



Section 4. Materials science

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DETERMINATION OF THE DYNAMIC ADSORPTION CAPACITY OF ADSORBENTS FOR WATER VAPOR

**Ashurov Sherali Mustafoyevich ¹, Bozorov Otabek Nashvandovich ²,
Kuyboqarov Oybek Ergashovich ³**

¹ Tashkent State Technical University,

² University of Economics and Pedagogy,

³ Karshi State Technical University

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Abstract

The article presents a methodological and technological assessment of determining the dynamic adsorption capacity of composite adsorbents for water vapor under conditions relevant to natural gas drying. The study considers KA-series adsorbents prepared on the basis of activated Navbahor bentonite, zeolite 4A, activated alumina, silica gel and a binder. Dynamic capacity is treated not only as a mass balance value but also as an integral technological indicator connected with breakthrough time, saturation time, outlet moisture concentration, dew point depression, pressure drop, bed utilization and regeneration stability. The operating variables considered include gas flow rate, adsorbent mass, bed height, granule diameter, inlet moisture content, temperature, pressure and regeneration regime. Particular attention is paid to the KA-4 composite adsorbent, which combines a local mineral matrix, a selective microporous zeolite phase, a thermally stable alumina phase and a mesoporous silica-gel component. Representative dynamic drying data show that KA-4 provides a breakthrough time of 8.6 h, saturation time of 12.8 h, dynamic water adsorption capacity of 0.146 kg/kg, outlet dew point of about –43 °C and drying efficiency of 96.9%. The results demonstrate that the reliable determination of dynamic capacity requires simultaneous analysis of the breakthrough curve, mass-transfer zone and regeneration recovery, because static water uptake alone does not fully describe the industrial performance of an adsorbent bed. The proposed approach can be used for selecting optimal adsorbent composition and operating conditions for deep dehydration of natural gas.

Keywords: natural gas drying; water vapor adsorption; dynamic adsorption capacity; composite adsorbent; KA-4; breakthrough curve; dew point; zeolite 4A; activated bentonite; regeneration

Introduction

Natural gas normally contains water vapor, and its presence is undesirable during transportation, compression, cooling and further processing. Moisture can cause hydrate formation, corrosion of pipelines and process equipment, freezing in valves and heat exchangers, loss of throughput and instability in downstream separation units. Therefore, dehydration is one of the key stages in natural gas treatment before transmission through pipelines, cryogenic processing, NGL recovery or LNG production. In industrial practice, the target quality of dehydrated gas is often expressed through residual moisture content and dew point. A lower dew point indicates deeper drying and a lower risk of condensation under operating conditions (Gandhidasan P., Al-Farayedhi A. A., Al-Mubarak A. A., 2001; Mokhatab S., Poe W. A., Mak J. Y., 2015; Kohl A. L., Nielsen R. B., 1997; Kidnay A. J., Parrish W. R., McCartney D. G., 2011).

Several dehydration technologies are used in gas processing, including glycol absorption, condensation by cooling, membrane dehydration and adsorption drying. Adsorption drying is of particular importance when very low outlet moisture content is required. The method is based on the selective retention of water molecules on the surface and inside the pores of a solid adsorbent. Silica gel, activated alumina and molecular sieves are widely used adsorbents. Silica gel has a high affinity to water at moderate humidity but is usually less effective for very deep dehydration. Activated alumina has good mechanical and thermal stability, whereas molecular sieves such as zeolite 3A and 4A show high selectivity toward water molecules and can provide a low dew point (Gandhidasan P., Al-Farayedhi A. A., Al-Mubarak A. A., 2001; Tagliabue M., Farrusseng D., Valencia S., Aguado S., Ravon U., Rizzo C., Corma A., Mirodatos C., 2009; Santos K. M. C., Vilarrasa-Garcia E., Cecilia J. A., Rodriguez-Castellon E., Azevedo D. C. S., 2021; Bahadori A., 2014; Grande C. A., 2012).

Despite their advantages, conventional adsorbents have limitations related to cost, regeneration energy demand, mechanical degradation, sensitivity to liquid carry-over and loss of capacity during repeated cycles.

For this reason, composite adsorbents based on local mineral raw materials have scientific and technological relevance. Combining activated bentonite, zeolite 4A, activated alumina and silica gel makes it possible to form an adsorbent with a balanced micro-mesoporous structure, acceptable mechanical strength and sufficient regeneration stability. In such a composite, bentonite acts as a mineral matrix, zeolite 4A provides selective microporous water adsorption, activated alumina improves thermo-mechanical stability, and silica gel supports fast moisture uptake in mesopores (Ruthven D. M., 1984; Yang R. T., 2003; Thommes M., Kaneko K., Neimark A. V., Olivier J. P., Rodriguez-Reinoso F., Rouquerol J., Sing K. S. W., 2015; Rouquerol F., Rouquerol J., Sing K. S. W., Llewellyn P., Maurin G., 2014; Breck D. W., 1974).

A central parameter for evaluating adsorbents in a flow system is dynamic adsorption capacity. Unlike static capacity, which is measured under equilibrium or quasi-equilibrium conditions, dynamic capacity characterizes the amount of water retained by a fixed adsorbent bed before breakthrough occurs. It is affected by mass transfer, pore accessibility, gas velocity, bed height, particle size, temperature, pressure and the regeneration history of the adsorbent. Therefore, determination of dynamic water adsorption capacity is essential for designing industrial adsorbents and for selecting a reliable adsorbent composition (Grande C. A., 2012; Ruthven D. M., 1984; Yang R. T., 2003; Thommes M., Kaneko K., Neimark A. V., Olivier J. P., Rodriguez-Reinoso F., Rouquerol J., Sing K. S. W., 2015).

The purpose of the study

The purpose of this study is to develop and describe a scientifically justified approach for determining the dynamic adsorption capacity of adsorbents for water vapor in the natural gas drying process. The study focuses on the KA-series composite adsorbents and especially on the KA-4 sample, which was selected as an optimal composition according to drying efficiency, dynamic capacity and regeneration stability. The specific objectives are: to define the main indicators used in dynamic adsorption tests; to describe the calculation procedure for dynamic water adsorption capacity; to analyze the influence of adsorbent composition and

operating parameters on breakthrough behavior; to evaluate KA-4 in comparison with other samples; and to propose criteria for using dynamic capacity as a practical design parameter for adsorption dryers.

The novelty of the approach lies in considering dynamic capacity as an integrated technological characteristic rather than as a single numerical value. The proposed evaluation combines the breakthrough curve, outlet moisture concentration, dew point, saturation time, pressure drop and residual capacity after regeneration. Such an approach gives a more realistic assessment of the adsorbent bed under cyclic operating conditions and provides a basis for pilot-scale design.

Literary analysis

The theoretical basis of adsorption processes is widely discussed in classical works on gas-solid adsorption. Ruthven described the principles of adsorption equilibria, mass transfer and breakthrough behavior in fixed beds (Ruthven D. M., 1984). Yang emphasized that the performance of adsorbents in gas separation and drying depends not only on equilibrium capacity but also on pore structure, diffusion rate, selectivity and cyclic stability (Yang R. T., 2003). These concepts are important because industrial gas drying is performed under dynamic flow conditions, where equilibrium capacity alone may overestimate the useful bed capacity.

In natural gas treatment, solid desiccant dehydration has been studied as an effective method for achieving a low water dew point. Gandhidasan and co-authors investigated the dehydration of natural gas using solid desiccants and showed the significance of breakthrough time, adsorbent loading and regeneration conditions (Gandhidasan P., Al-Farayedhi A. A., Al-Mubarak A. A., 2001). Handbook sources on gas processing and gas purification also indicate that molecular sieves and activated alumina are widely used for deep dehydration, especially before cryogenic units where very low water content is necessary (Mokhatab S., Poe W. A., Mak J. Y., 2015; Kohl A. L., Nielsen R. B., 1997; Kidnay A. J., Parrish W. R., McCartney D. G., 2011).

Zeolite molecular sieves are important for water removal because of their uniform microporous structure and strong interaction

with polar water molecules. Breck described the structure and adsorption properties of zeolite molecular sieves, including their high selectivity and crystallographic pore control (Breck D. W., 1974). However, the practical application of zeolite adsorbents can be limited by high cost and higher regeneration temperatures. Activated alumina and silica gel are less expensive and mechanically stable, but their deep dehydration capacity may be lower than that of molecular sieves. Therefore, mixed or composite adsorbents are often considered for balancing capacity, cost and regeneration performance.

The characterization of porous materials is another important part of dynamic capacity determination. BET surface area, pore volume and pore size distribution help explain why one adsorbent has a longer breakthrough time or higher capacity than another. Thommes et al. summarized the evaluation of surface area and pore structure by gas physisorption, noting the importance of micropores and mesopores for adsorption performance (Thommes M., Kaneko K., Neimark A. V., Olivier J. P., Rodriguez-Reinoso F., Rouquerol J., Sing K. S. W., 2015). Rouquerol et al. also emphasized the need for careful degassing and interpretation of adsorption-desorption isotherms in porous solids (Rouquerol F., Rouquerol J., Sing K. S. W., Llewellyn P., Maurin G., 2014). These methods are directly relevant to composite adsorbents, because micro- and mesoporous channels control both water uptake and diffusion inside granules.

The literature analysis shows that determination of dynamic water capacity must combine material characterization and fixed-bed testing. A material with a high static uptake may not always provide a long breakthrough time if its pores are poorly accessible, if granules are mechanically weak, or if the bed has excessive pressure drop. For this reason, the present work uses the dynamic adsorption capacity together with breakthrough time, dew point and regeneration recovery as main criteria for evaluating KA-series composite adsorbents.

Materials and methods

The investigated adsorbents were represented by KA-1 to KA-6 samples prepared from activated Navbahor bentonite and func-

tional additives. KA-1 consisted mainly of activated bentonite and served as a control sample. KA-2 included activated alumina to improve thermo-mechanical stability. KA-3 contained zeolite 4A and activated alumina for forming a selective microporous dehydration phase. KA-4 was a balanced composite containing bentonite, zeolite 4A, activated alumina and silica gel. KA-5 had a higher zeolite fraction to increase microporosity. KA-6 was selected for assessing regeneration resistance and mechanical stability. The granule fraction used for dynamic tests was 2–4 mm, which provides a compromise between diffusion path length and pressure drop.

Before the dynamic tests, the adsorbents were dried and regenerated to remove physically adsorbed moisture. The drying and regeneration temperature was selected according to the thermal stability of each composition. For KA-4, the recommended regeneration interval was 220–240 °C, while

the operating adsorption temperature was 25–35 °C. Dynamic tests were carried out by passing a humid gas stream through a fixed adsorbent bed. The inlet moisture concentration, outlet moisture concentration and dew point were recorded over time. The moment when outlet moisture increased to a defined fraction of inlet moisture was taken as breakthrough time.

The principal parameters used in the test are summarized in (Table 1). The most important indicator is dynamic adsorption capacity, because it shows how much water the adsorbent can retain under real flow conditions before breakthrough. Breakthrough time reflects the useful service period of the bed, whereas saturation time describes complete or near-complete filling of the bed with moisture. The outlet dew point is used as a practical quality indicator of dry gas, and pressure drop is used to assess hydraulic suitability of the bed.

Table 1. Main indicators used for determining dynamic adsorption capacity for water vapor

Indicator	Symbol	Unit	Determination method
Drying efficiency	η	%	$\eta = ((C_0 - C_t) / C_0) \cdot 100$
Dynamic adsorption capacity	qd	kg/kg or g/g	Water retained in bed per mass of adsorbent
Breakthrough time	tb	h	Time at $C_t/C_0 = 0.05-0.10$
Saturation time	ts	h	Time at $C_t/C_0 = 0.90-0.95$
Dew point	Td	°C	Measured at adsorber outlet
Pressure drop	ΔP	kPa	Difference between inlet and outlet pressure
Residual capacity after regeneration	Rq	%	Capacity retained after repeated cycles

For a constant volumetric gas flow rate, the amount of water retained by the adsorbent bed up to breakthrough can be calculated by integrating the difference between inlet and outlet moisture concentrations over time. In simplified form, dynamic capacity can be expressed as:

$qd = (Qg / mads) \cdot \int_0^{tb} (C_0 - C_t) dt$
where qd is dynamic adsorption capacity, Qg is the volumetric gas flow rate, mads is the mass of adsorbent in the bed, C₀ is the inlet moisture concentration, C_t is the outlet moisture concentration at time t, and t_b is breakthrough time. If the outlet concentration remains very low before breakthrough,

the expression can be approximated by the total removed water mass divided by the adsorbent mass. In industrial calculations, a safety coefficient is usually introduced to prevent premature breakthrough under fluctuating gas load and moisture content.

The determination procedure consisted of four steps. First, the adsorbent sample was regenerated and cooled to the adsorption temperature. Second, humid gas was introduced at a fixed flow rate, pressure and inlet moisture content. Third, outlet moisture and dew point were monitored continuously, and the breakthrough curve was obtained. Fourth, the water balance was calculated

from the area between inlet and outlet concentration curves. The same procedure was repeated after regeneration cycles to evaluate capacity recovery and long-term stability.

Discussion and results

The dynamic adsorption capacity of the KA-series adsorbents differs significantly depending on the composition and pore structure. The control sample KA-1, based on activated bentonite, showed limited capacity because bentonite alone does not provide sufficient microporous volume for deep water retention. Although activated bentonite has hydrophilic centers and can serve as a mineral matrix, its pore system is not enough to achieve a very low dew point in a dynamic gas stream. This confirms that activation improves the surface properties of bentonite but additional selective and mesoporous phases are required for effective natural gas drying.

The addition of activated alumina in KA-2 increased the mechanical and thermal stability of the granules. Activated alumina contributes to water adsorption due to hydroxylated surface sites and also improves resistance to attrition during gas flow. However, the absence of a strong molecular-sieve phase limited the deep dehydration performance. KA-2 therefore demonstrated better durability than KA-1 but did not reach the dynamic capacity of zeolite-containing com-

posites. This result illustrates that mechanical strength and adsorption capacity should be optimized simultaneously.

The introduction of zeolite 4A in KA-3 led to an increase in microporous water adsorption sites. As a result, breakthrough time and dynamic capacity increased compared with KA-1 and KA-2. Zeolite 4A is especially effective for water molecules because its microporous structure is suitable for selective uptake of small polar molecules. Nevertheless, a purely micropore-dominated system can suffer from diffusion limitations if mesoporous transport channels are insufficient. Therefore, the presence of a mesoporous component is beneficial for accelerating the movement of water vapor into internal adsorption sites.

KA-4 showed the most balanced performance among the investigated samples. In this adsorbent, bentonite provides the mineral framework, zeolite 4A provides selective microporous adsorption, activated alumina improves stability, and silica gel contributes to rapid moisture uptake in mesopores. The combination of these components forms a multi-level pore structure. Micropores are responsible for deep dehydration and low dew point, mesopores improve mass transfer, and the mineral binder phase maintains granule integrity. This structural balance explains the high dynamic capacity and long breakthrough time of KA-4.

Table 2. Representative dynamic drying results for KA-series composite adsorbents

Sample	Breakthrough time, h	Saturation time, h	Dynamic capacity, kg/kg	Dew point, °C	Efficiency, %
KA-1	3.1	5.8	0.061	−18	78.4
KA-2	4.6	7.4	0.084	−25	85.9
KA-3	6.7	10.2	0.119	−35	92.4
KA-4	8.6	12.8	0.146	−43	96.9
KA-5	8.1	12.1	0.149	−45	96.3
KA-6	7.5	11.5	0.137	−40	95.1

The representative results in (Table 2) show that dynamic capacity increased from 0.061 kg/kg for KA-1 to 0.146 kg/kg for KA-4. The corresponding drying efficiency increased from 78.4% to 96.9%, and the outlet dew point decreased from −18 °C to −43 °C. These changes demonstrate the effect of

component modification and pore structure development. KA-5 showed a slightly higher dynamic capacity of 0.149 kg/kg, but its higher zeolite content may increase regeneration demand and pressure drop. Therefore, KA-4 was evaluated as the optimal sample based on the total balance of capacity, dew

point, pressure drop, mechanical strength and regeneration stability.

The breakthrough curve is the key experimental output in determining dynamic adsorption capacity. At the beginning of the test, the outlet moisture concentration remains close to zero, indicating that the adsorbent bed retains almost all incoming water vapor. As the mass-transfer zone moves through the bed, the outlet moisture begins to increase. The breakthrough point is reached when C_t/C_0 becomes 0.05–0.10. At this moment, the adsorbent still contains unused capacity near the outlet, but the dry gas specification is no longer safely maintained. For process design, the bed should be switched to regeneration before full saturation to avoid water slip into the product gas.

The dynamic capacity is affected by gas flow rate. When gas flow increases, the contact time between gas and adsorbent decreases, and the mass-transfer zone moves faster through the bed. As a result, breakthrough occurs earlier and the useful capacity decreases. For KA-4, increasing the gas flow rate from 0.5 to 2.0 m³/h shortened the breakthrough time from 10.2 to 5.3 h and reduced dynamic capacity from 0.151 to 0.116 kg/kg. At the same time, the pressure drop increased from 4.2 to 18.9 kPa. This confirms that excessive gas velocity prevents full utilization of the adsorbent bed and increases energy consumption.

Bed height also has a significant effect. A higher bed contains more adsorbent and provides a longer mass-transfer zone reserve. Therefore, breakthrough time increases as bed height increases. However, an excessively high bed creates larger pressure drop and may require stronger compression. For KA-4, a bed height of 300 mm gave a breakthrough time of 8.6 h and a pressure drop of 7.8 kPa, which represents an acceptable balance. At 400 mm, breakthrough time increased further, but pressure drop became less favorable. Thus, the optimum bed height should be selected not only by maximum breakthrough time but also by hydraulic resistance and equipment limitations.

Temperature strongly influences physical adsorption. Water adsorption on solid desiccants is usually favored at lower temperature because adsorption is exothermic. As temperature increases, adsorbed water

molecules are more easily desorbed and the equilibrium capacity decreases. In the KA-4 data, increasing temperature from 20 to 50 °C decreased dynamic capacity from 0.152 to 0.111 kg/kg and worsened the outlet dew point from –46 °C to –29 °C. A practical operating interval of 25–35 °C was therefore recommended for adsorption, with 30 °C as a convenient laboratory regime.

Pressure can improve drying performance by increasing gas density and the contact probability between water molecules and adsorbent active sites. For KA-4, increasing pressure from 0.5 to 2.5 MPa improved breakthrough time and drying efficiency. At 2.5 MPa, the outlet dew point reached approximately –43 °C and the drying efficiency was 96.9%. At 4.0 MPa, the efficiency increased only slightly, while the equipment requirements became higher. This means that the most rational pressure should be selected by considering both technical performance and apparatus safety. For pilot and industrial operation, an interval of 2.5–4.0 MPa is acceptable for the proposed system.

Granule diameter is another important factor in dynamic capacity determination. Small granules reduce internal diffusion resistance and can improve mass transfer, but they also increase pressure drop and may cause bed compaction. Large granules lower pressure drop but slow down diffusion into the internal pore system. In the present technological assessment, 2–4 mm granules were selected as the recommended fraction. This size range provides an acceptable compromise between contact efficiency, mechanical stability and hydraulic resistance. It is also suitable for fixed-bed adsorbers operating in cyclic drying-regeneration mode.

Regeneration history must be considered when determining dynamic capacity. An adsorbent may show good initial capacity, but if regeneration is incomplete, residual water remains in the pores and the next cycle starts with partially occupied active sites. For KA-4, regeneration temperatures below 200 °C were insufficient for complete capacity recovery, while 220–240 °C provided a recovery level of about 94–96%. Regeneration at 260 °C gave only a small additional improvement but increased energy consumption and could affect long-term structural stability.

Therefore, a regeneration interval of 220–240 °C for 2–4 h is recommended.

The determination of dynamic capacity should also include reproducibility and mass balance control. The inlet moisture concentration must be kept stable, the flow rate should be measured accurately, and the outlet moisture analyzer must be calibrated. The mass of the adsorbent should be reported on a dry basis after regeneration. If the gas contains liquid droplets, heavy hydrocarbons or solid impurities, a separator and filter should be used before the adsorber, because contamination can block pores and distort the capacity value. Such precautions are necessary for obtaining reliable data and for transferring laboratory results to pilot-scale design.

The obtained results show that dynamic water adsorption capacity is a practical criterion for ranking adsorbents, but it must be interpreted together with dew point and breakthrough behavior. KA-5 can have slightly higher water uptake because of increased microporosity, but KA-4 has a better technological balance. The KA-4 sample provides a long service time, low outlet dew point, high efficiency and suitable regeneration behavior. Thus, KA-4 can be recommended as the main adsorbent for further studies on natural gas adsorption drying and for scale-up calculations.

Conclusion

1. Dynamic water adsorption capacity is one of the main criteria for evaluating adsorbents used in natural gas dehydration because it describes the useful amount of water retained under real flow conditions before breakthrough.

2. The determination of dynamic capacity should be based on breakthrough curves, outlet moisture concentration, dew point, saturation time, pressure drop and mass balance calculations.
3. The KA-series adsorbents show that modification of activated bentonite with zeolite 4A, activated alumina and silica gel significantly increases water uptake and drying efficiency.
4. KA-4 demonstrated the most balanced technological performance, providing a breakthrough time of 8.6 h, saturation time of 12.8 h, dynamic capacity of 0.146 kg/kg, dew point of about –43 °C and drying efficiency of 96.9%.
5. The optimal operating conditions for KA-4 include 2–4 mm granules, adsorption temperature of 25–35 °C, pressure of 2.5–4.0 MPa, adsorption cycle of 8–12 h and regeneration at 220–240 °C for 2–4 h.
6. Excessive gas flow rate decreases contact time and dynamic capacity, while excessive bed height increases pressure drop – P. Therefore, the optimum should be selected by balancing adsorption performance and hydraulic resistance.
7. Regeneration temperature strongly affects capacity recovery; for KA-4, 240 °C for about 3 h provides an optimal compromise between recovery efficiency and energy consumption.
8. The proposed dynamic capacity determination method can be used for comparing composite adsorbents and for designing pilot or industrial fixed-bed natural gas drying units.

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© Ashurov Sh. M., Bozorov O. N., Kuyboqarov O. E.

Contact: kuyboqarovo@mail.ru