Dehydration of Nitrogen Gas Using Hierarchical Sodium P Zeolite as a Solid Desiccant

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Abstract
In this study, hierarchical NaP zeolite was prepared for dehydration of a wet nitrogen gas stream using an adsorption system. Hierarchical NaP zeolite was synthesized via an easy hydrothermal process and characterized using several techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), BET surface area analysis and Fourier transform infrared (FT-IR) spectroscopy. An adsorption system comprising a desiccant packed column, humidity analyzer, and dry and wet gas cylinders was designed to evaluate the adsorption behavior of the synthesized NaP zeolite. Results indicated that hierarchical structure of NaP zeolite made a high surface area for water removal from a non-corrosive gas stream at ambient condition as it could reduce the water content of the gas from an initial concentration of 53 ppmv to approximately 5 ppmv after just one minute of the experiment.

1. INTRODUCTION

The presence of water vapor has been considered as a critical concern in many industries such as chemical and petrochemical industry, cosmetic, food conservation and processing, paper making, architecture materials, microelectronic, etc. [1,2]. On the other hand, due to the crucial needs in natural gas resources as fuels, water content elimination from this fuels have great importance as it can cause corrosion and hydrate formation ending in pipeline blockage [3,4]. There are different strategies to remove water vapor or humidity from gas streams involving supply a solid or liquid desiccant, membranes, refrigeration, supersonic methods and so on [5,6]. An adsorption dehydration route using a solid bed is a process in which a solid desiccant with some effective properties like high surface area, adsorption capacity, mechanical strength as well as being inexpensive, non-corrosive/toxic and chemically-inert serves for removing water vapor from a gaseous mixture [7,8]. Covering these features, the gas stream could reach too low water content and dew point temperature less than -50 $^{\circ}$C [3]. Up till now, various studies have been devoted to assessing water sorption capabilities of several adsorbents including silica materials, bio- and halide-based compounds, metal organic frameworks, functionalized polymers and etc. [1]. In an interesting study reported by Zhang et al., composite sorbents of calcium chloride-impregnated silica gel were prepared and thermo-

gravimetric measurements of water adsorption were fulfilled [9]. The results indicated that original macro-porous silica gel had the highest adsorption rate while the presence of $CaCl_2$ in the composite porous structure played a key role to enhance the adsorption capacity and its amount had to be optimized. Wei and co-workers synthesized hierarchically-structured aluminosilicates and compared their water adsorption abilities with those of 3A zeolite and silica gel [1]. Former showed more water uptake and desorption rate due to the high and strong acid sites as well as bimodal porosity in their structures, respectively. Farag et al. investigated the natural gas dehydration using a pilot scale adsorption dehydration unit and 3A molecular sieve as a solid desiccant [7]. The effect of gas flow rate and water vapor concentration on dehydration of natural gas was studied. They claimed that with inlet water vapor concentration of 340 ppm, increasing the gas flow rate from 50 to 120 L h^{-1} decreases the maximum percentages of water vapor removal from 92 to 84 % due to the decrease of the residence time of the gas by increase in its flow rate. Moreover, increasing the inlet gas water content from 340 to 550 ppm resulted in an increase of driving force, and consequently dehydration efficiency enhanced from 86 to 91 %. Very recently in 2020, Pliekhov and co-workers synthesized zeolite FAU type-Y [10]. Dealumination modification treatments of the prepared zeolite were performed using three

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procedures as steaming, EDTA (Ethylenediaminetetraacetic acid) and HCl dealumination to study the water adsorption behavior of the zeolite samples. The results demonstrated that secure and controllable process of Al removal from zeolite Y framework with EDTA (increasing the Si/Al ratio), in comparison with steaming and HCl dealumination, significantly changed the hydrophilic property of the modified zeolite; the changes were as reduction in original intense water affinity by forming silica rich area on the surface of the zeolite. In addition, it was found that increasing the Si/Al ratio (optimum Si/Al = 4 or 30% of removed Al) lowers desorption temperature by reducing the strength of the water interaction. Besides, zeolite molecular sieves 3A and 4Awere used to selectively dehydrate the alcoholic mediums (ethanol, n-propanol and n-butanol) by some researchers [11]. In fact, in order to reach very low water content, the most suitable desiccants are zeolitic molecular sieves whose adequacy originate from their ordered porous structure and influence on their surrounding environment. They impose great physical forces on the molecules around them, especially polar ones, as they contain cations in their channels and cavities attracting negative-charged end of molecules; the more polar molecules, the higher affinity would be caused resulting in high adsorption capacity even at low partial pressures [7,12]. Zeolites, with various framework topologies formed by primary SiO_4^{4-} and AlO_4^{5-} tetrahedral sharing their oxygen atoms in the corners, are recognized as the well-known micro-porous molecular sieves which exhibit extensive adsorptive, catalytic and ion-exchange applications [13-18]. In this regard, Barrer and co-workers described a synthetic structure for the first time as NaP zeolite possessing intersecting channels of 0.31×0.44 nm and 0.26×0.49 nm and framework topology type of Gismondine (GIS) with respect to the International Zeolite Association (IZA) [19]. Water softening (detergent builder), removal of heavy metals and radioactive species such as U, Sr and Cs from wastewaters, being as catalytic supports as well as separation of tiny liquid or gas molecules are among the enormous applications of NaP zeolite [20-23]. Taking gas dehydration systems using zeolites into account, hydrophilic zeolitic structures such as FAU (Faujasite) and LTA (Linde Type A) have been exploited commercially to dry wet gas streams via adsorption dehydration processes. However, to the best of our knowledge, there is no report on NaP zeolite utilization as a desiccant in a solid bed to dehydrate a gas stream. Therefore, in this study, a pure phase hierarchically-structured NaP zeolite was easily prepared and the humidity adsorption behavior of the sample was investigated while had been packed in a column for dehydration of a wet nitrogen gas stream at ambient temperature and pressure.

2. EXPERIMENTAL

2.1. Chemicals
The reagents used for the preparation of hierarchical NaP zeolite were sodium silicate solution (water glass, Merck, 27% SiO_2 and 8% Na_2O) as silicon source and sodium aluminate (Aldrich, 53% Al_2O_3 and 42.5% Na_2O) as aluminum supplying precursor, and sodium hydroxide (Ameretat Shimi- Iran). Moreover, all solutions were provided by using distilled water to dissolve starting materials and prepare the synthetic zeolite gel.

2.2. Preparation of hierarchical NaP zeolite
Hierarchical NaP zeolite was hydrothermally synthesized similar to what reported in previous works [24,25]. The synthesis gel formula was $14.0Na_2O: 18.0SiO_2: 1.0Al_2O_3: 840.0H_2O$, in molar ratio of precursors. At first, an alkali solution was prepared by dissolving 3.36 g of NaOH pellets in 70 mL of distilled water. Thereafter, an appropriate amount of sodium aluminate powder (1.92 g) was added to the solution as stirring. The next step involved addition of 15.64 mL of sodium silicate solution, droply, to the mixture. After agitation and subsequently transferring into a stainless steel autoclave, the zeolite gel underwent crystallization treatment at 100°C in an oil bath for 12 hours. Finally, the slurry was filtered and washed with distilled water until pH~ 7 and the end product (white powder) was dried in an oven overnight (at ~ 100 °C).

2.3. Characterization and measurement
Nitrogen adsorption–desorption analysis (at -196 °C) disclosed the porosity characteristic of hierarchical NaP zeolite (degassed at 305 °C) on a Belsorp-Max equipment (BEL Japan Inc., Japan). BET specific surface area and pore size distribution of the sample were determined by BET (Brunauer, Emmett, and Teller) and BJH (Barrett–Joyner–Halenda) equations, respectively. A scanning electron microscope (SEM, VEGA‖TESCAN) and an energy dispersive x-ray spectroscopy (EDS) system (IDEFIX setup, SAMx company) were used to record the morphological properties and elemental composition of the product, respectively. In addition, investigation of the infrared spectral features of the NaP zeolite was performed on a FT-IR spectrometer (SHIMADZU-8400-s) and an x-ray diffractometer (JEOL-JDX 8030) recorded the x-ray diffraction pattern (XRD) while operating at 30 kV and 20 mA by Cu-Kα radiation. Moreover, the transmission electron microscopy (TEM) images were observed by a

Philips CM300 apparatus and dew point (moisture content) measurements were carried out using a commercial automatic dew point meter (SHAW, model SADP, UK).

2.4. Dehydration system

In order to evaluate the humidity removal process from wet nitrogen gas using prepared zeolitic adsorbent, a set-up illustrated in Fig. 1(A) was designed. The main parts of the set-up are composed of two wet and dry nitrogen cylinders with approximately 53 and 4.3 ppmv water content, respectively, adsorption column and humidity analyzer. The pipes and container material was 316-stainless steel due to the non-corrosive nature of nitrogen gas. The wet nitrogen gas cylinder was prepared as described below (Fig. 1(B)):

First, N_2 gas cylinder underwent vacuum condition three times by opening the valves 1 and 3 and closing the number 2, so that the container and pipes would be completely cleaned. Then, valve 3 was closed and immediately after opening the valve 2, the syringe was depleted and water introduced into the cylinder (route 1). To fill the cylinder with nitrogen gas (volume of 40 liters and pressure of 150 bars), it was injected into the container by closing the valve 2 and via route 2. In the final stage, the cylinder was rolled several times to yield a homogeneous mixture of water in nitrogen gas.
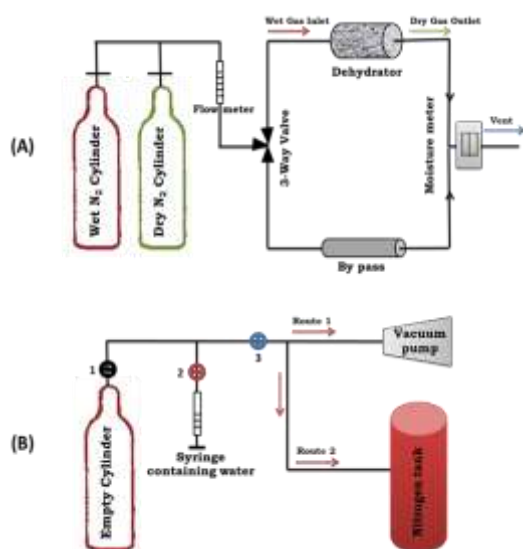

Fig. 1. (A) The experimental set-up designed for nitrogen gas stream dehydration and (B) preparation of a wet nitrogen gas cylinder.

2.5. Dehydration experiment

In order to get the set-up ready for dehumidification tests, zeolite samples (zeolitic powders were pressed into plates under pressure of

60 MPa to gain spherical particles with size of about 300 micrometers after meshing) were put into a furnace under nitrogen atmosphere (300 ˚C for 5 hours) before start of the trials. After that, different amount of sorbent was weighted and embedded in the adsorption column. A thick layer of ceramic wool was replaced in both side of the column to keep the sorbent fixed. Moreover, mesh screens were put in the entrance and output of the adsorption tube to prevent zeolite particles disturbing the analyzer performance. Afterward, the dry nitrogen gas was transferred through a bypass line and the water content was interred which envisaged to be the same amount of the initial water extent in dry N_2 gas. Subsequently, the gas flow direction was changed and dry nitrogen passed over the sorbent particles in order to eliminate the possible impurities and humidity in the medium. In the next step, wet nitrogen gas traversed along the bypass and then across the adsorption column. The amount of water in the outlet gas stream was gauged using an on-line moisture meter.

3. RESULT AND DISCUSSION

3.1. Structural characteristics of hierarchical NaP zeolite

X-ray diffraction pattern of hierarchical NaP zeolite synthesized at 100 ˚C during 12 hours is depicted in Fig. 2. As can be seen, characteristic peaks of NaP framework appeared around 2 theta angles at 12.4, 17.6, 21.6, 28.1 and 33.3 degrees in good agreement with JCPDS card number 01-071-0962 [23, 26]. The presence of sharp peaks with high intensities denoted complete particle growth and crystallization and as a result, successful synthesis of NaP zeolite.

It should be noted that the observed split in some peaks is due to the two types of unit cell of NaP zeolite framework as tetragonal and cubic showing the same XRD patterns and just a slight difference owing to the distortion on the network; less symmetric tetragonal phase exhibits some doublets [19,27].
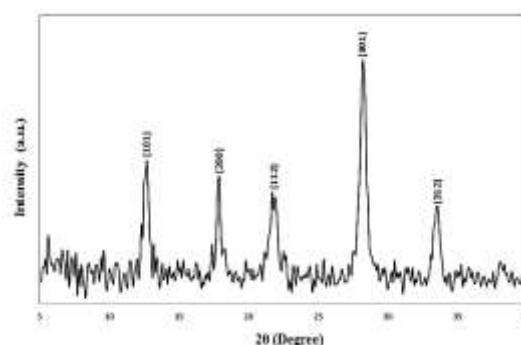

Fig. 2. XRD pattern of hierarchical NaP zeolite synthesized at 100 ˚C and 12 h (oil bath heating).

Fig. 3 represents the FT-IR spectrum of the NaP zeolite. In general, infrared transmission bands assigned to GIS framework appear in far and middle IR. Asymmetric stretching vibration of Si- and/or Al-O is obvious at wavenumber 1015 cm^{-1} (the strongest band) and symmetric one is shown at wavenumber 745 cm^{-1}[28]. Si- and/or Al-O bending vibration is also depicted at the wavenumber range of 605-430 cm^{-1} [23]. Moreover, the presence of trapped water molecules in zeolite channels and hydroxyl groups results in characteristic bands at wavenumbers 1640 and 3500 cm^{-1}, respectively [27].
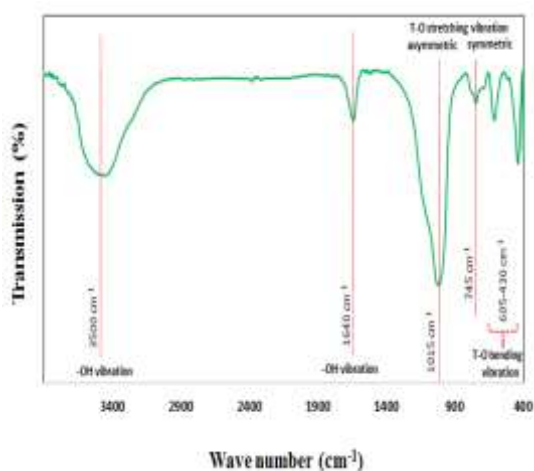

Fig. 3. FT-IR spectrum of hierarchical NaP zeolite synthesized at 100 ˚C and 12 h (oil bath heating).

SEM and TEM images of the synthesized hierarchical NaP zeolite are shown in Fig. 4. As can be seen, pure phase microspheres (particle size of about 2 μm) with well-grown knobby surface of hierarchical NaP zeolite are observable in the pictures which are similar to what reported before in the other studies [24,29,30]. Remarkably relevant to the assumption that a pseudo-spherical morphology would be obtained if NaP zeolite is synthesized with Si/Al about 2 or more, herein, the Si/Al of the prepared NaP zeolite is determined 2.27 in accordance with EDS analysis (Fig. 4) [24,28,31].

N$_2$ sorption isotherm of zeolite samples is represented in Fig. 5. According to IUPAC classification, due to mesoporous nature of the synthesized NaP zeolite, the type IV isotherm is observed. Moreover, capillary condensation phenomenon and intergrowth of the crystallites ended in a hysteresis loop (H$_3$-type) at relative pressure range of 0.4-0.8 declaring slit-shaped pores in the porous structure of the zeolite [31,32,33]. BET specific surface area and the average pore size calculated from BJH pore size distribution curve were defined 68.56 m^2 g^{-1} and ca. 3.28 nm, respectively.

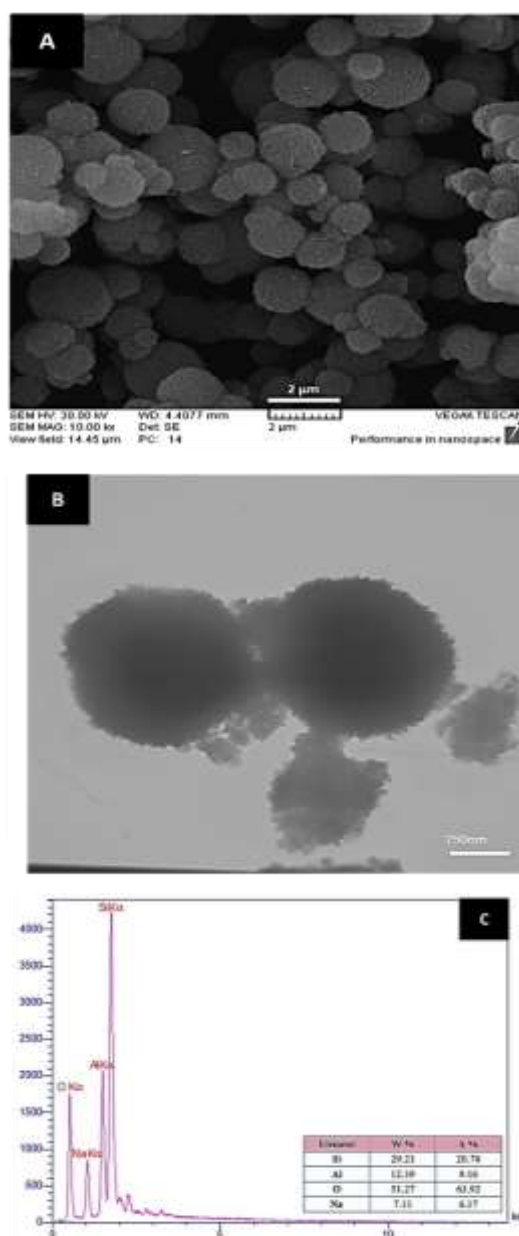**Fig. 4.** (A) SEM image, (B) TEM image and (C) EDS analysis of hierarchical NaP zeolite synthesized at 100 ˚C and 12 h (oil bath heating).
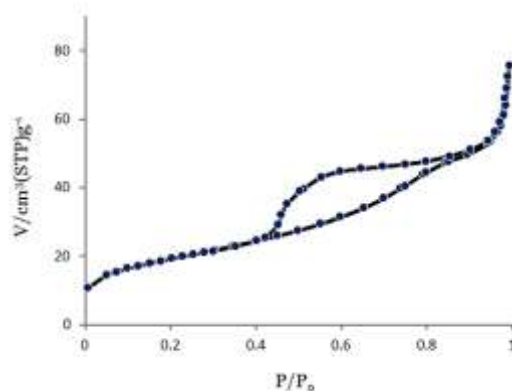

Fig. 5. N$_2$ sorption isotherm of hierarchical NaP zeolite.

3.2. Dehydration tests
3.2.1. Blank test
In order to calibrate the designed set-up, a blank experiment was fulfilled. In fact, to ascertain that the moisture removal efficiency was not due to the system component tendency to sorb water molecules, dry and wet nitrogen gas went through the column containing ceramic wool while no sorbent was therein. The curve of water content in the passing gas as a function of time has been plotted in Fig. 6. The given data imply that ceramic wool or body of the column and pipes are not responsible for water removal because as time goes by, the curve is flat and the moisture content is constant at the initial concentration (53 ppmv).
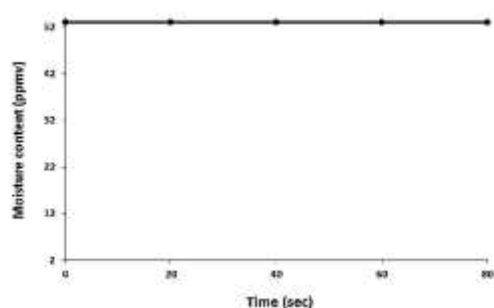

Fig. 6. Blank test (T: 27 ˚C, P: 100 KPa, initial moisture concentration: 53 ppmv, flow rate: ca. 3000 mL/min and no zeolite sample). Humidity adsorption was not occurred by the system components.

3.2.2. Wet nitrogen gas stream dehydration
The dehydration performance of hierarchical NaP zeolite (2.5 g) was assessed in breakthrough experiment at 27 ˚C and pressure 100 KPa. The water vapor was considered broken through the adsorption bed as its amount reached 2.4 ppmv in the effluent stream [34]. The breakthrough curve of dehydration process is illustrated in Fig. 7.
As it is obvious, the only 1 minute was sufficient to remove 90% of whole water extent of the moist nitrogen gas. That decreasing trend continued until the portion of water molecules got 3 ppmv at time

11 minutes. The water concentration in the passing nitrogen gas across the dehydration column reached 2.4 ppmv (dew point – 70.5 ˚C) at time 27 minutes with 95.5 % efficiency of the sorbent. Thereafter, partial increase was observed in the value; it assumed that was the commencement of sorbent saturation.
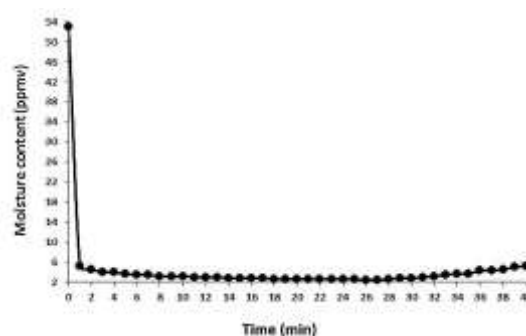

Fig. 7. Nitrogen gas dehydration (T: 27 ˚C, P: 100 KPa, initial moisture concentration: 53 ppmv, flow rate: ca. 3000 mL/min and mass of zeolite sample: 2.5 g).

Considering 27 minutes as the breakthrough time, the adsorption capacity of NaP zeolite was calculated as the amount of sorbed water (g)/ mass of zeolite (g) and the amount of sorbed water was determined as equation 1:

$$V= \Sigma(C_i-C_f)\times B\times \Delta t \qquad (1)$$

which V, C_i, C_f, B and Δt were amount of sorbed water (mL), initial water concentration (53 ppmv), final water concentration (ppmv) in the nitrogen gas passed through the desiccant bed, flow rate (3000 mL/min) and time interval (min), respectively. Accounting the sorption data, adsorption capacity was obtained 1.62 g g^{-1} for hierarchical sodium P zeolite. The results of other works reported on water adsorption behavior of various solid desiccants are collected in Table 1. Totally, outstanding results for high-speed gas drying process at ambient conditions were obtained using NaP zeolite synthesized in this study; however, more investigation on temperature and pressure variation is also proposed.

Table 1. Water adsorption capacity of different adsorbents.

Adsorbent	T (˚C)	P/P$_0$	Adsorption capacity (gg^{-1})	Ref.
CaCl$_2$	35	0.9	4.5	[9]
Macroporous silica gel	35	0.9	~ 0.6	[9]
Silica	35	0.4	0.18	[35]
13X-zeolite	35	0.4	0.11	[35]
Carbon molecular sieve (Carboxen 569)	30	-	~ 0.06	[36]
Carbon molecular sieve (Carbosieve SI11)	30	-	~ 0.09	[36]
kag-MOF-1	35	~ 0.0	~ 0.11	[37]
Ni/MOF-74	25	1.0	0.58	[38]
CuBTC	25	1.0	0.72	[38]
Mg exchanged- zeolite LTA	25	0.2	~ 0.29	[39]
Na- zeolite LTA	25	0.2	~ 0.22	[39]
Hierarchical NaP zeolite	27	1.0	1.62	This work

4. CONCLUSION

High-qualified hierarchically-structured sodium P zeolite was prepared at 100 ˚C in an oil bath heating medium during 12 hours. The product served as a solid desiccant in dehydration of wet nitrogen gas stream as a moist gas model. The results demonstrated that the moist nitrogen gas was deeply dehydrated (90%) only within 1 minute and drying treatment continued until 27 minutes with 95.5 % efficiency of the sorbent. The breakthrough capacity of NaP zeolite was obtained 1.62 g g^{-1}. This work suggests that the synthesized hierarchical NaP zeolite can be used as a potential candidate to remove water molecules from gaseous streams for widespread applications such as water harvesting from air or gas purification purposes.

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رطوبت‌زدایی از گاز نیتروژن با استفاده از زئولیت سلسله مراتبی NaP به عنوان خشک‌کن جامد

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چکیده

در این پژوهش، زئولیت سلسله مراتبی NaP جهت خشک نمودن جریان گاز نیتروژن مرطوب با استفاده از یک سیستم جذب سطحی تهیه شد. زئولیت سلسله مراتبی NaP از طریق یک فرآیند هیدروترمال آسان سنتز و با استفاده از تکنیک‌های مختلف مانند میکروسکوپ الکترونی روبشی (SEM)، پراش اشعه ایکس (XRD)، آنالیز مساحت سطح BET و طیف‌سنجی تبدیل فوریه مادون قرمز (FT-IR) مشخصه‌یابی گردید. به علاوه، یک سیستم جذب سطحی متشکل از ستون خشک‌کن، آنالایزر رطوبت و سیلندرهای گازی خشک و مرطوب به منظور ارزیابی رفتار جذبی زئولیت NaP تهیه شده، طراحی گردید. نتایج نشان داد که ساختار سلسله مراتبی زئولیت NaP مساحت سطح بالایی را برای حذف آب از جریان گازی غیر خورنده در شرایطی محیطی فراهم آورده است به طوری که تنها پس از گذشت یک دقیقه از زمان آزمایش، محتوای آب موجود در جریان گازی از غلظت اولیه ۵۳ ppmv به حدود ۵ ppmv تقلیل یافت.

واژه‌های کلیدی

زئولیت NaP، سلسله مراتبی، جریان گازی، جاذب، رطوبت‌زدایی