Study on Adsorption Characteristics of C_3F_8 in Retired Sulfur Hexafluoride Gas

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Abreact: With the gradual promotion of SF_6/N_2 hybrid insulating gas, the retired sulfur hexafluoride (SF_6) gas will multiply dramatically, which needs to be strictly purified and reused in order to reduce carbon emissions. However, the existing purification methods are difficult to remove the low concentration of C_3F_8 impurities in sulfur hexafluoride gas, which greatly affects the recycling of SF_6 gas. Based on this, this paper built a test platform to study the adsorption characteristics of C_3F_8 in decommissioned SF_6 gas, adopts three kinds of commercially available molecular sieve materials to simulate the adsorption process of SF_6 gas in GIS power equipment in the current engineering application, measures the adsorption performance of low concentration C_3F_8 (ppm level) impurities in decommissioned SF_6 gas on molecular sieve materials, and screens out the optimal adsorption properties on molecular sieve materials by combining with SEM, TG, FTIR, and BET characterizations. Based on the characterization of SEM, TG, FTIR and BET, the best adsorption material ST-100 molecular sieve was selected. And on this basis, the adsorption performance of ST-100 on low-concentration C_3F_8 was investigated in accordance with the changing law of the mass of adsorbent, inlet pressure and inlet flow rate, and the regeneration and stability of ST-100 was verified through the adsorption-desorption cycle. The results showed that ST-100 molecular sieve is an efficient adsorbent for C_3F_8, with the best adsorption performance at the temperature of 30 ℃, the mass of 1.5 kg, the inlet flow rate of 200 mL/min, and the pressure of 0.3 MPa, with the adsorption rate as high as 99%; and the adsorption rate was still maintained at 90% after five rounds of adsorption-desorption cycle tests, with good cyclic adsorption stability.

Keywords: Retired sulfur hexafluoride gas; C_3F_8 impurities; ST-100 molecular sieve; adsorption characteristics

1. Introduction

Sulfur hexafluoride (SF_6) gas is widely used in GIS power equipment because of its stable chemical properties [1,2], excellent insulating properties and efficient arc extinguishing performance. However, the greenhouse effect of SF_6 gas is extremely strong, and its global warming potential (GWP) is 23,500 times that of CO_2, which is one of the six greenhouse gases explicitly required by the Kyoto Protocol to limit emissions. In order to respond to the challenges in the field of climate and environment together and to improve the national independent contribution, China has set the strategic goal of "striving to achieve carbon peak by 2030 and carbon neutrality by 2060". The State Grid has responded positively to the call to

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reduce the use and emissions of the greenhouse gas SF_6 by developing a route to replace the sulfur hexafluoride/nitrogen (SF_6/N_2) gas mixture [3,4]. However, with the promotion of SF_6/N_2 gas mixture, China will face a large number of retired sulfur hexafluoride gases. If these retired SF_6 gases are directly emitted into the atmosphere, it will inevitably aggravate the greenhouse effect and global warming. In this situation, it is necessary to purify and refine the retired SF_6 gas impurities to realize recycling and reuse, in order to improve the environmental friendliness of power grid operation [5].

The traditional SF_6 gas purification and treatment program mainly adopts distillation and deep-cooling separation, but these two methods have common shortcomings, e.g., low purification rate, high energy consumption, and do not conform to the concept of low carbon and energy saving [6]. And the C_3F_8 content in SF_6 gas is extremely low, and its chemical properties are similar to sulfur hexafluoride, the use of deep cooling separation purification can't meet the national standard GB/T12022-2014 《industrial sulfur hexafluoride》 [7] stipulated in the SF_6 gas C_3F_8 gas content requirements ($\leq$ 50ppmw), to the recycling and reuse of SF_6 gas work has brought a great obstacle. In contrast, the adsorbent separation method with mild processing conditions has the advantages of simple operation, strong selectivity, high adsorption efficiency and renewable adsorbent materials, which has been applied to the separation of various special gas impurities, such as the separation of CH_4/N_2, C_2H_6/C_3H_8, the storage of H_2, and the capture of CO_2 [8-10]. In summary, the use of more energy-saving and cost-effective adsorption separation technology to remove the low concentration of C_3F_8 impurities from decommissioned SF_6 gases is a promising purification technology [11], and there are few research reports on the use of adsorption separation method to remove C_3F_8. Measure the adsorption performance of low concentration of C_3F_8 (ppm level) in retired SF_6 gas on the molecular sieve material, combined with SEM, thermogravimetry, Fourier transform infrared spectroscopy and specific surface area analysis and other means of characterization, screened out the optimal adsorption of molecular sieve ST-100. On this basis, the variation of adsorption performance of ST-100 for low concentration C_3F_8 with adsorbent mass, inlet pressure and inlet flow rate was studied, and the renewability and stability of ST-100 molecular sieve were verified by adsorption-desorption cycle, in order to provide data reference for C_3F_8 removal by adsorption separation technology in power system engineering.

2. Adsorption principle and adsorbent selection

2.1 Adsorption principle

The principle of adsorption separation technology is to realize the purpose of separating gases according to the difference in the interaction force between adsorbent and adsorbent within the adsorbent pore [12,13], the adsorbent that is preferentially adsorbed interacts with the adsorbent in a stronger way. The action mechanism of adsorption separation technology mainly includes two kinds of chemical adsorption and physical adsorption. The former belongs to monolayer adsorption, the force formed between the adsorbent and the adsorbent is stronger, and the material obtained is more selective, but the desorption needs to consume more energy, which is unfavorable to the regeneration of the adsorbent. In contrast, physical adsorption is the formation of multilayer adsorption of adsorbent on the pore surface of adsorbent, and the force formed between adsorbent and adsorbent is usually a weak van der Waals force, which generally

yields relatively low selectivity, easy desorption of adsorbent adsorbate by adsorbate, and controllable and reversible adsorption process. Due to the low content of C_3F_8 impurities in the decommissioned SF_6 gas, in order to guarantee the reuse effect of the adsorbent, the adsorbents used in this paper are purified and purified by physical adsorption method of sulfur hexafluoride gas.

2.2 Adsorbent selection

For adsorption separation technology, the physical and chemical properties of the adsorbent directly determine the adsorption performance. At present, SF_6 gas impurity adsorbent materials commonly used in GIS power equipment include activated alumina, molecular sieve, silica gel, activated carbon [14], etc. Compared with other types of adsorbent materials, molecular sieve is a porous adsorbent with specific and uniform pore size, which only allows molecules smaller than its microporous aperture to be adsorbed [15,16], and molecules larger than its size are excluded. Therefore, for the C_3F_8 molecules with larger size in the retired SF_6 gas studied in this paper, the adsorption and separation can be carried out by using molecular sieves with suitable pore sizes to realize the purpose of separating, purifying and recycling SF_6 gas.

3. Experimental Methods

3.1 Materials

Considering the actual concentration of C_3F_8 gas in the retired SF_6 gas, the adsorption gases used in this paper are low concentration C_3F_8 standard gas (C_3F_8 concentration of 100 ppm, concentration error of ±2 ppm, diluted background gas is SF_6) provided by Wuhan Newradar Gas Co., Ltd. And CMS was purchased from Guangzhou Chemxin Environmental Material Co., Ltd. The flow meter used in this experiment was purchased from Honeywell Characteristic Materials and Technology Group (VersaFlow series, the maximum flow rate of 1L/min), and the vacuum pump and gas chromatography analyzer (the carrier gas is He) were produced by T&P Union (Beijing) Co., Ltd.

3.2 Adsorption test platform

In order to explore the adsorption characteristics of C_3F_8 impurities in the retired SF_6 gases, this paper build a test platform as shown in Fig.1. The test platform includes three parts: vacuum activation system, adsorption system and gas component analysis system.

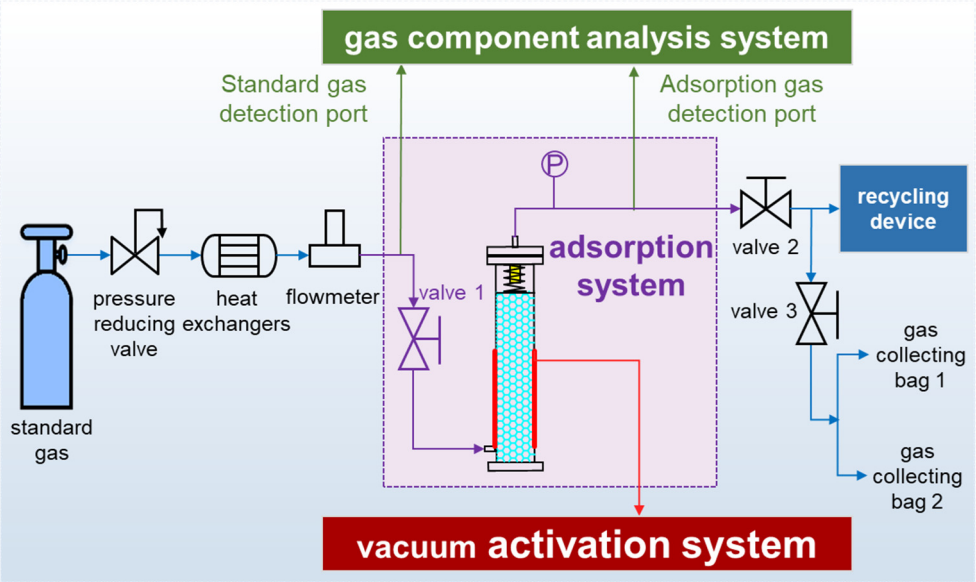

Fig. 1 Experimental platform for investigating the adsorption characteristics of C_3F_8 in decommissioned SF_6 gas.

The vacuum activation system in the above test platform consists of a vacuum pump and a heating device, which is used for vacuuming the test platform, heating and activating the adsorbent and regenerating the molecular sieve degassing. The test tank used in the adsorption system is a cylindrical stainless steel airtight container with an inner gas chamber volume of about 5L and openings at the top and bottom, which is used for filling adsorbent, pipeline vacuuming, C_3F_8 gas detection and test gas recovery. The adsorption tank is equipped with a pressure gauge at the detection interface, which can detect the absolute pressure in the tank in real time, as shown in Fig. 2. It should be noted that the interior of the adsorption tank should be wiped with alcohol before the start of each test in order to fully remove the impurities on the inner wall of the tank. In addition, the front end of the adsorption tank was equipped with a precision mass flow meter for regulating and controlling the flow rate of the standard gas inlet. The gas component analysis system is a gas chromatograph containing two pulsed helium ionization detectors (PDHID, detection accuracy of 0.01×10^{-6}), the detection is maintained at a constant column temperature of 50 ℃, the bridge current of 150 mA, the injection pressure of 100 kPa, and the measurement time of 12 min, and the quantitative detection is carried out after rinsing the quantitative ring and the pipeline with a sample flow rate of 50 mL/min for 1 min [17]. After passing through the molecular sieve, the concentration of C_3F_8 impurity components in the test gas was quantitatively detected. Since the retention value of C_3F_8 molecules under the same chromatographic conditions is certain, this paper utilizes the peak area external standard method to quantitatively calibrate the C_3F_8 components when the retention time is 9.26 min. The quantitative equations for the gas chromatograph are as follows:

$$C_i = \frac{C_s}{A_s} A_i = K_i A_i \tag{1}$$

Here, A_s is the chromatographic peak area of C_3F_8 in the standard gas; A_i is the chromatographic peak area of C_3F_8 in the test gas; C_s is the concentration of C_3F_8 in the standard gas, i.e., 100.00 ppm; C_i is the concentration of C_3F_8 in the test gas; and K_i is the absolute correction factor of C_3F_8 gas, i.e., 0.5403×10^{-3}.

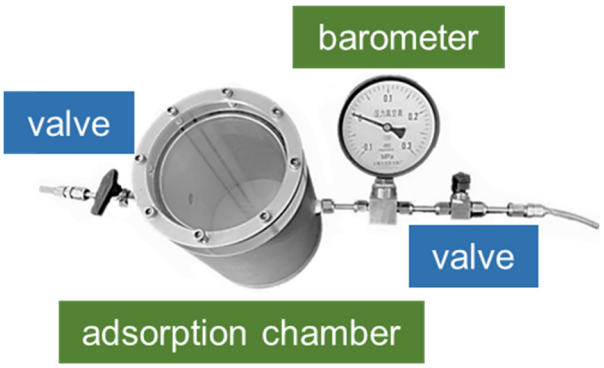

Fig. 2 Adsorption tank and pressure gauge connection.

3.3 Adsorption experimental steps

Before the adsorption test starts, the molecular sieve should be put into the vacuum drying oven at 150℃ for heating treatment to remove the residual water and other impurities in the molecular sieve, so that the adsorbent can be activated thoroughly. After activation, the treated molecular sieve is weighed according to the demand (each adsorbent filling quality not more than ± 0.1 g) and put into the above test tank, then the tank is completely sealed, and the vacuum activation system is started to heat up the adsorbent tank for 2.5 hours, and at the same time, the whole test platform including the adsorbent tank, pipeline and detection port is vacuumed for 3h to eliminate the influence of other gas impurities in the tank on the experiment. After the end of vacuuming, the standard gas valve was opened, and the standard gas of the target pressure was injected into the vacuum test tank at 30℃ to start the adsorption experiment. During the test, the concentration of C_3F_8 impurity components in the exit gas after the test was measured by sampling with a gas chromatograph, the test results were recorded, and the adsorption performance of the molecular sieve on C_3F_8 was evaluated by the change of gas concentration.

3.4 Adsorbent characterization

The micro-morphological structure of ST-100 molecular sieves was characterized and analyzed by a high-resolution cold field emission scanning electron microscope (SEM, Hitachi HitachiSU8010). The pyrolytic properties of ST-100 molecular sieves were studied by using a thermogravimetric analyzer (TGA/DSC3+, Mettler Toledo, Switzerland) under a 50 mL/min N_2 carrier gas flow with a 30 K/min heating rate [18]. The characteristic peaks of the C_3F_8 component in ST-100 molecular sieves before and after the adsorption test were analyzed using a Fourier Transform Infrared Spectrometer (FTIR, NicoletIS10, Thermo-Fisher Scientific, Inc.) at a wavelength of 4500-500 cm-1. The N_2 adsorption-desorption isotherms of the adsorbed materials at 77 K temperature were tested using a fully automated specific surface area and void analyzer (BET, Micromeritics ASAP 2460) and the specific surface area and pore size of the ST-100 molecular sieves were analyzed based on them [19].

4. Results and discussion

4.1 Adsorbent characterization

Three kinds of treated CMS, ST-1000, CF-100 molecular sieve adsorbent material with 1.5kg each weighing into the above test tank, respectively, in a vacuum state injected 0.1MPa standard gas for static adsorption, to be adsorption saturation (and the adsorption tank connected to the pressure gauge unchanged that is, "adsorption saturation After adsorption saturation (when the pressure gauge connected with the adsorption tank remains unchanged, it is called "adsorption saturation"), the concentration of C_3F_8 was measured by taking samples. In order to quantitatively analyze the adsorption effect of the above three molecular sieves on C_3F_8 fluorocarbons, this paper adopts the adsorption rate for reaction. The adsorption rate (φ) is calculated as follows:

$$\varphi = \frac{C_s - C_i}{C_s} \times 100\% \quad (2)$$

Same as above, where C_s is the concentration of C_3F_8 in the standard gas (100.00 ppm), and C_i is the concentration of C_3F_8 in the test gas.

As shown in Fig. 3(a), compared with the adsorption of CMS and CF-100 on C_3F_8 impurities in the standard gas, the adsorption performance of ST-100 molecular sieve on C_3F_8 impurities in SF_6 gas was obviously better, and its adsorption rate was close to 100%. As shown in the FTIR spectrum of Fig. 3(b), compared with the pre-adsorbed ST-100 material, the ST-100 molecular sieve after the adsorption test had a stronger C_3F_8 characteristic peak at 1596.14 cm-1 wavelength, indicating that ST-100 adsorbed the C_3F_8 molecule. Fig. 3(c) demonstrates the heat weight loss curve of ST-100 molecular sieve before and after the adsorption test, in which the horizontal coordinate is the temperature and the vertical coordinate is the weight percentage, reflecting the change curve of the adsorbent weight with the temperature during the heating process of ST-100 at 30 K/min elevated temperature. It can be seen from this thermal weight loss curve that compared with the pre-adsorbed ST-100 material, the adsorbed ST-100 material loses more weight with the increase of temperature, and the weight loss rate reaches the maximum at 99.82 ℃, and the weight loss curve of the adsorbed material of ST-100 tends to flatten out at 300 ℃, which indicates that the ST-100 material adsorbs a large number of C_3F_8 molecules, which is the same as the conclusion drawn from the above mentioned Fourier infrared spectroscopy analysis. This is the same as the conclusion drawn from the above FTIR spectroscopic analysis. Fig. 3(d) shows the morphology of the pre-activated ST-100 material, and Fig. 3(e) shows the morphology of the activated ST-100 material after heating and vacuuming, and it can be found that the activated ST-100 molecular sieve is a porous fluffy agglomerate with a specific and uniform pore size. the diameter of the C_3F_8 molecules is 0.74 nm, and its 3D structure is shown in Fi Fig. 3(f). Fig. 3(g) shows the N_2 adsorption-desorption isotherm and pore size distribution of ST-100 molecular sieve after vacuum activation at 77 K. It can be clearly seen that the N_2 adsorption-desorption isotherm of ST-100 exhibits a typical I-type curve, which indicates that ST-100 belongs to the microporous materials with pore size less than 1 nm. The BET specific surface area and pore volume of ST-100 were further calculated to be 170.6869 m^2/g and 0.1552 cm^3/g, respectively, and its pore size distribution was mainly concentrated at 0.76nm, and these uniformly distributed porous structures and suitable pore sizes provided more active sites for the adsorption of C_3F_8 impurities.

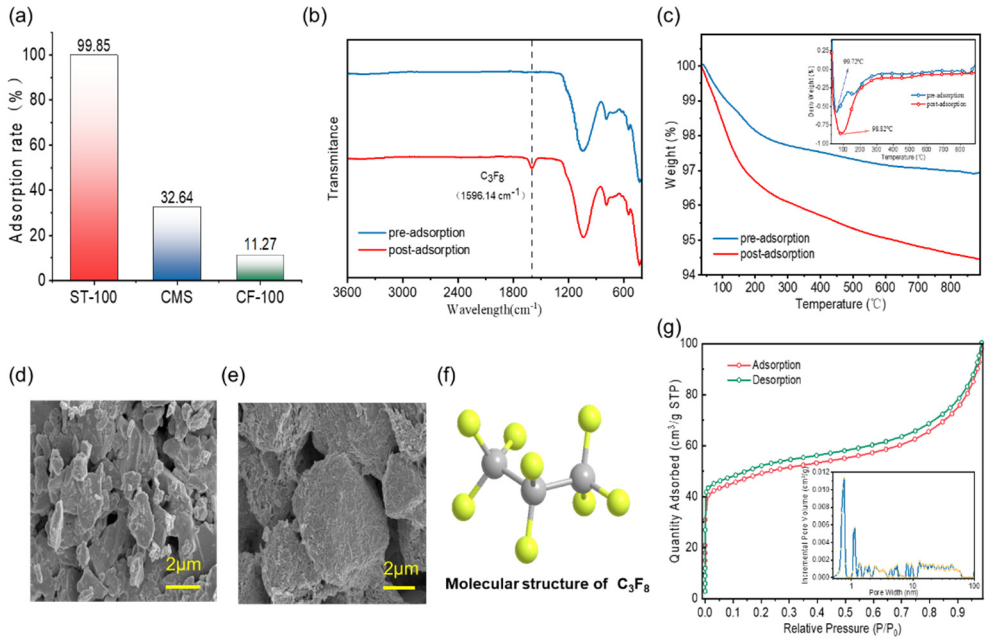

Fig. 3 (a) C_3F_8 adsorption efficiency of CMS, ST-100, and CF-100; (b) FTIR spectra of ST-100 before and after adsorption experiments; (c) TG spectra of ST-100 at 30 K/min heating rate before and after adsorption experiments; (d) SEM morphology of ST-100 before adsorption experiments; (e) SEM morphology of ST-100 after adsorption saturation; (f) 3D structure of C_3F_8 molecule; (g) N_2 adsorption/desorption isotherm and pore size distribution of ST-100 at 77 K temperature.

4.2 Effect of different factors on adsorption experiments

4.2.1 Effect of adsorbent quality on the adsorption of C_3F_8

The adsorbent loading method and test procedure were the same as the above test, and the treated ST-100 molecular sieves were put into the above test tanks at 0.5, 1.0, 1.5, 2.0, 2.5 kg each after weighing, and 0.1 MPa standard gas was injected into the adsorbent tanks for adsorption test at a flow rate of 100 mL/min under vacuum state, and the adsorption test was carried out after reaching adsorption saturation with a After reaching adsorption saturation, the concentration of C_3F_8 impurity component in the test gas was detected by sampling with gas chromatograph, and the adsorption rate of C_3F_8 was calculated and analyzed with the influence of the change of the mass of ST-100 adsorbent material by using Equation (2). As shown in Fig. 4(a), the adsorption rate of C_3F_8 exceeded 95% when the mass of adsorbent was 1.5 kg. From the test results, it is seen that with the increase of the mass of ST-100 molecular sieve, the adsorption effect of ST-100 on C_3F_8 becomes better with it, but the removal effect of C_3F_8 does not change significantly when the adsorbent exceeds a certain dosage.

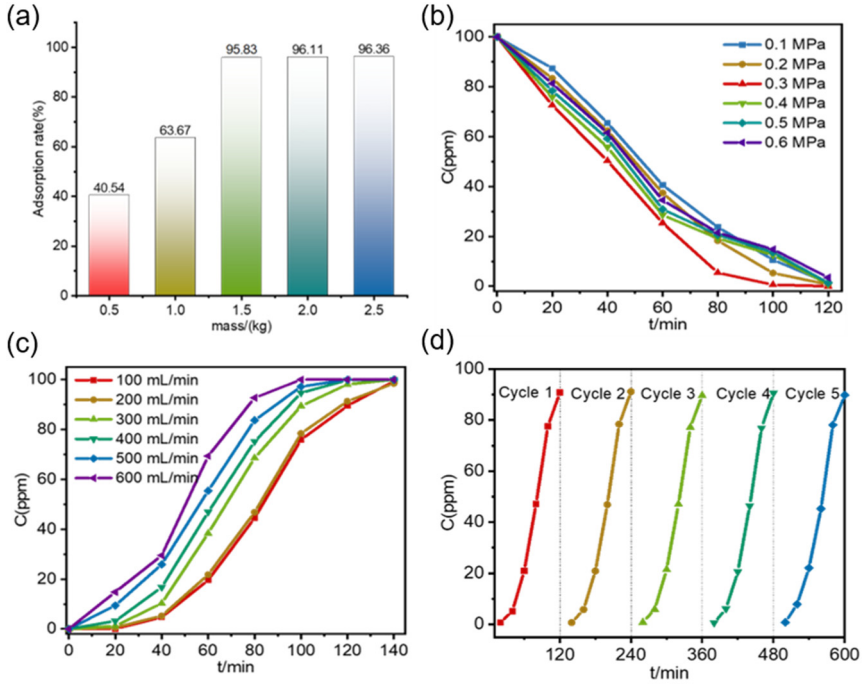

Fig. 4 (a) Effect of ST-100 molecular sieve mass on C_3F_8; (b) Adsorption curves of C_3F_8 gas content with time under different inlet pressures; (c) Adsorption curves of C_3F_8 gas content with time under different flow rates; (d) Cyclic stability of ST-100 molecular sieve for adsorption of C_3F_8.

4.2.2 Effect of inlet pressure on the adsorption of C_3F_8

The ST-100 molecular sieve treated by 1.5kg was put into the above test tank, and the inlet pressure of the standard gas was controlled at 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 MPa, respectively. The concentration C (ppm) of C_3F_8 impurity components in the test gas was determined by gas chromatograph every 20min, and the time t was used as the Abscissa and the C_3F_8 content in the gas to be detected as the ordinate. The adsorption curve of C_3F_8 content varying with time under different pressure was drawn. As shown in fi Fig. 4(b), when the inlet pressure is 0.3MPa, the adsorption rate and adsorption performance of ST-100 molecular sieve for C_3F_8 in the standard gas are obviously superior. The above experimental results show that the C_3F_8 content in the tank approaches to zero with the increase of adsorption time, but the higher the inlet pressure does not mean the better the adsorption effect, which may be due to the increase of C_3F_8 content in the tank. Therefore, when using ST-100 molecular sieve for subsequent adsorption experiments, 0.3MPa was selected as the appropriate inlet pressure.

4.2.3 Effect of inlet flow on the adsorption of C_3F_8

Referring to the literature, it is found that the gas flow rate has a great influence on the adsorption effect of molecular sieve, so the effect of ST-100 on the adsorption effect of C_3F_8 component in standard gas under different inlet flow rate was investigated. The ST-100 molecular sieve treated by 1.5kg was put into the above test tank, and the adsorption test was carried out by injecting the standard gas of 0.3Mpa into the tank at 100,200,300,400,500 and 600mL/min flow rate

respectively under vacuum. The concentration of C_3F_8 impurity component C (ppm) was detected by gas chromatograph every 20min, and the time t was used as the Abscissa and the C_3F_8 gas content in the gas was taken as the ordinate. The time-dependent adsorption curves of C_3F_8 gas at different flow rates are drawn, and the results are shown in Fig 4 (c). The experimental results show that with the increase of gas flow rate from 100mL/min to 600ml, the adsorption effect of C_3F_8 on STRMI 100 molecular sieve decreases gradually, and the adsorption saturation time shortens, from 140min to 85min. This may be due to the decrease of the contact time between C_3F_8 molecules and ST-100 adsorbents with the increase of inlet flow rate, which shortens the time for C_3F_8 to reach adsorption equilibrium. However, if the gas flow rate is too slow, it will reduce the purification efficiency of C_3F_8 impurities in SF_6 gas. Therefore, 200mL/min was selected as the appropriate gas flow rate.

4.2.4 Effect of inlet flow on the adsorption of C_3F_8

The cyclic adsorption stability of the material is crucial for its economic feasibility and sustainability. In this paper, the adsorbent mass was controlled to be 1.5 kg, the inlet pressure to be 0.3 MPa, and the inlet flow rate to be 200 mL/min, and five rounds of adsorption-desorption cyclic tests were carried out by utilizing the test platform constructed above and the length of the adsorption time of keeping the ventilation was 120 min in each round of the test, in order to verify the renewability and stability of ST-100. Among them, the desorption of ST-100 was accomplished by the vacuum activation system described previously, i.e., vacuum degassing and regeneration at 423K. As shown in Fig. 3(d), after five rounds of adsorption/desorption cycle tests of ST-100 on C_3F_8 in succession, the absorption rate of C_3F_8 remained almost unchanged at about 90%, indicating that ST-100 has good cyclic adsorption stability.

5. Conclusions

This paper built a test platform to study the adsorption characteristics of C_3F_8 adsorption in decommissioned SF_6 gases, and three molecular sieves, CMS, ST-100 and CF-100, which have been commercially available, were used to simulate the adsorption of SF_6 gases in the GIS power equipment, and the optimal adsorption molecular sieve, ST-100 adsorbent, was screened by combining the characterization means of SEM, TG, FTIR and BET, and the mass of adsorbent The effects of adsorbent quality, inlet pressure and inlet flow rate on C_3F_8 adsorption were investigated, and the cyclic stability of ST-100 was verified through several rounds of adsorption/desorption tests. The main conclusions are as follows:

(1) When the temperature is 30 ℃, the mass of adsorbents is 1.5kg and the adsorption pressure is 0.1MPa, the experimental platform built in this paper is used for static adsorption, and compared with CMS and CF-100 molecular sieves, ST-100 has better adsorption performance for C_3F_8 gas in SF_6 standard gas, and its adsorption rate is close to 100%.

(2) ST-100 molecular sieve is a porous fluffy aggregate with specific and uniform pore size. The specific surface area and pore volume of BET are 170.6869 m^2/g and 0.1552cm^3/g, respectively. The pore size distribution is mainly concentrated in 0.76nm. These uniformly distributed porous structures and suitable pores provide great possibility for the adsorption of impurities in C_3F_8 (molecular diameter is 0.74nm).

(3) The adsorption performance of ST-100 molecular sieve depends to a large extent on the mass of adsorbent, inlet pressure and inlet flow rate. When the mass of adsorbent is 1.5kg, inlet pressure is 0.3MPa and flow rate is 200mL/min, the adsorption performance is the best.

(4) After 5 rounds of adsorption-desorption cycle test with 120min aeration time, the adsorption rate of ST-100 is still maintained at 90%, with good cyclic adsorption stability.

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