar substances on the non-polar ones. The adsorption of polar substances on the non-polar and non-polar substances on polar adsorbents typically occurs after exceeding their relative pressure of 0.5 in the environs of adsorbent. As relative pressure, there is meant the actual partial pressure of the substance in relation to the pressure of their saturated vapor at the given temperature (Kohl and Nielsen, 1997).

The specific surface of adsorbents shall mean the total surface of the pores, which are located inside the adsorption material. A porous system for some highly developed adsorbents is so that the total specific surface can reach more than 1 500 m^2/g adsorbent (Kast, 1981).

The adsorption of water and other substances from natural gas occurs in the smallest pores, so-called submicro- and the micro-pores. Only in the case of the so-called capillary condensation, also mezo-pores are filled. Capillary condensation of water occurs only at higher concentrations of water vapor in a gas in more than 30 % of rel. humidity (relative vapor pressure higher than 0.3). Macro-pores have a function only as the transport pores to the internal porous system.

In addition to the internal surface, the pore adsorption volume of the material is another important property of adsorption, i.e., the volume which is filled with adsorbed substances in the case of full saturation of adsorbent until the steady state.

The most widely used industrially produced types of adsorption materials used for gas drying include (Kast, 1988):

- γ-alumina,
- silica gel,
- natural and synthetic zeolites.

Alumina is made of more than up to 85 % of Al_2O_3; the rest represent various mineral ingredients. For its strongly hydrophilic character, it is suitable mainly for adsorption of polar compounds from gaseous mixtures and as a carrier of catalysts. The most stable form of aluminum oxide is α-Al_2O_3, called corundum. Corundum is mechanically strong, but has a low porosity and a specific surface. Active alumina is γ-Al_2O_3, which is produced by calcination of aluminum hydroxide monohydrate or trihydrate. The pore volume and surface area depends on the crystalline modification, on the amount of water and the content of alkali and alkaline earth metals. The porosity of γ-Al_2O_3 is between 50 and 70 %, and its specific surface reaches values of 120 to 150 m^2/g.

Silica gel is adsorbent based on silica, which can be in different degrees of hydration. By weight, silica gel is 95 % made up of SiO_2 and the remaining 5 % are other oxides. Because of having a hydrophilic character, it is also used for gas drying.

The size of the inner surface of the silica gel reaches up to 700 m^2/g; the pores in the structure are represented by micro- pores in the dimension of 1-1.5 nm and mezo- pores in diameter up to 5 nm.

By repeated desorption of water, when silica gel is long-term exposed to temperatures above the 200 °C, irreversible reduction of its adsorption capacity occurs. This phenomenon constitutes a major disadvantage for the industrial use of silica gel. Several tests of silica gel in industrial facilities have shown that in the course of long-term using, the reduction of its adsorption capacity occurs, which is doubled at the beginning and in some cases tripled in comparison with molecular sieves. The adsorption capacity of the silica is also significantly reduced by the presence of hydrogen sulfide in the dried gas.

Zeolites consist of natural or synthetic alumosilicates. Other cations are found in its porous system. Zeolites usually have strongly hydrophilic character caused by electrostatic forces of theirs crystallographic structures. Given the exact defined structure of input holes in the porous system, zeolites are used as the molecular sieves for separation, cleaning and drying of gaseous mixtures and as the catalysts.

Exactly defined, the size of the channel structure differentiates zeolites from amorphous micro porous materials, such as activated carbon or γ-alumina, as they always have a wide pore size distribution. According to the ratio of the Si/Al zeolites can be divided into two basic groups:
- Zeolite with a low ratio of Si/Al (< 10).
- Zeolite wit high ratio of Si/Al (> 10).

Zeolites with a low ratio of Si/Al show far higher hydrophilicity than zeolites with high ratio Si/Al. It is obvious that with the increasing share of AlO_4 tetraeder in the zeolite structure also his hydrophilicity increases.

The maximum permitted content of AlO_4 tetraeder in the zeolite is equal to 50 % mol. Zeolite with this content of AlO_4 has a molar ratio Si/Al = 1 and is known as the zeolite X. This zeolite type possesses a totally regular alternation of SiO_4 and AlO_4 tetraeders. Such zeolites have the greatest polarity and therefore are best suited for the adsorption of water vapor (Yang, 2003; Rybár et. al., 2015).

4.1 Expression of the adsorption capacities of adsorbents using basic adsorption equations

Adsorption capacities of adsorbents for different adsorbed substances are most commonly expressed using the so-called adsorption isotherm. The isotherm represents the dependency of the quantity of the adsorbed substance on its concentration (part. pressure) in the adsorbent environs at a given temperature. Many adsorption theories are used to describe this process. From the basic equation of adsorption, the Dubinin theory is the most used, which is as the only one of the fundamental theories that allow affecting the influence of temperature on the adsorption capacity (temperature is given in this equation as a variable parameter).

This adsorption theory was originally developed by Dubinin and Raduschkevich (Dubinin, 1967; Dubinin 1989) for adsorption on charcoal, it was later modified on other adsorption materials. The basis of Dubinin adsorption theory constitutes a potential theory developed by Polanyi. Polanyi theory assumes adsorption in layers that cause the creation of potential field on the adsorbent surface. Dubinin founded his theory on the assumption that not the surface size is the decisive parameter for adsorption adsorbent, but the size of the adsorbent pore volume. During the adsorption occurs towards filling of the adsorbent pore volume. Dubinin derived the known form of adsorption equation that has the following form for subcritical adsorpt temperatures:

$$X_m = \frac{w_0}{v} \exp\left[-\frac{RT}{\beta E_0} \ln \frac{p_s}{p} \right]^n ,$$

(1)

and then after the merger of constants $(2,303 \cdot R/E_0)^n$ to constant (B):

$$X_m = \frac{w_0}{v} \exp\left[-B\left(\frac{T}{\beta} \log \frac{p_s}{p} \right)^n \right] ,$$

(2)

where X_m is the amount of substance adsorbed in the adsorbent pores (mmol/g), w_0 is the volume of adsorption pore (cm^3/g), v is the molar volume of adsorpt at the adsorption temperature (cm^3/mmol), B is the adsorbent specific constant, T is the adsorption temperature (K), p is the pressure of adsorptive (N/m^2), p_s is the pressure of the saturated adsorptive vapor at the temperature T (N/m^2).

In order to be able to compare the behavior of various adsorptives, we select one of them as the standard and the affinity coefficient $\beta = E/E_0$ (E_0 is the energy of a standard adsorptive) is used for comparison. The adsorptive is adsorbed more strongly for the values of $\beta > 1$, and fainter than the standard adsorptive for the values of $\beta < 1$.

Dubinin exponent n varies in the range of 1 to 3, while for activated carbon the best results are achieved by applying the coefficient of equal or close to 2 and for zeolithic adsorbent by applying the coefficient equal to or close to the 3.

From the equation, it is clear that the volume of the adsorption pores is crucial to the adsorption capacity of each adsorbent. Complete filling occurs only when such adsorptive pressure around the adsorbent is achieved,

which is equal to the pressure of his saturated vapor at the given temperature. The form of adsorption isotherm is significantly influenced by the size of the exponent *n*. As already mentioned, this exponent reached values of 3 on zeolites, resulting in a graphic representation of the adsorption isotherm to the rapid increase in the area of low pressure, and subsequently in the field higher pressures only to very slow growth at the maximum value. In practical applications by water adsorption in zeolites, it is therefore already achieved high ads. capacity at low gas humidity, which is no else too increase by the following increasing of gas humidity.

If the simultaneous adsorption of more substances occurs, the adsorbed substances share together the adsorption space (adsorption pore volume), which leads to a reduction of adsorption capacity, compared to the state where they are adsorbed in the adsorbent both separately. In the course of adsorbent loading by multicomponent adsorption (e.g., two-component) in flow adsorber, there is the state where the zone is created into the adsorber in the space closest to the entry flow of media, in which both substances in a specific ratio are adsorbed. In this zone, a gradual decrease in the concentration of the better-adsorbed component occurs almost to zero, and then flows just worse adsorbed component, which is captured in the next (second) adsorption zone. When the concentration of this component declines to the zero value, the zone of non-saturated adsorbent begins. All zones are moved during the adsorption in the direction of fluid flow to the end of the adsorption bed. The movement of the first zone in which both components are adsorbed causes a gradual crowding of the part of worse adsorbed second component of the second zone (in which only worse adsorbed component is captured), which occurs in the second zone, to a gradual concentration growth of this component in the moving "concentration wave". After a certain time, the second zone will arrive at the end of the adsorption bed, and worse adsorbed component starts to penetrate into the cleaned gas. Because of the concentration growth of this component, its concentration in the gas at a certain time reach much higher values than at the entrance to the adsorber. After a certain time, also the first zone arrives at the end of the adsorption bed and also better-adsorbed component starts to penetrate into the cleaned gas. After a certain time, the concentration of both components at the output of the adsorption bed shall be relevant to their input concentrations.

This behavior of adsorbents in multicomponent systems can be very dangerous because it leads to the concentration growth and the consequent worse adsorbed components crowd in much higher concentrations than are their concentration on the adsorber entry. This phenomenon is in operational practice, manifested both in the adsorption from gaseous phase and adsorption from liquid phase (Dubinin and Kadlec, 1987; Dubinin and Plavnik, 1968; Dubinin, 1979).

4.2 Adsorption capacity of commercially available adsorbents for water

Several research articles bring the information concerning the adsorption capacity of different commercially available adsorbents for water estimated in various ways by atmospheric or elevated pressure. From the results, it is possible to estimate the impact of possible co-adsorption of other components of the gas under different conditions. Some of the published data also indicate the length of the adsorption zone (part of the adsorption bed, where adsorption of water occurs at some time), which provides information about the use of adsorbent and its possible saturation with water until steady state at the moment of penetration of water vapor into the dried gas (growing of water vapor concentrations in the dried gas). Some of the published data, however, indicate only the maximum adsorption capacity obtained in saturation of the entire volume of the adsorbent until the steady state and say nothing about the kinetics of adsorption.

The results of tests of various commercially available adsorbents for gas drying transferred from (Staf, 2001) are listed in Table 1. The testing was performed using N_2 adsorption and desorption by 77 K.

Tab. 1. Properties of the adsorbents tested in the air drying.

Specification of the adsorbent	BET-surface [m²/g]	the ads. pore volume [ml/g]
molecular sieve 5A (Tamis)	449	0.264
molecular sieve 13X (Tamis)	328	0.267
Clinoptilolith (Nižný Hrabovec)	24	0.074
KC-Envisorb (B) + (Engelhard)	725	0.804
KC-Trockenperlen (Engelhard)	596	0.401
Baylith (Bayer Leverkusen)	-*	-*

* not estimated

Using measurements in the laboratory conditions (Staf, 2001), the adsorption capacities of selected adsorbents for water vapor were estimated. The testing was performed using the method of air flow as the carrier

gas at different temperatures. The sorption capacities were estimated at selected samples of adsorbents after their water vapor saturation up to a steady state by different gas humidity.

The results of these measurements are listed in Table 2 and represents equilibrium adsorption capacities for water at the temperature of 20 ° C, using air as the carrier gas at a normal pressure at various gas humidity.

Tab. 2. The adsorption isotherms of tested adsorbents for water at 20 °C.

rel. gas humidity:	10 % rel.	30 % rel.	50 % rel.	70 % rel.	90 % rel.
adsorbent	adsorption capacity for water vapor at 20 °C [% mass.]				
molecular sieve 5A	13.6	15.7	17.0	18.0	19.5
molecular sieve 13X	5.7	10.2	12.9	14.9	20.3
Clinoptilolith	6.3	7.4	8.1	9.0	11.2
KC-Envisorb (B) +	3.9	9.0	17.3	36	67.1
KC-Trockenperlen	5.2	13.3	24.1	36.3	40.9
Baylith (4A)	18.1	19.0	19.8	20.3	21.5

Molecular sieve adsorbents 5 A, 13X and Baylith belongs to the group of synthetic zeolites and Clinoptilolith to the group of natural zeolites. Clinoptilolith sample used for the test was mined in the quarry Nižný Hrabovec in Eastern Slovakia. Adsorbent KC-Envisorb B + is combined adsorbent containing activated carbon and silica gel as the adsorbent. KC-Trockenperlen is adsorbent based on silica gel. The adsorbent based on silica gel may exhibit higher ads. capacity for water vapor at higher relative gas humidity, than synthetic zeolites. However, the kinetics of water adsorption on the silica gel is very slow, due to the creation of hydrogen bridges, leading to unreasonably long, flat breakthrough curves of water vapor and quickly declining efficiency of gas drying during the loading of the adsorbent with water vapor (Ciahotny et. al., 2014). The comparison of breakthrough curves for water vapor measured using the adsorbents molecular sieves 5A and 13X and silica gel KC Trockenperlen WS is given in Fig. 1.
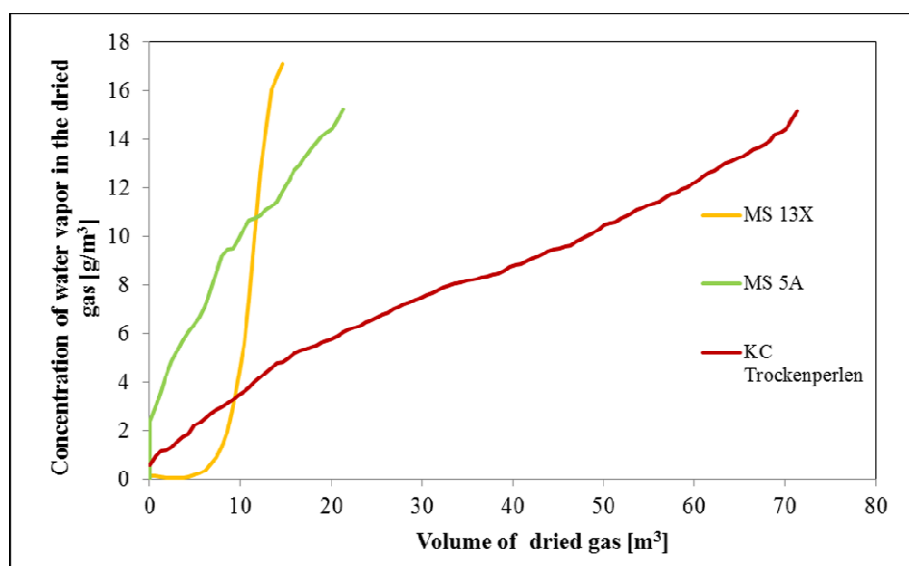

Fig. 1. Breakthrough curves for water vapor measured at selected adsorbents [20].

From the measured breakthrough curves shown in Fig. 2, it is apparent that the shortest adsorption zone for water vapor and the highest efficiency of the drying possess the molecular sieve 13X. Conversely, silica gel KC Trockenperlen shows slow kinetics of water vapor sorption and a very long adsorption zone, the result of which is the insufficient efficiency of the gas drying.

The adsorption capacities for water estimated in (Ciahotny et. al., 2014) using methane as the carrier gas (pressure 4 MPa, dew point +17 °C) amount to 15.6 % by mass by molecular sieve 5A, 19.5 % by mass by molecular sieve 13X and 41.1 % by mass by KC Trockenperlen.

Similar adsorptions capacities for water at Baylith and Clinoptilolith were estimated in (Preis, 1998). The measurements were carried out by 25 °C and using methane as the carrier gas. By the gas pressure of

0.2 MPa and part. pressure of water steam 2 400 Pa reached the ads. capacity by Clinoptilolith 10 % by mass and 22 % by mass by Baylith. By the elevated gas pressure of 4 MPa and the same part pressure of water steam dropped the adsorption capacity for water by Clinoptilolith to 7 % by mass and by Baylith to 14 % by mass.

Adsorption capacities of selected adsorbents for water listed in Table 2 were used to calculate the adsorption isotherm for water at a temperature of 20 °C. The equation (2) in its linearized form was applied to the calculation:

$$log X_m = -0.434B \left(\frac{T}{\beta}\right)^n \left(log \frac{p_s}{p}\right)^n + log \frac{w_0}{v} \qquad (3)$$

The value of "*n*" was chosen as 1 because the measured adsorption capacities compared in dependency to the water steam pressure using a linearized form of the Dubinin equation showed the best linearity using this value. From the shape of the lines obtained for each adsorbent values w_0 were calculated, which are, together with the parameters of the individual lines, listed for all adsorbents in Table 3.

Tab. 3. Parameters of linearized forms of adsorption isotherms for each adsorbent.

Specification of the adsorbent	straight slope [-]	calculated ads. pore volume [ml/g]
molecular sieve 5A (Tamis)	-0.177	0.192
molecular sieve 13X (Tamis)	-0.4893	0.178
Clinoptilolith (Nižný Hrabovec)	-0.1847	0.096
KC-Envisorb (B) + (Engelhard)	-1.1493	0.556
KC-Trockenperlen WS (Engelhard)	-0.9393	0.449
Baylith (Bayer Leverkusen)	-0.1265	0.217

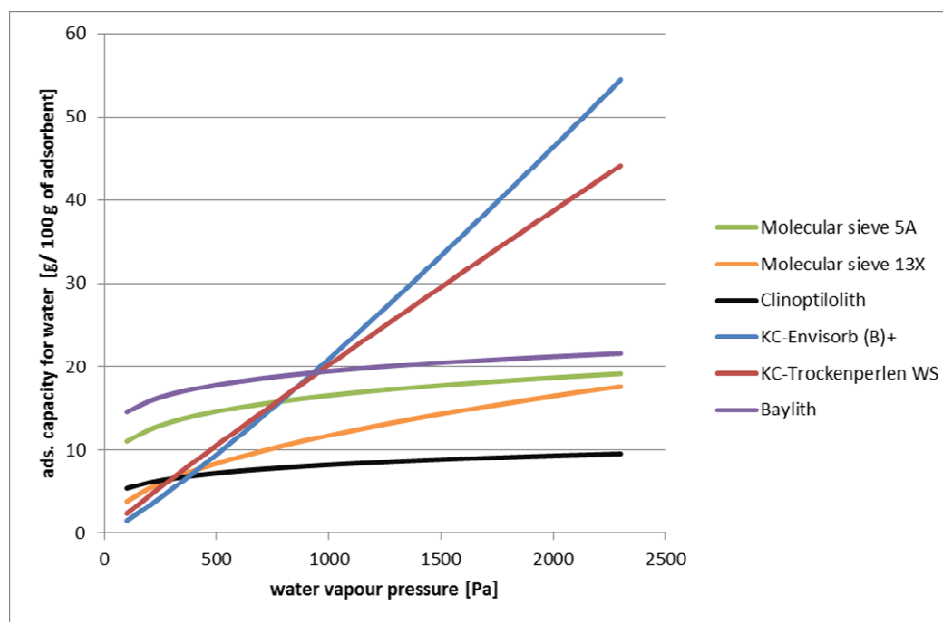Calculated forms of adsorption isotherm are illustrated in Fig. 2.

Fig. 2. Calculated forms of ads. isotherms for water.

The adsorbents Baylith and molecular sieve 5 A seems to be the adsorbents having the best adsorptions capacity for water by the natural gas drying containing appropriated humidity corresponding to the water steam pressure of 334 Pa in the gas (max. dewpoint of -7 °C by 7 MPa).

5. Conclusion

Adsorption drying technologies are always used for the drying of natural gas at CNG station. This technology reaches the desired degree of gas drying without any problems if the correct adsorption material is used for water vapor from the dried gas. It is appropriate to use first of all molecular sieves 4A, 5A or 13X as

the adsorbents. Adsorbents with smaller pore sizes (4A, 5A) in comparison with the molecular sieve 13X have slower kinetics sorption of water molecules, but higher total adsorption capacity for water vapor. By the adsorbent Baylit (4A), the adsorption capacity for water vapor is in the ranges from 18 to 22 % (by mass) according to the relevant humidity of gas. Increasing gas pressure of 0.2 MPa to 4 MPa, leads, however, to a significant decrease of the adsorption capacity for water vapor to 14 % due to the co-adsorption of methane.

The adsorption capacity of molecular sieves 5A for water vapor varies in the range from 13 to 20 % (by mass) according to the moisture in dried gas. Decrease in the adsorption capacity of water under the influence of methane co-adsorption for elevated pressure is by molecular sieve 5A not as high as by molecular sieves 4A.

Adsorption capacity for water vapor for molecular sieves 13X ranges from 6 to 20 % (by mass) and is more dependent on the moisture of dried gas, than in the case of adsorbents 4A and 5A. The adsorbent 13X have referred to another molecular sieve the highest rate of sorption velocity of water molecules. Their application is very suitable in particular for the gas drying containing high amount of water vapor.

From the point of view of the drying equipment concept, systems without regeneration of saturated adsorbent and also systems with integrated adsorbent regeneration directly in the dryer are used. Systems without integrated adsorbent regeneration are easier, cheaper and less demanding for the service and maintenance. They are used where there are considered small flow rates of gas by low gas moisture. Conversely, systems with integrated regeneration of adsorbent have to be used everywhere where large amounts of dried gas containing a higher amount of water vapor are considered. These systems are often used for natural gas drying after its extracting from the deposit or gas extracted from the underground gas storage. Compared to systems working on the principle of absorption of water vapor are advantageous, because simultaneously with water vapor are from the gas removed also higher hydrocarbons, which cause often problems by long distance transport of natural gas in transit pipelines. Drying systems based on adsorption are used eg. for natural gas drying extracted from the deposit Sosnogorsk (Russia) or gas drying extracted from the Uhřice underground gas storage in the Czech Republic.

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