

Studies of Reversible Hydrogen Binding in Nano-Sized Materials

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Abstract – Experimental review of materials suitable for reversible hydrogen binding in nanoporous and nanosized structures of materials, based on natural zeolite (clinoptilolite) and graphene (exfoliated electrochemically from raw graphite), were analyzed. Characterization of materials with SEM, XRD, EDS and Raman spectroscopy methods and aspects of synthesis of a nanostructured zeolite and a few-layer graphite material was done in this work. It was established from gas analyzer results that hydrogen mass fraction in natural zeolite ranged from 1.1 % to 1.4 %, but in the few-layer graphite material – from 0.39 % to 0.46 %.

Keywords – Hydrogen, storage, graphene, zeolite, graphite.

I. INTRODUCTION

To facilitate the transition from fossil fuels to technologies based on clean and renewable resources, problems concerning the energy storage must be solved. Wind and sun are the most popular renewable energy sources, but they are available occasionally or periodically, that is, not always when consumption requires [1]. Compared to electricity, hydrogen is an ideal energy carrier, because it can be stored almost without losses. In transport sector hydrogen fuel cell technologies are currently being commercialized (*Toyota, Hyundai* from 2015), hydrogen storage in high pressure (700 bar) tanks is used [2]. The technology and economical means to make carbon-composite high pressure tanks capable of providing travel distances up to 700 km – are well understood and the costs are reasonable. World's largest hydrogen association – International Partnership of Hydrogen Energy (IPHE) has gathered information about an impressive number of already realized hydrogen demonstration projects (Table I).

In the near future the demand for inexpensive and “green” hydrogen will increase, especially for transportation purposes. High pressure composite vessels can be charged only using powerful multi-stage compressors, which is profitable at filling stations, but not in the private sector and small farms. Creating a capacious, not too heavy and large containers with porous, solid materials containing stored hydrogen could solve this problem, because these materials can be recharged at low pressures, for example, at which typical electrolyzers work (up to 30 bar). Regarding the application of this technology for vehicles, the materials with high volumetric and gravimetric densities of hydrogen storage are required [4]. United States Department of Energy (DOE) is focusing primarily on the research and development of on-board vehicular hydrogen storage systems that will allow for a driving range of greater than 300 miles (480 km) while meeting packaging, cost,

safety, and performance requirements to be competitive with currently used vehicles. The targets set by DOE [4] for these solid materials to reach in 2015 are based on application requirements (not on current hydrogen storage technology capabilities) are 6 % of mass fraction for system gravimetric density and 45 g/L for system volumetric density.

TABLE I

SUMMARY OF IPHE DEMONSTRATION PROJECTS [3]

IPHE Partner Country	A	B	C	D	F
Australia	0	0	0	0	2
Canada	16	5	12	34	6
China	3	20	-	6	-
Germany	7	28	13	20	55
Japan	12	45	8	5	2232
Korea	5	40	0	4	240
New Zealand	0	0	0	0	2
Norway	2	0	15	0	1
United States	58	92	25	34	~ 2000
Total	103	230	73	103	~ 4500

A – number of hydrogen refilling stations; B – fuel cell vehicles;
 C – ICE compressed hydrogen vehicles; D – hydrogen buses;
 F – stationary fuel cells.

Optimum thermodynamic and kinetic windows are needed for hydrogen absorption and desorption in solids at reasonable temperatures and pressures [5]. The pores in zeolites with dimensions of 4 Å – 7 Å are suitable to store hydrogen molecules with kinetic diameter of 2.89 Å) up to 1.74 % weight fraction, as it is shown, at low temperature (– 196 °C) and H₂ pressure (15 bar) [6]. Recently, it was announced that at low temperatures (– 196 °C) zeolite templated carbons show exceptional gravimetric hydrogen uptake – weight fraction 7.3 % at 20 bar [7], which is the highest recorded for carbon materials. Only some of the research is devoted to hydrogen storage capability in zeolite-type structures at higher temperatures. Weitkamp *et al.* in 1993 [8] reported that zeolites containing sodalite cages in their structure show hydrogen storage capacity 9.2 cm³/g, if loaded at 573 K and at 10.0 MPa.

Using theoretical considerations the authors [9] suggested that a multilayer graphene storage system has good reversibility for chemisorbed hydrogen on corrugated graphene sheets (weight fraction up to 8 %). The authors [10] proposed to enhance the hydrogen chemisorption by doping the graphitic material with Pd or Pt catalyst on which

hydrogen molecules disassociate and the atomic hydrogen then can easier fill the pores of the material. The drift or the spillover of atomic hydrogen onto carbon surface was investigated ten years ago, when Lueking *et al.* [11] detected that the hydrogen uptake as mass fraction in Pd-doped carbon nanotubes (SWNT) was up to 0.1 % at 1 bar and 25 °C. A third possibility also exists – to adjust hydrogen adsorption enthalpy by introducing nano-grains of hydride-forming material between the sheets of graphene in stacks, for example, lithium or magnesium [12].

Our research [13] shows that it is possible to reach hydrogen adsorption capacity of 6.2 % mass fraction for Mg-ion exchanged zeolite (clinoptilolite) using a specific technique – the loading of material with hydrogen at high temperature and following it with cooling down to room temperature. The mechanism of hydrogen encapsulation in the pores of zeolite is proposed to explain such a high hydrogen uptake.

In this paper we used different techniques to test previous results and to characterize zeolite and few-layer graphite materials for hydrogen storage capability at room temperature.

II. EXPERIMENTAL PART

Samples were prepared by ion exchange with MgCl₂ (1 mol/l) and washing in deionized water. Zeolite – clinoptilolite sample was characterized using XRD as natural clinoptilolite with some ferrierite impurities [14] and furtherer investigated by EDS to determine more precise Si/Al ratio (Table II and Table III). Due to different hydrogen loading pressure (5 bar) and exposure to air, varying results were expected.

TABLE II

NATURAL CLINOPTILOLITE – EDS SPECTRA WITH SI/AL RATIO 10.319

Element	Series	Norm. at., %	Error, %
Aluminium	K-series	2.618932574	0.218606073
Silicon	K-series	28.80796765	1.889704057
Oxygen	K-series	65.70334441	6.983858719
Potassium	K-series	1.011897176	0.107846702
Sodium	K-series	0.352886243	0.065595332
Iron	K-series	0.537640653	0.106374724
Carbon	K-series	0.313475372	0.391040007
Magnesium	K-series	0.332134442	0.058916271
Calcium	K-series	0.321721484	0.060207677

To obtain a few-layer graphite (FLG), the electrochemical exfoliation was used, using graphite industrial waste rod as the working electrode. Setup parameters were as follows: 5:5 (s) pulse sequence, ± 10 V amplitude. Electrolyte with varying concentrations (0.25 mol/l, 0.5 mol/l or 1 mol/l) and counter electrode (Cu plate or graphite) were used to find optimal parameters for exfoliation process. An important step is purification – FLG material is the lightest material and can be partially separated using centrifugation or sedimentation. From our experience, sedimentation was more useful. To

obtain material with higher quality, samples were exposed to Ar/H₂ gas flow at 300 °C for 3 h, thus guarantying a higher degree of reduction [14,15].

The initial samples were characterized by X-ray diffraction (XRD) using *Bruker D8* model, scanning electron microscopy (SEM) using *Hitachi S-400N* model, the elemental composition of samples was determined using energy dispersive spectroscopy (EDS).

TABLE III

SYNTHESIZED CLINOPTILOLITE SAMPLE EDS WITH SI/AL RATIO 8.372

Element	Series	Norm. at., %	Error, %
Oxygen	K-series	62.03790834	5.859888236
Silicon	K-series	29.63537623	1.882520741
Aluminium	K-series	3.541808206	0.265028459
Potassium	K-series	2.728274732	0.202826426
Sodium	K-series	0.372240588	0.056766053
Magnesium	K-series	0.087593441	0.033077223
Carbon	K-series	1.596798465	0.344494868

III. METHODS

Two significantly different methods to measure hydrogen uptake were used. The first was Sievert type volumetric method using *PCT – Pro 2000 (SETARAM Instrumentation)* – a fairly slow experiment to determine kinetics and PCT graphs. The second was gas analysis of samples using *HORIBA EMGA – 830AC Hydrogen Oxygen Nitrogen* gas analyzer (experiment limited till 100 s due to sensor saturation).

Using this method the loaded sample was burned in He plasma and compared to hydrogen free samples, which had been degassed. The percentage of gas content was recalculated from the mass of sample.

IV. RESULTS

The partial sorption level of clinoptilolite determined from volumetric measurements was ~ 0.6 % mass fraction, also – after hydrogenation, natural clinoptilolite sample had changed color to more grey tone both in Sievert type device and in external hydrogenation apparatus for gas analysis measurements.

XRD studies show that zeolite indeed had the heulandite type structure as determined by XRF as clinoptilolite; yet the FLG's were recognized as graphite (Fig. 8).

Gas analyzer data shows hydrogen content in natural zeolite from 1.1 % to 1.4 % of mass fraction (Fig. 1, Fig. 2) depending on the loading method (PCT apparatus or the external hydrogenation) for gas analysis measurements.

SEM images of FLG samples (Fig. 3, Fig. 4) show opened few-layer graphite/graphene structures, with higher surface area than it would be, if stacked together (determined by BET method 0.43 m²/g for Mg FLG and 12.4 m²/g for reduced FLG). The calculated thickness from SEM images (Fig. 3, Fig. 4) was around 37 and 54 nm as multilayer material and up to 200 nm in thickness, containing voids between graphene sheets sized from

couple of nm and up to several μm . Using SEM images of graphene/graphite type structures, it was determined that chemical exfoliation produces open FLG structures. Reducing process broke up the stacks of material, leaving open structures deep in the particles, but also exposed single sheets of FLG.

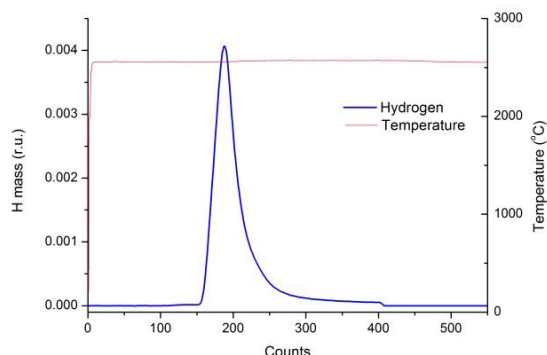


Fig. 1. Gas analyzer data for zeolite after hydrogenation in a Sievert type device *PCT-PRO 2000*.

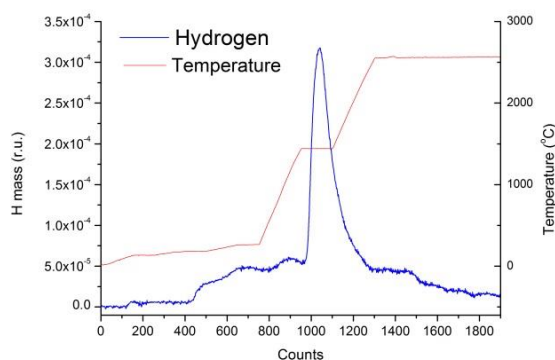


Fig. 2. Gas analyzer data for zeolite after hydrogenation by an external hydrogenation device.



Fig. 3. SEM image of FLG particles.



Fig. 4. SEM image of FLG particle structures.

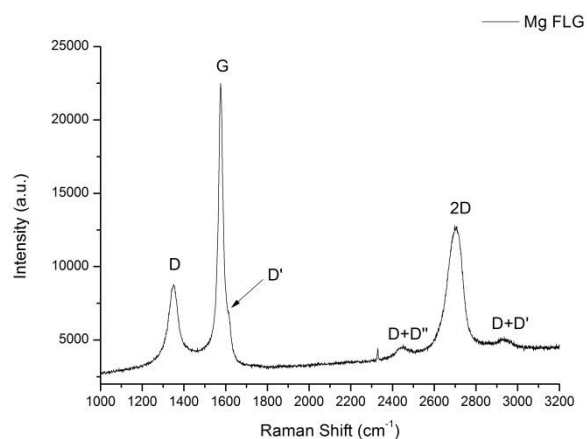


Fig. 5. Raman spectra of Mg FLG structures.

XRD (Fig. 6) showed graphitic structure with additional peaks of an impurity from production.

EDS XRF analysis showed the presence of elements with larger atomic number than Na. The spectra showed mostly Fe, Si and some Ti impurities in the graphite substrate material. The EDS analysis for FLG shows sulfur impurities and some chlorine, as well as Al, Si, Fe, and Ti (Table IV).

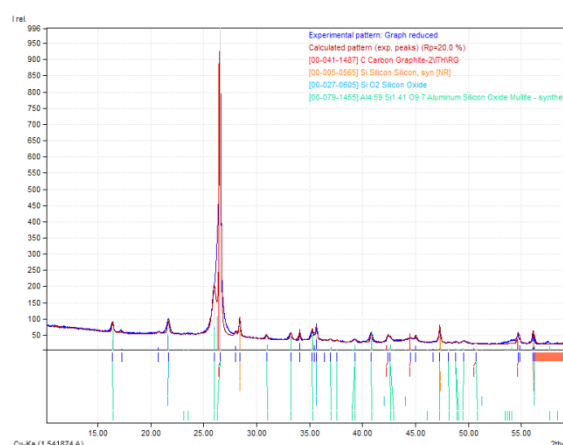


Fig. 6. XRD pattern of FLG powder.

Raman scattering was used to identify the presence of graphene (as it can be seen in Fig. 5) for obtained multistacks of graphene sheets. Micro-Raman measurements were conducted using a *Renishaw inVia* spectrometer equipped with an argon laser with wave length 514.5 nm. Raman scattering

spectra were acquired at room temperature through a microscope objective with 20× magnification. The spectral signal was dispersed using grating that had 2400 grooves in 1 mm onto a Peltier cooled (−60 °C) CCD detector. The laser excitation power was 10 mW. G peak showed the presence of graphitic structures. D peak showed the amount of defects in graphene planes. Intensity ratio of 2D/G peaks indicated the number of layers in graphitic materials. The 2D peak had lower intensity than the G peak – thus indicating that the material consisted of more than one layer. D' peak is usually produced by intravalent scattering or hole scattering in the material. D' + D and D'' + D indicated edge defects – alignment between planes was not consistent and zigzag / armchair edge mix-up was present in FLG material [17].

TABLE IV
Mg FLG EDS DATA

Element	Series	Norm. at. %	Error. %
Oxygen	K-series	28.31609752	10.65808247
Carbon	K-series	70.18533945	21.68381901
Sulfur	K-series	0.684214243	0.084989106
Silicon	K-series	0.129782837	0.038174867
Aluminium	K-series	0.058680308	0.032189277
Sodium	K-series	0.307062192	0.060594815
Potassium	K-series	0.091614128	0.035981395
Chlorine	K-series	0.227209318	0.047522642

Preliminary experiments were done to test hydrogen absorption capability in graphene sheet stacks, using thermal absorption/desorption methods. Two different samples were collected from both exfoliation and plasma methods – the light fraction of material, which floated on the top of solution, and the heavy fraction, which sank at the bottom of the solution.

A study of hydrogenated Mg FLG samples was done using Horiba EMGA – 830AC gas analyzer. During the burning of samples at different rates reaching 6 kW and around 3000 °C, the absolute hydrogen mass fraction in samples was determined to be 0.39 % – 0.46 %.

V. DISCUSSION

Synthesized materials had acceptable quality, showed the needed phases for zeolites and the desired pike in Raman spectra (for few-layer graphite powder), see Fig. 5. Better purifying methods could allow the use of waste material as a reliable resource for synthesis of few-layer graphite powder.

Clinoptilolite shows a promising tendency for higher hydrogen storage capacity, but further studies are needed to repeat the obtained results. Especially for gas analyzer experiments that show the exact temperatures in which most desorption occurs, it would be best to use this technique step by step – not applying the max voltage and destroying the mineral.

Hydrogen can interact with graphene in FLG structures by physisorption and chemisorption; another possibility is to

exploit intercalation of hydrogen between FLG sheet stacks (distance between the sheets is important) [18].

As reported above, graphene sheets that were not doped showed hydrogen storage possibilities around 0.55 % mass fraction. Intercalation of FLG with magnesium or lithium ions would be necessary to enhance the amount of bound hydrogen in the material. Reached results show more precise information about processes which are happening during hydrogen sorption in high surface area materials.

IV. CONCLUSION

In our work an artificial zeolite, which corresponds to clinoptilolite regarding XRD analysis, was successfully synthesized. Based on XRD data, the tested samples of natural zeolite were clinoptilolite with crystalline ferrierite and quartz impurities. From volumetric measurements it was determined that hydrogen uptake in the natural zeolite at room temperature was around ~ 0.6 % mass fraction and from gas analyzer data it was determined that the mass fraction was from 1.1 % to 1.4 % .

Few-layer graphite material with thickness of sheet stacks around 37 nm and 54 nm was obtained from raw graphite rod using simple electrochemical exfoliation method. Measured Raman spectra proved the existence of graphene sheets in the exfoliated few-layer graphite. The gas analyzer was applied to measure the amount of hydrogen in the hydrogenated exfoliated few-layer graphite and the values from 0.39 % to 0.46 % mass fraction were obtained.

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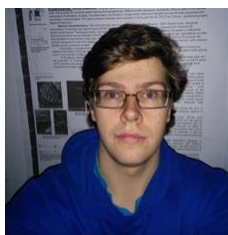
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Pēteris Lesničenoks, Jānis Zemītis, Jānis Kleperis, Georgijs Čikvaidze, Reinis Ignatāns. Atgriezeniskas ūdeņraža saistīšanās spējas pētījumi nanomateriālos.

Salīdzinot elektroenerģijas uzglabāšanas veidus, ūdeņradis ir daudz efektīvāks enerģijas uzkrāšanas līdzeklis nekā baterijas vai kondensatori, jo to iespējams uzglabāt gandrīz bez zudumiem. Šī brīža tehnoloģijas ļauj uzglabāt ūdeņradi tītajos oglekļa šķiedru kompozīta balonos augstā spiedienā, taču zema spiediena tehnoloģijas ir nozīmīgs solis drošības un nākotnes pielietojumu, kā arī tehnoloģijas vienkāršošanas virzienā. Darba mērķis bija atrast efektīvus ūdeņraža sorbcijas materiālus, kā arī modificēt pieejamos reciklējamus tehniskos metalurģijas atkritumus, lai

tos būtu iespējams izmantot ūdeņraža enerģētikas jomā. Darbā dots īss pārskats par materiāliem un to piemērotību atgriezeniskai ūdeņraža saistīšanai mezoporainu materiālu nanoporās un uz virsmas, kā arī eksperimentāli iegūti rezultāti par dabīgā ceolīta (klinoptilolīta) un vairākslāņu grafiņa (iegūts elektroķīmiskas atslāņošanas ceļā no grafiņa) ūdeņraža adsorbcijas spēju. Gan ceolītu, gan vairākslāņu grafiņa materiālu morfoloģija, sastāvs un struktūra raksturoti izmantojot skenējošo elektronu mikroskopiju, rentgenstruktūranalīzi Furjē transformāciju infrasarkanā spektroskopiju, enerģijas dispersijas rentgenstaru spektroskopiju (EDS) un Ramana spektroskopiju. Pēc termiskas apstrādes ūdeņraža atmosfērā (5 bar) adsorbcijas/desorbcijas iekārtā *PCT-Pro2000* un/vai speciāli izveidotā neliela tilpuma cilindriskā kamerā, materiālos saistītā ūdeņraža daudzums noteikts izmantojot gāzu analizatoru *Horiba EMGA – 830 AC*, kurā paraugu sadedzināja hēlija plazmā. Ceolīta paraugu ūdeņraža sorbcijas spēja ir robežās no 0.6 % līdz 1.4 % (masas daļa), bet vairākslāņu grafiņa materiāliem tā ir robežās no 0.39 % līdz 0.46 %.

Strādājot ar elektroķīmisko vairākslāņu grafiņa iegūšanas metodi, noskaidrota optimālā elektrolīta molaritāte ātrai vairākslāņu grafiņa materiāla iegūšanai – 1 mol/l H_2SO_4 bez papildus piedevām. Tālākos pētījumos tiks skaidrota vairākslāņu grafiņa struktūrā interkalētu dažādu metālu jonu (Mg, Li, Pd) ietekme uz ūdeņraža sorbcijas/desorbcijas spēju materiālā, ar mērķi atrast labāko materiālu ūdeņraža uzglabāšanai.

Петерис Лесниченокс, Янис Земитис, Янис Клеперис, Георгий Чикваидзе, Реннис Игнатанс. Исследования способности обратимого связывания водорода в наноразмерных материалах.

По сравнению с современными способами хранения электроэнергии (например, батареями или конденсаторами), водород более эффективен, т.к. его можно хранить почти без потерь. В наше время для хранения водорода используют специальные композитные баллоны из углеродного волокна под высоким давлением, поэтому технологии хранения при низком давлении – это важный шаг на пути к безопасному использованию водорода в будущем и упрощению этого метода хранения. Цель работы состоит в том, чтобы найти наиболее эффективные материалы для сорбции водорода, а также успешно модифицировать доступные перерабатываемые технические отходы металлургии таким образом, чтобы они могли бы быть использованы в отрасли водородной энергетики.

В работе представлен краткий обзор исследований материалов и их способностей обратимого связывания водорода в порах и на поверхности наноматериалов. Обзор основан на исследованиях природного цеолита (клиноптилолита) и графена, полученного из графита методом электрохимической эксфолиации. Свойства порошковых материалов (цеолиты и образец из нескольких слоев графита (FLG)) были изучены при помощи СЭМ, РДА, ИК-Фурье, ЭДС и РАМАН. Согласно результатам, полученным при помощи газового анализатора *Horiba EMGA – 830 AC*, в котором образец сжигается в плазме гелия, способность поглощать водород у цеолита находится в интервале от 0,6 – 1,4 массовых %, а для образцов из нескольких слоев графита от 0,39 – 0,46 массовых %. Образцы насыщались водородом двумя методами: при помощи установки для определения сорбции/десорбции, изменением объема *PCT – Pro 2000*, а так же при использовании специально изготовленной цилиндрической камеры, которую можно нагревать и создавать внутри вакуум. В обоих случаях эксперимент проводился при давлении водорода 5 бар.

При использовании электрохимического метода для быстрого получения порошкового образца из нескольких слоев графита (FLG) была выявлена оптимальная молярность электролита – 1 моль / л (H_2SO_4). Дальнейшие исследования будут сосредоточены на изучении влияния интеркаляции ионов металлов (Mg, Li, Pd) в структуры из нескольких слоев графита на сорбцию/десорбцию водорода в материале с целью найти наиболее эффективный материал для хранения водорода.