

Adsorption Performance of Regenerated Molecular Sieves for Moisture Removal in Compressed Oxygen Gas

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ABSTRACT - This study focuses on evaluating the adsorption performance of regenerated molecular sieves repurposed as moisture adsorbents for compressed oxygen gas. The spent adsorbents are collected from a Phase Technology Freezing Point analyzer after saturation is achieved through repeated operational cycles. Two thermal regeneration methods are investigated, namely (1) staged heating in a furnace at 350 °C for 2 hours followed by a 30-minute vacuum treatment, and (2) staged heating in a gas chromatography (GC) oven at 350 °C with a dry-nitrogen purge for an hour, followed by another 1 hour without purging and a 30-minute vacuum duration. Furthermore, the physical characteristics after regeneration are assessed using the UOP Method 422 for particle size distribution (PSD) through micromesh sieving. Moisture adsorption performance is evaluated according to ASTM D1142 using a dew-point measurement with compressed oxygen at operating pressures and flow rates of 30–50 psi and 0.5–1.0 L/min, respectively. The results indicate that regenerated molecular sieves retain approximately 80–90% of the adsorption capacity of new material, which demonstrates that the GC-oven regeneration method exhibits slightly superior performance compared to furnace regeneration. These findings prove that appropriate thermal regeneration enables effective reuse of spent molecular sieves, providing a technically viable and economically favorable strategy for moisture removal in oxygen-drying applications while reducing laboratory waste.

Keywords: molecular sieve regeneration, moisture adsorption, yoon–nelson kinetics, compressed oxygen drying, dew point measurement.

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INTRODUCTION

Compressed oxygen gas is widely used across various industrial sectors, including chemical manufacturing, power generation, oil and gas processing, as well as laboratory and medical applications that require high-purity oxygen. During these applications, the moisture content must be maintained at low levels because the presence of water vapor can lead to several operational issues, such as corrosion of piping and other equipment, ice formation under low-temperature conditions, including interference with the stability and accuracy of analytical instruments. Additionally, trace amounts of moisture can affect analytical results in freezing point measurement instruments, potentially leading to incorrect data interpretation.

Adsorption technology using molecular sieves is the most widely applied method for achieving low moisture levels. These molecular sieves are microporous zeolite-based materials with a uniform pore structure on the angstrom scale, which enables the selective removal of molecules based on size and polarity (Breck 1974). Commonly adopted methods include adsorption, using zeolite as the adsorbent due to its high adsorption capacity, low energy requirements, cost-effectiveness, and well-established technology (Eviani Mitra et al., 2025). Meanwhile, the strong affinity of water molecules for zeolitic frameworks arises from a combination of physical adsorption and electrostatic interactions within the pore network, making molecular sieves highly effective for gas-phase drying applications (Ruthven 1984). Zeolite types 3A, 4A, 5A, and 13X have been used for gas drying and purification due to its stability, regenerability, and high-water adsorption capacity.

Adsorbents tend to become saturated, leading to a significant decline in water uptake capacity. In both laboratory and industrial practices, saturated molecular sieves are often replaced with new adsorbent material without undergoing regeneration. Considering that the method is convenient, it is economically inefficient and environmentally unsustainable due to the large amounts of solid waste it generates. As a result, adsorbent regeneration has become increasingly relevant for operational efficiency and

sustainability. Previous studies have shown that thermal regeneration can partially restore the adsorption capacity of zeolite molecular sieves. However, the effectiveness of regeneration depends strongly on the heating conditions and the adsorbent's operational history (Melchiori & Rodrigues 2016).

Based on this perspective, the Phase Technology Freezing Point instrument uses molecular sieves in the sample conditioning system to remove moisture prior to analysis. Repeated operation causes the adsorbent within the instrument to reach saturation, and it is typically discarded without further evaluation. Presently, no scientific studies have investigated the potential reuse of spent molecular sieves from Phase Technology instruments for other applications, namely drying compressed oxygen gas. This outlined a clear knowledge gap regarding the adsorption performance and regeneration feasibility of molecular sieves recovered from analytical instrumentation.

This study focused on evaluating the regeneration of saturated molecular sieves from a Phase Technology instrument. The entire process was carried out using two thermal methods in a standard laboratory setting, namely heating in a furnace or stepwise heating in a gas chromatography (GC) oven assisted by a dry nitrogen flow. Furthermore, GC is a common chromatography type used in analytical chemistry to separate and analyze compounds that can be vaporized without decomposition. Typical uses of GC include testing the purity of a particular substance and separating the components of a mixture (Nofrizal et al., 2013).

Chromatography refers to a physical separation method in which the components to be separated are distributed between two phases. This includes the stationary phase with a large surface area, and the mobile phase that permeates the stationary phase (Soekapradja & Herlina 1990).

The adsorbent was applied to remove moisture from compressed oxygen gas after regeneration, and its performance was evaluated using a Shaw Dew Point Meter in accordance with ASTM D1142. Therefore, this study aims to provide a

strong technical foundation for assessing the feasibility and effectiveness of reusing spent molecular sieves. The process offered a more economical and sustainable operational strategy for laboratories and facilities using similar analytical instruments.

METHODOLOGY

The current study consisted of four main stages: 1). Preparation of saturated molecular sieve samples; 2). Physical characterization using UOP 422 for particle size distribution (PSD); 3). Thermal regeneration using two different procedures; and 4). Breakthrough performance evaluation using ASTM D1142 with a shaw dew point meter. The stages were designed to provide a comprehensive understanding of how regeneration affected the physical properties and adsorption performance of the molecular sieve.

Preparation of saturated molecular sieve

Spent molecular sieve samples were obtained from a phase technology freezing point analyzer, in which the adsorbent was part of the sample conditioning system to remove moisture prior to analysis. The adsorbent reached saturation after repeated operational cycles and was removed from the instrument. The recovered molecular sieves were stored in sealed containers to prevent additional moisture uptake before regeneration and testing (Phase Technology 2020). The instrument automatically notified after approximately 300 analysis cycles, suggesting that the internal molecular sieve cartridge needed to be replaced due to a loss of adsorption capacity. The spent adsorbent was carefully removed from its compartment and transferred to an airtight container to prevent additional moisture uptake prior to regeneration or activation. This step ensured that the initial condition of the sample accurately represented real saturation conditions encountered during instrument operation.

Adsorbent characterization using UOP 422 (particle size distribution)

The physical characteristics of the adsorbent were examined using UOP method 422, a standardized procedure for determining PSD

through micromesh sieving (UOP 2009). This method was selected because of the suitability for evaluating changes in particle integrity, degradation, or mechanical wear encountered during usage and thermal regeneration.

Prior to sieving, the sample was dried and subsequently passed through a sequence of calibrated sieves with decreasing mesh sizes. The mass retained on each sieve was recorded and expressed as a percentage of the total sample mass. The resulting PSD curve was used to assess the following:

- Abrasion or mechanical attrition during instrument operation.
- Uniformity of particle size, which affected mass transfer, pressure drop, and adsorption efficiency.

The mass fraction was calculated using:

$$\text{Mass Fraction(\%)} = \frac{m_i}{\sum m_i} \times 100 \quad (1)$$

where m_i represents the mass retained on the i -th sieve.

PSD analysis provided a representative, and quantitative avenue. The essence was to determine whether the adsorbent remained physically suitable for reuse following the regeneration process (UOP 2009).

Regeneration procedures

Two thermal regeneration procedures were evaluated. The essence was to determine the most effective method for restoring adsorption capacity. The thermal regeneration procedures are as follows

- Furnace regeneration (stepwise heating)

The spent molecular sieve was placed in a heat-resistant glass beaker. Additionally, it was regenerated using a stepwise thermal treatment inside the furnace. The temperature profile consisted of:

- 100°C for 15 minutes (removal of free moisture)
- 200°C for 15 minutes (removal of weakly bound moisture)

- 350°C for 2 hours (deep desorption within zeolite pores)

The material was immediately transferred into a sealed molecular sieve container after heating, and subjected to vacuum treatment for approximately 30 minutes. The essence was to remove any residual water that may re-adsorb during cooling. Furthermore, stepwise heating minimized thermal shock that could cause cracking or structural weakening of the zeolite beads.

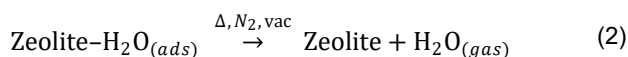
- GC oven regeneration with nitrogen purge (developed method)

The subsequent regeneration method was performed using a domestic GC oven. The sample was placed in a perforated stainless-steel tube and subjected to the following thermal steps:

- 100°C for 15 minutes
- 200°C for 15 minutes
- 350°C for 1 hour with dry nitrogen purge

The nitrogen purge was maintained at low flow rate to promote the stripping of desorbed water molecules from the pore structure. After an hour of purging, the nitrogen flow was stopped and heating was continued at 350°C for another 1 hour. Furthermore, the tube was removed and subjected to vacuum drying for 30 minutes.

The regeneration mechanism was stated as follows:



This method was intended to enhance desorption efficiency while minimizing re-adsorption during cooling. This was consistent with reported regeneration strategies for zeolitic adsorbents (Melchiori & Rodrigues 2016).

Breakthrough moisture adsorption test (ASTM D1142)

The adsorption performance of the regenerated molecular sieve was evaluated following ASTM D1142 – standard test method for water vapor content of gaseous fuels by measurement of dew-point temperature (ASTM International 2010).

ASTM D-1142 used three methods to determine/ calculate the water vapor content. This included: 1). Direct reading from a chart showing the relationship between water content, temperature, and saturation pressure; 2). Calculation based on Raoult’s law (ideal model); and 3). The use of Bukacek's correlation, responsible for providing a modified Raoult’s law expression with a deviation factor for improved accuracy in sweet natural gases (Handoyo & Suhendi 2022). Moisture measurements were carried out using a Shaw Dew Point Meter, which can detect ultra-low moisture levels suitable for high-purity oxygen applications (Shaw Moisture Meters 2018).

In this context, the regenerated adsorbent was loaded into a laboratory-scale glass adsorption column configured for downflow operation. Compressed oxygen gas was passed through the column at a constant flow rate of 0.5–1.0 L/min and operating pressure of 30–50 psi. Additionally, the outlet gas stream was continuously monitored using the Shaw meter to record dew-point temperature or ppmwt moisture content as a function of time.

The breakthrough curve was generated by plotting outlet moisture concentration versus time. At the beginning of the test, the outlet moisture remained low due to the high adsorption capacity of the fresh adsorbent. Meanwhile, as saturation progressed, the outlet moisture concentration gradually increased. The breakthrough time (t_b) refers to the time at which a significant increase in outlet moisture concentration was observed:

$$t_b = t \mid \text{moisture}_{out} \uparrow \text{significantly} \quad (3)$$

This parameter is used as a practical indicator of adsorption capacity. Additionally, the parameter enabled direct comparison of adsorption performance between different regeneration methods.

Yoon-Nelson mathematical model for breakthrough analysis

Breakthrough data were analyzed using the Yoon–Nelson kinetic model, which assumes the rate of decrease in adsorption probability is

proportional to both the probability of adsorption and breakthrough. This model was selected due to the simplicity and suitability for fixed-bed adsorption systems without requiring detailed information on adsorbent properties or bed geometry (Yoon & Nelson 1984).

The model is stated as follows:

$$\ln\left(\frac{C_{in} - C_{out}}{C_{in} - C_{out}}\right) = k_{YN}(t - \tau) \quad (4)$$

where:

- C_{in} = inlet moisture concentration (ppmwt).
- C_{out} = outlet moisture concentration (ppmwt).
- k_{YN} = Yoon–Nelson rate constant (min^{-1}).
- τ = time required for 50% breakthrough (min).

By plotting:

$$\ln\left(\frac{C_{in} - C_{out}}{C_{in} - C_{out}}\right) \text{ vs. } t \quad (5)$$

a straight line is obtained, where:

- slope = k_{YN} (6)

- intercept = $-k_{YN}\tau$ (7)

This allowed the computation of characteristic breakthrough time:

$$\tau = -\frac{\text{intercept}}{k_{YN}} \quad (8)$$

The model provided a quantitative basis for comparing the saturation behavior of the new adsorbent. This included the furnace-regenerated adsorbent, and GC+N₂.

RESULT AND DISCUSSION

Breakthrough moisture behavior

All adsorbent conditions showed a rapid increase in outlet moisture concentration at the initial stage (0–20 min), followed by a gradual approach toward saturation between 70–100 min. The initial outlet concentration for all cases started

at approximately 4.59 ppmwt, depicting comparable pre-conditioning performance upon start-up.

At moderate time intervals (25–45 min), the furnace-regenerated adsorbent showed the fastest moisture increase. It reached >30 ppmwt before both GC+N₂ and fresh adsorbents. The finding suggested a reduction in adsorption capacity and faster saturation. However, the fresh molecular sieve showed the slowest breakthrough trajectory. The GC+N₂ regeneration exhibited intermediate behavior, approaching the performance of the fresh adsorbent more closely than the furnace method.

These breakthrough trends suggested that the regeneration process significantly impacted pore accessibility and surface reactivity, finally dictating the adsorption kinetics and service life of the adsorbent. The finding is consistent with classical breakthrough behavior reported for moisture adsorption in zeolitic materials (Ruthven 1984; Wang et al., 2019).

Effect of regeneration method on adsorption performance

Comparative analysis showed a clear hierarchy of moisture adsorption performance:

$$\text{Fresh} > \text{GC} + \text{N}_2 \text{ Regeneration} > \text{Furnace Regeneration} \quad (9)$$

The superior performance of GC + N₂-regenerated sieves was attributed to the inert nitrogen environment, which prevented oxidative degradation and reduced uncontrolled re-adsorption of moisture during cooling. This was also observed in thermally regenerated zeolite molecular sieves (Melchiori & Rodrigues 2016). The furnace method, which lacked similar protection, allowed the partial collapse or contamination of pore sites due to rehydration, resulting in decreased adsorption efficiency.

Experimental results showed that furnace-regenerated sieves saturated nearly twice as fast as fresh material. Meanwhile, GC+N₂ regeneration retained approximately 80–90% of the original adsorption capability, confirming its suitability for reuse.

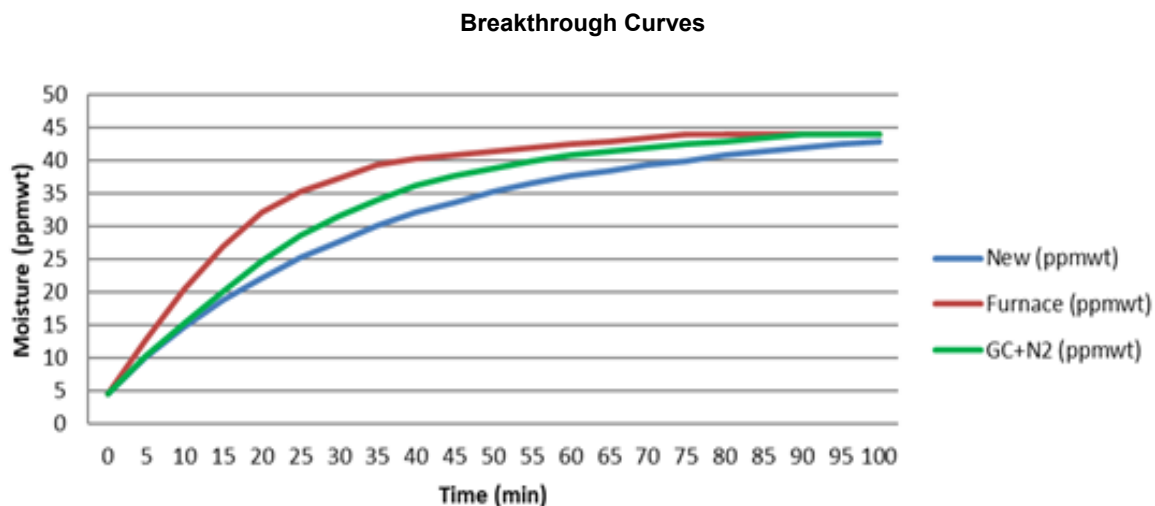


Figure 1. Shows the breakthrough curves of compressed oxygen moisture removal using fresh and regenerated molecular sieves

Yoon-Nelson kinetic modeling

The kinetic constants obtained using the Yoon–Nelson model is shown in Table 1. The model provided high accuracy, with determination coefficients (R^2) ranging from 0.94–0.97, validating its applicability for describing moisture adsorption breakthrough behavior in compressed oxygen drying systems (Yoon & Nelson, 1984). Similar accuracy levels for water vapor adsorption on zeolite-based adsorbents have been reported in previous studies using the Yoon–Nelson model (Azmi et al., 2016).

Table 1. Summary of the kinetic modelling using the Yoon-Nelson

Case	K _{YN} (1/min)	τ (min)	R^2
Fresh	0.048	24.63	0.9669
Furnace	0.078	13.07	0.9418
Regeneration			
GC+N ₂	0.065	20.14	0.9736
Regeneration			

The fresh molecular sieve had the lowest kinetic constant ($k_{YN} = 0.048 \text{ min}^{-1}$) and highest characteristic breakthrough time ($\hat{\delta} = 24.63 \text{ min}$), suggesting slower saturation and heightened adsorption capacity. However, furnace-regenerated adsorbents exhibited the highest kinetic constant ($k_{YN} = 0.078 \text{ min}^{-1}$) and the shortest $\hat{\delta}$ value (13.07 min), indicating degraded pore structure and

reduced adsorption efficiency. The GC+N₂-regenerated adsorbent exhibited intermediate kinetic behavior, similar to the fresh material. This suggested the effective recovery of adsorption sites following regeneration.

Mechanistic interpretation

Zeolite-based molecular sieves removed water vapor through strong electrostatic interactions between polar water molecules and extra-framework cations located within the microporous structure of the lattice. The uniform pore size and high surface polarity of the materials provided a strong driving force for moisture adsorption, particularly in gas-phase drying applications (Breck 1974; Ruthven 1984). The effective regeneration of molecular sieves required complete desorption of adsorbed water while preserving the integrity of the microporous framework and active adsorption sites.

In the case of furnace regeneration, the adsorbent was exposed to elevated temperatures in an open or non-inert environment. Although thermal energy desorbs water molecules during heating, the absence of a controlled atmosphere during cooling allows rapid rehydration of the zeolite surface upon exposure to ambient moisture. In addition, repeated thermal cycling without inert protection often led to localized structural stress, partial pore blockage, or contamination of

adsorption sites. This eventually reduced the effective adsorption surface area (Ruthven, 1984). The diverse effects are consistent with the higher kinetic constant (k_{YN}) and lesser breakthrough time ($\hat{\delta}$) observed for furnace-regenerated molecular sieves.

Following the description above, the use of a GC oven with dry nitrogen purging during regeneration provided a more controlled desorption environment. The continuous flow of inert nitrogen gas facilitated the removal of desorbed water vapor from the vicinity of the adsorbent surface, thereby preventing re-adsorption during both heating and cooling stages. This method preserved pore accessibility and maintained a higher fraction of active adsorption sites, resulting in adsorption kinetics and breakthrough behavior similar to fresh molecular sieves. In addition, similar benefits of inert-gas-assisted thermal regeneration for zeolitic molecular sieves have been reported in previous studies, particularly for gas drying applications (Melchiori & Rodrigues 2016).

The mechanistic differences between the two regeneration methods directly explained the observed variations in Yoon–Nelson kinetic parameters. Lower k_{YN} values and longer $\hat{\delta}$ times for GC+N₂-regenerated adsorbents suggested slower saturation and more effective use of adsorption sites. Meanwhile, furnace regeneration led to a rapid breakthrough due to partial loss of pore functionality. These findings outlined the importance of controlled regeneration environments in extending the service life and maintaining the adsorption performance of molecular sieve materials.

Practical implications: oxygen cylinder drying performance

Field application performance showed that the regenerated GC+N₂ adsorbent maintained low moisture levels (<5 ppmwt) for 6–8 months in oxygen-cylinder analysis systems, similar to that of fresh molecular sieves. The furnace-regenerated material degraded more rapidly, with outlet moisture reaching 35.00 ppmwt, serving as the operational breakpoint for replacement. The Yoon–Nelson model was adopted to interpret laboratory

kinetic behavior in correlation with real-time service performance. This reinforced the value of predicting adsorbent lifespans in analytical oxygen drying systems.

Summary of findings

GC + N₂ regeneration restored 80–90% of the adsorption performance compared to the fresh material. Furnace regeneration resulted in faster saturation and reduced adsorption performance due to uncontrolled rehydration and thermal stress. Yoon–Nelson kinetics provided a robust predictive tool, which strongly correlated with laboratory and operational adsorption behavior. Moisture breakthrough limit (35.00 ppmwt) conformed with $\hat{\delta}$ -derived service performance. This validated the model applicability for replacement scheduling.

CONCLUSION

In conclusion, this study showed that the regeneration process played a critical role in determining the effectiveness of molecular sieve adsorbents for moisture removal from compressed oxygen. Fresh molecular sieve had the greatest adsorption performance, as depicted by the lowest Yoon–Nelson rate constant ($k_{YN} = 0.048 \text{ min}^{-1}$) and highest characteristic time ($\tau = 24.63 \text{ min}$). This corresponded to the slowest saturation and largest effective adsorption capacity.

Regeneration using a GC oven with nitrogen purging produced approximately 80–90% of the original adsorption capacity. The regenerated adsorbent produced intermediate kinetic parameters ($k_{YN} = 0.065 \text{ min}^{-1}$, $\tau = 20.14 \text{ min}$). This confirmed that nitrogen purging effectively promoted water desorption while preserving the zeolitic microporous structure. Furnace regeneration led to minimal adsorption performance, characterized by the highest rate constant ($k_{YN} = 0.078 \text{ min}^{-1}$) and lowest characteristic time ($\tau = 13.07 \text{ min}$). This behavior was attributed to uncontrolled re-adsorption of moisture during cooling and potential structural degradation caused by high-temperature treatment in the absence of inert gases.

The Yoon–Nelson model provided an excellent description of the breakthrough data, with R^2 values between 0.94 and 0.97. These results showed that the model was a reliable tool for evaluating adsorption kinetics as well as comparing different kinetics with regeneration strategies. The practical replacement criterion of 35.00 ppmwt outlet moisture was consistent with the kinetic trends predicted by the Yoon–Nelson model. This could be applied as a technical guideline for scheduling adsorber replacement or regeneration in oxygen freezing point analysis systems.

GC+N₂ regeneration was generally recommended as the preferred strategy for reusing spent molecular sieves in compressed oxygen drying applications. This offered an optimal balance between adsorption performance, operational reliability, and waste minimization.

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GLOSSARY OF TERMS AND SYMBOLS

Terms & Symbols	Description	Unit
C_{out}	Outlet moisture concentration after passing through adsorbent bed	ppmwt
C_{in}	Inlet moisture concentration entering the adsorption bed	ppmwt
k_{YN}	Yoon–Nelson adsorption rate constant	min ⁻¹
τ	Yoon–Nelson characteristic time for 50% breakthrough	min
t	Total adsorption time within breakthrough experiment	min
$\ln\left(\frac{C_{out}}{C_{in} - C_{out}}\right)$	Linearized Yoon–Nelson model function	
R^2	Coefficient of determination (goodness of fit for	
GC	Gas Chromatography	
GC+N ₂	GC oven regeneration with nitrogen purge	
N ₂	Nitrogen gas	
ppmwt	Parts per million by weight	
MS	Molecular sieve	
FP Analyzer	Freezing Point Analyzer	
QC	Quality Control	
ASTM D1142	Standard test method for determining water vapor content in gaseous fuels via dew-point measurement.	
Adsorption	Surface-based capture process of molecules (e.g., water) on a porous solid medium.	
Molecular Sieve	Microporous aluminosilicate material (zeolite) used to selectively adsorb moisture based on molecular size and polarity.	

Regeneration	Process of restoring adsorption capacity by removing previously adsorbed moisture using heat and/or purge gas.
Desorption	Release of adsorbed molecules (water) from the adsorbent surface due to heating or purging.
Saturation	Condition at which the adsorbent can no longer remove moisture effectively.
Freezing Point Analyzer (Phase Technology)	Instrument used to measure fuel freezing point and require moisture-free sample gas.
Shaw Dew Point Meter	High-sensitivity instrument for measuring dew point or moisture content (ppmwt) in gaseous samples.
PSD (Particle Size Distribution)	Quantitative distribution of adsorbent particle sizes determined using sieve analysis (UOP Method 422).
GC Oven (Gas Chromatography Oven)	Heating chamber used in this study for controlled regeneration with nitrogen purging.
Breakthrough Curve	Plot of outlet concentration vs. time indicating the progression of adsorbent saturation.

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