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FREUNDLICH, LANGMUIR, TEMKIN AND HARKINS-JURA ISOTHERMS STUDIES OF H_2 ADSORPTION ON POROUS ADSORBENTS

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Abstract. The hydrogen adsorption and desorption isotherms of multiwalled carbon nanotube sample (MWCNT), an iron loaded multiwalled carbon nanotube (Fe_MWCNT), two zeolites (Na_Y_Zeo and NH4_Y_Zeo) and MCM-41 were measured at 77 K and atmospheric pressure by using the volumetric adsorption apparatus. The adsorption data were evaluated by several isotherm equations such as Langmuir, Freundlich, Temkin and Harkins-Jura isotherm models but were best described by the Freundlich isotherm model as it gave the highest correlation. The amount of adsorbed hydrogen by weight depended on the micropore volume of the sample, except for MWCNT and Fe_MWCNT. The porous samples were characterized by scanning electron microscopy (SEM) and N_2 adsorption isotherms. The maximum hydrogen storage of 1.96 wt % at 77 K was achieved by Fe_MWCNT. Microporous Na_Y_Zeo and NH_4_Y_Zeo showed higher hydrogen adsorption capacities than the mesoporous MCM-41. The hydrogen adsorption properties of these porous adsorbents may be further enhanced by different metal doping, thus paving the way for further study.

Keywords: hydrogen adsorption capacity, multiwalled carbon nanotube, zeolite, MCM-41, iron multiwalled carbon nanotube composite.

1. Introduction

The adsorption of hydrogen on porous adsorbents is widely studied for hydrogen storage applications at different pressures. In recent years, several porous materials such as activated carbon, single and multi-walled carbon nanotubes (SWCNT and MWCNT), zeolites, MCM-41 and metal organic frameworks (MOFs) have been proposed as good adsorbents for hydrogen adsorption [1-6]. Four methods for hydrogen storage can be anticipated: liquefaction, compression, physical sorption and storage in the form of metallic hydrides. Liquefaction and compression have major disadvantages. Hydrogen storage by physical adsorption on porous adsorbents can be an interesting method in stationary applications, wherein weight and volume are not limiting factors. It is a method in which H_2 molecules are weakly adsorbed on the surface of the adsorbent. One way to improve the kinetics of storage is to maintain the molecular identity of H_2 during the process which is possible by physical adsorption [7, 8]. Recent studies showed that the hydrogen adsorption capacities of carbon based porous adsorbents are quite promising, further studies are still needed in this field. Fierro ***et al.*** [7] investigated H_2 storage in activated carbon which exhibited a high specific surface area up to 2772 m^2/g and demonstrated a large hydrogen uptake of 5.3 wt %. Choi and Park [9] reported an adsorption capacity of 2.5 wt % at 77 K and 0.1 MPa for a sucrose-based microporous carbons with 1552 m^2/g specific surface area. Akasaka ***et al.*** [10] reported that porous carbon material having a specific surface area of 2070 m^2/g adsorbed 4.0 wt % of hydrogen at 77 K and 4 MPa.

MCM-41, which is a type of zeolite, is a member of mesoporous adsorbents family. MCM-41 has good chemical, mechanical and thermal stability, high surface area, uniform mesoporous structure, as a result of which it may be considered as a good adsorbent for hydrogen sorption [6]. Sheppard and Buckley [11] reported that pure MCM-41 samples having surface areas of 916–1060 m^2/g adsorbed 2.01 wt % of hydrogen at 77 K. Park ***et al.*** [12] have reported that the H_2 storage of MCM-41 at 298 K and 9.5 MPa was 0.68 wt % when incorporated with nickel (Ni-MCM41). Zeolites belong to the most prominent class of microporous materials which have been used in many applications in gas separations and catalysis. Recently, zeolites have also been considered as potential adsorbents for hydrogen adsorption [13]. Du and Wu [14] have reported that the maximum gravimetric hydrogen storage of NaX-type zeolite was 2.55 wt % at 4 MPa and 77 K.

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In this study, we have investigated the adsorption properties for H_2 on five typical adsorbents with different porosity and texture, ***i.e.*** multiwalled carbon nanotube, Fe-loaded multiwalled carbon nanotube, two types zeolites (Na_Y_zeolite and NH4_Y_zeolite) and MCM-41, using a low temperature adsorption apparatus.

2. Experimental

2.1. Iron-Multiwalled Carbon Nanotube Composite Sample

Commercial MWCNT (CNT Co., Korea) is produced by thermal CVD method with length of 1–25 μm, and diameter of 10–40 nm. In order to obtain a modified form, raw MWCNT was treated with $Fe(NO_3)_3$ in ethanol at 333 K. The treatment was carried out by adding 2 g MWCNT into a $Fe(NO_3)_3$ solution in ethanol (30 ml). The mixture was treated in a water bath at 333 K for 6 h. Then, the MWCNT sample was separated by filtration, washed with ethanol and dried at 373 K for 24 h. The resulting Fe^{+3}-grafted sample is designated as Fe_MWCNT.

2.2. Characterization of Porous Adsorbents

Na_Y_zeolite, NH4_Y_zeolite and MCM-41 were supplied by Sigma-Aldrich. Pore structure of the five adsorbents were evaluated by measuring N_2 adsorption and desorption isotherms at 77 K (Micromeritics TriStar II 3020). The specific surfaces were calculated using the Brunauer-Emmett-Teller (BET) and Langmuir equations. The total pore volume was calculated at a relative pressure $P/P_0 = 0.995$. The micropore volume was determined by the *t*-method analysis [15, 16].

2.3. Hydrogen Adsorption

Hydrogen adsorption experiments were carried out under the atmospheric pressure at 77 K. Measurements were carried out in an Autosorb iQ (Quantachrome) apparatus. Prior to measurements, 0.1 g of each sample was outgassed for 10 h at 473 K. The adsorption data were evaluated by several isotherm equations such as Langmuir, Freundlich, Temkin and Harkins-Jura isotherm models.

3. Results and Discussion

3.1. Characterization of Porous Adsorbents

Nitrogen adsorption-desorption isotherm is a method for obtaining a comprehensive characterization of porous adsorbents with respect to the specific surface area and porosity [17, 18]. Nitrogen adsorption-desorption isotherms of two zeolites (Fig. 1) are a mixture of type I and type IV isotherms with hysteresis loops caused by a capillary condensation in the mesopore, which means that zeolite samples have a combination of microporous and mesoporous structures. The pore structures of two zeolites were calculated by the *t*-method from the adsorption branch of the N_2 isotherms. Table 1 shows the BET and Langmuir surface areas, total pore volumes and average pore sizes for the porous adsorbents. The BET and Langmuir surface areas of the Na_Y_Zeo are larger than the surface areas of NH4_Y_Zeo. Total pore volumes of Na_Y_Zeo and NH4_Y_Zeo were 0.368 and 0.358 ml/g, respectively.

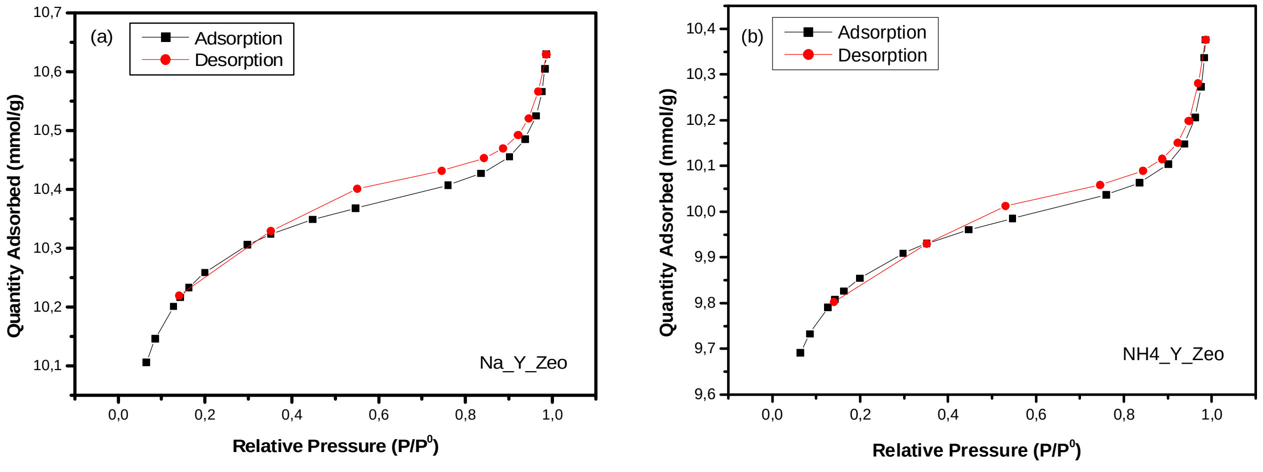


Fig. 1. The adsorption-desorption isotherms of Na_Y_Zeo (a) and NH_4_Y_Zeo (b)

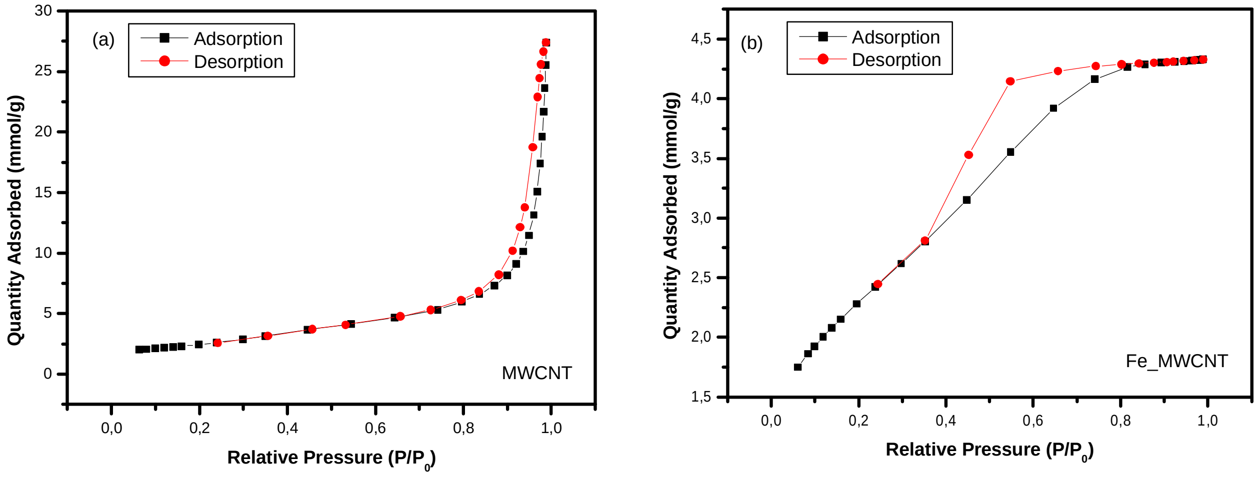


Fig. 2. The adsorption-desorption isotherms of MWCNT (a) and Fe_MWCNT (b)

Table 1

Textural characteristics of the porous adsorbents by N_2 adsorption at 77 K

Adsorbents	BET surface area, m^2/g	Langmuir surface area, m^2/g	Total pore volume, cm^3/g	Micropore volume, cm^3/g	Average pore width, nm
MCM-41	913.50	1264.50	0.755	0.019	4.310
Na_Y_Zeo	766.61	1008.31	0.368	0.339	1.918
NH4_Y_Zeo	736.92	969.38	0.358	0.323	1.945
MWCNT	193.70	265.20	0.751	0.0017	15.514
Fe_MWCNT	180.80	259.80	0.516	0.0011	12.312

Nitrogen adsorption-desorption isotherms of MWCNT (a) and Fe_MWCNT (b) are shown in Fig. 2. The existence of hysteresis loop will be possibly due to the existence of mesoporous. The hysteresis loop of Fe-MWCNT shown in Fig. 2b is the form of type H2 loop according to IUPAC classification. It was found that adsorbents that give rise to H2 hysteresis are often disordered and the distribution of pore size and shape is not well defined. The hysteresis loop in the isotherm of Fe-MWCNT closes before reaching a relative pressure of 0.35 in the desorption process except when microporosity is present [19, 20]. Loading of Fe decreased the BET surface area and the pore volume on MWCNT. The BET surface area and total pore volume of Fe_MWCNT decreased by 6.7 and 31.3 %, respectively, compared to those of MWCNT. This effect is attributable to the porous filling with the iron oxide. Similar results were reported for adsorption hydrogen using a low-cost metal oxide activated carbon. Moradi [21] produced the low-cost activated carbon loaded with iron oxide (5-Fe-MAC), and reported that the total pore volume decreased by 0.09 cm^3/g by loading iron oxide. Ohno *et al*. [22] reported that the total pore volume of the activated carbon decreased by 0.2 cm^3/g by loading of Pd for the activated carbon loaded with palladium (AC2-Pd-1).

Fig. 3 shows N_2 adsorption-desorption isotherms for MCM-41. The rise in the N_2 adsorption by a high pressure (P/P_0) is because of multilayer adsorption on the mesoporous, macroporous and on the external surface [21]. Table 1 shows the textural properties of MCM-41 as shown by N_2 adsorption-desorption isotherms. The BET surface area, Langmuir surface area, total pore volume, micropore volume and average pore width of MCM-41 were 913.5 m^2/g, 1264 m^2/g, 0.755 cm^3/g, 0.019 cm^3/g, and 4.310 nm, respectively. As a comparison between the textural properties of the zeolite samples and MCM-41, MCM-41 showed the highest average pore width and the lowest micropore volume. The highest BET surface area among porous adsorbents was obtained as 913.5 m^2/g.

The pore structures of the adsorbents were calculated by the *t*-method analysis from the adsorption branch of the N_2 adsorption isotherms. According to the categorization of pores (IUPAC), three pore groups exist: micropore < 2 nm; 2 nm < mesopore < 50 nm; and macropore > 50 nm. The average pore size was estimated from N_2 adsorption isotherms. The total pore volume, micropore volume and average pore width of the porous adsorbents are summarized in Table 1.

As a comparison between the textural properties of the porous samples, MWCNT has an average pore size of 15.514 nm, which is approximately the same as 1.918, 1.945 and 4.310 nm for Na_Y_Zeo, NH4_Y_Zeo and MCM-41, respectively. MWCNT showed the highest avarage pore width and the lowest micropore volume except for Fe_MWCNT composite sample.

SEM was used to observe the surface physical morphology of the Fe_MWCNT. SEM image of the Fe_MWCNT is shown in Fig. 4.

The EDX measurement is usually used to confirm the percentage, the atomic ratio of components on the surface of the composites [23]. EDX spectrum of the Fe_MWCNT is shown in Fig. 5. The Fe content of Fe_MWCNT as measured by EDX is 19.2 wt %, which is rather high for a composite.

3.2. Hydrogen Adsorption

Hydrogen adsorption isotherms were obtained on the porous adsorbents at the atmospheric pressure and 77 K. The amounts of hydrogen adsorbed were determined using the values of the adsorbed volumes at 0.99 P/P_0 and ideal gas equation. The adsorption data were fitted in Langmuir, Freundlich, Temkin and Harkins-Jura isotherms. Adsorption isotherms describe the interaction between hydrogen and the porous adsorbents [17]. The parameters obtained from the four isotherm models were represented in Table 2. It can be seen that the values of linear regression coefficient (R^2) are situated within the range of 0.993–0.999, demonstrating that the experimental data fitted well with the Freundlich isotherm equation. Moreover, it was reported that the $1/n$ value (Freundlich isotherm constant) can be used to calculate the adsorption capacity and intensity of the reaction and to explore the favourability of a sorption process. The $1/n$ value indicates the type of sorption process to be irreversible ($1/n = 0$), favorable ($0 < 1/n < 1$) and unfavorable ($1/n > 1$) [24]. For the adsorption of hydrogen on the porous adsorbents, it can be seen (Table 2) that the values of $1/n$ are situated in the range of 0–1, demonstrating that it is favorable for the hydrogen adsorption process. These results prove that the hydrogen adsorption on the porous adsorbents followed the Freundlich isotherm model and such adsorption mainly occurred on the heterogeneous surface of the porous adsorbents used in this study.

As shown in Table 2, it can be found that the values of R^2 are located in the range of 0.907–0.996, suggesting that these experimental data fitted well with the Langmuir isotherm model. The correlation coefficient of NH4_Y_Zeo was found to be higher than that of other porous adsorbents. Langmuir adsorption capacities, Q_0, were found to be 0.0103, 0.0235, 0.0201, 0.0074 and 0.0312 g/g for MCM-41, Na_Y_Zeo, NH4_Y_Zeo, MWCNT and Fe-MWCNT adsorbents, respectively. The highest value of Q_0 was found as 0.0313 g/g for the Fe-MWCNT composite sample. This finding indicates that the hydrogen adsorption on the porous adsorbent samples follows the Langmuir monolayer adsorption. As summarized in Table 2, it can be observed that the values of R^2 are in the range of 0.989–0.996, revealing that the experimental data fitted well with the Temkin isotherm model. The adsorption energy, b, is positive for all the porous adsorbents. The values obtained were 367.72, 122.81, 189.3, 466.31 and 100.4 kJ/mol for MCM-41, Na_Y_Zeo, NH4_Y_Zeo, MWCNT and Fe-MWCNT adsorbents, respectively. They indicate that the hydrogen adsorption reaction on the porous adsorbents is exothermic [25].

It can be noted from Table 2 that the values of R^2 are located in the range of 0.888–0.979 for Harkins-Jura isotherm model, which indicate the worse fits to the hydrogen adsorption on the porous adsorbent samples. The R^2 value is closer to unity for Langmuir isotherm model than that for the Harkins-Jura isotherm models for hydrogen adsorption. These results reveal that the hydrogen adsorption on MCM-41, Na_Y_Zeo, NH4_Y_Zeo, MWCNT and Fe-MWCNT are against the rule of multilayer adsorption.

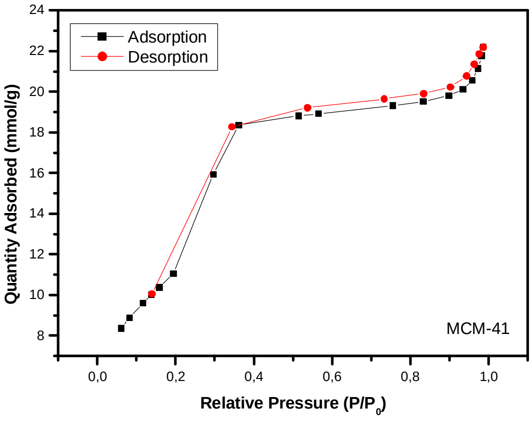


Fig. 3. The adsorption-desorption isotherms of MCM-41

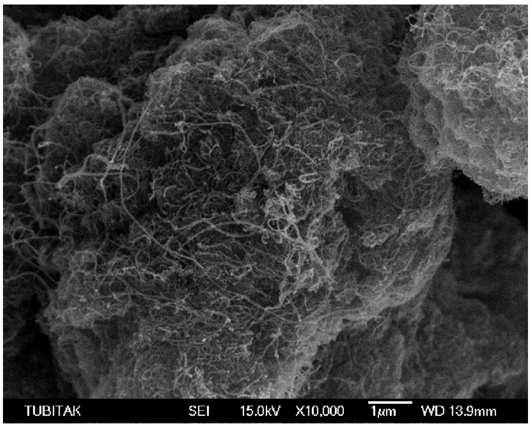
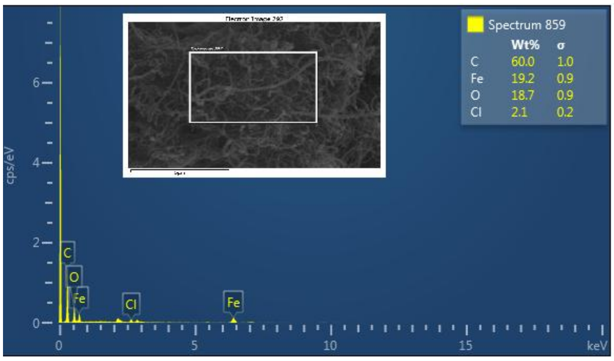


Fig. 4. Scanning electron micrograph of Fe_MWCNT



	Wt%	σ
C	60.0	1.0
Fe	19.2	0.9
O	18.7	0.9
Cl	2.1	0.2

Fig. 5. EDX spectrum of the area indicated in the inset: SEM of Fe_MWCNT composite; inset: Table presents the atomic ratio, percentage of the components in the composite

Table 2

Freundlich, Langmuir, Temkin and Harkins-Jura isotherm parameters for H_2 adsorption

Parameters	Adsorbents				
	MCM-41	Na_Y_Zeo	NH4_Y_Zeo	MWCNT	Fe-MWCNT
Freundlich isotherm model					
K_F(g/g)(cm^3/g)$^{1/n}$	9.22E-3	0.0192	0.0183	6.41E-3	0.0191
1/n	0.2458	0.4228	0.2403	0.2902	0.5876
R^2	0.999	0.995	0.999	0.993	0.993
Langmuir isotherm model					
Q_0 (g/g)	0.0103	0.0235	0.0201	0.0074	0.0312
K_L(cm^3/g)	7.2712	3.476	7.786	5.577	1.487
R^2	0.995	0.995	0.996	0.988	0.907
Temkin isotherm model					
A (cm^3/g)	180.24	33.37	200.34	95.39	16.66
b (kJ/mol)	367.72	122.81	189.3	466.31	100.4
R^2	0.989	0.995	0.996	0.973	0.925
Harkins-Jura isotherm model					
A	0.970	0.0764	0.9535	0.672	-0.151
B	9.53E-5	1.35E-4	3.76E-4	3.48E-5	6.34E-5
R^2	0.979	0.888	0.962	0.970	0.894

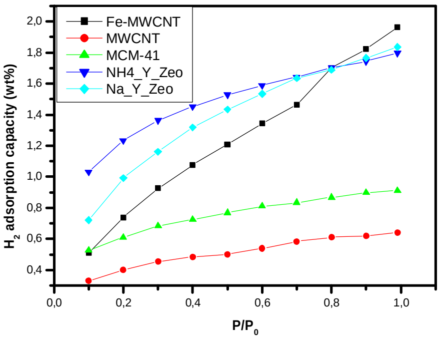


Fig. 6. Hydrogen adsorption capacities porous adsorbents at 77 K

Table 3

H_2 adsorption capacities (wt %) of porous adsorbents

Adsorbents	H_2 adsorption capacity, %
MCM-41	0.91
Na_Y_Zeo	1.83
NH4_Y_Zeo	1.79
MWCNT	0.64
Fe_MWCNT	1.96

Notice that, by comparing the values of R^2 of the examined four isotherm models, it can be concluded that the Freundlich, Langmuir and Temkin isotherm models gave much better fitting than the Harkins-Jura isotherm model.

Fig. 6 shows the hydrogen adsorption capacity (wt %) collected at 77 K and P/P_0 from 0 to 1 for MWCNT, Fe_MWCNT, MCM-41, Na_Y_Zeo and NH4_Y_Zeo.

The hydrogen adsorption capacities of these porous adsorbents are shown in Table 3. The hydrogen storage capacities of Na_Y_Zeo, NH4_Y_Zeo and MCM-41 were 1.84, 1.80 and 0.91 wt , respectively. Hydrogen adsorption capacity of the original sample (MWCNT) was 0.64 wt % and it was increased to 1.96 wt % for the iron loaded sample (Fe_MWCNT). This corresponds to 3.06 times increase in the hydrogen sorption capacity. The highest hydrogen storage capacity of 1.96 wt % was achieved with Fe_MWCNT. It could be confirmed that loading of iron on the carbon surface plays an important role in determining the porous structure and amount of hydrogen adsorbed. A similar phenomenon was reported by Moradi *et al.* [21]. Certain Na_Y_Zeo and NH4_Y_Zeo showed higher hydrogen adsorption capacities than MCM-41, depending on their porous structures. Then, we looked at the relationship between hydrogen adsorption capacity and porosity. The relationship between the hydrogen adsorption capacities and the BET surface area for these three porous adsorbents did not show a proportional relation. The hydrogen adsorption capacities increased with increasing micropore volume size. These results indicate that the hydrogen storage ability at 77 K is affected by the micropore volume size. A similar phenomenon was reported by Akasaka *et al.* [10] and Minoda *et al.* [26].

4. Conclusions

We have investigated the adsorption properties for hydrogen on five typical adsorbent materials with different texture, surface area and porosity. The H_2 adsorption isotherms are modeled using four isotherm models. The adsorption of H_2 on these porous adsorbent samples followed the Freundlich, Langmuir and Temkin isotherm models. The adsorption heat calculated from Temkin isotherm model was restricted within 100–466 kJ/mol. The micropore volume was more important than a total pore volume for the hydrogen storage when the specific surface area and pore volume were large enough. The amount of hydrogen adsorbed on Fe_MWCNT was larger than the amount adsorbed on the unmodified MWCNT sample. The maximum hydrogen storage of 1.96 wt % at 77 K was achieved by Fe_MWCNT. Microporous Na_Y_Zeo and NH4_Y_Zeo showed higher hydrogen adsorption capacities than mesoporous MCM-41. The hydrogen adsorption properties of these porous adsorbents may be further enhanced by different metal doping, thus paving the way for further study.

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ДОСЛІДЖЕННЯ ІЗОТЕРМ ФРЕЙНДЛІХА, ЛЕНГМЮРА, ТЕМКІНА ТА ГАРКІНСА-ЮРИ ПРИ АДСОРБЦІЇ H_2 НА ПОРИСТИХ АДСОРБЕНТАХ

Анотація. *Вивчено ізотерми адсорбції та десорбції водню для багатошарової карбонової нанотрубки (MWCNT), багатошарової карбонової нанотрубки модифікованої залізом (Fe_MWCNT), двох цеолітів (Na_Y_Zeo і NH4-Y_Zeo) та MCM-41 за температури 77 К і атмосферного тиску. Адсорбційні характеристики оцінено декількома ізотермічними рівняннями, такими як моделі Ленгмюра, Фрейндліха, Темкіна та Гаркінса-Юри. Визначено, що ізотерма Фрейндліха найбільш повно описує процес, оскільки має найвищу кореляцію. Встановлено, що масова кількість адсорбованого водню залежить від об'єму мікропори зразка, крім MWCNT та Fe_MWCNT. Характеристику пористих зразків визначено за допомогою скануючої електронної мікроскопії та ізотерм адсорбції* N_2*.Визначено, що максимальний запас водню 1,96 мас. % досягається за 77 К при використанні Fe_MWCNT. Мікропористий Na_Y_Zeo та NH4_Y_Zeo виявляють більшу адсорбційну здатність водню, ніж мезопористий MCM-41. Показана можливість покращення адсорбційні властивостей цих пористих адсорбентів щодо водню внаслідок введення інших металів.*

Ключові слова: *адсорбційна здатність водню, багатошарова карбонова нанотрубка, цеоліт, MCM-41, композит залізо/багатошарова карбонова нанотрубка.*