Effect of activated carbons derived from apricot stones or polyolefin wax on hydrogen sorption properties of MgH₂

E. Grigorova[1*], M. Khristov[1], I. Stoycheva[2], B. Tsyntsarski[2], D. Nihtianova[1,3], P. Markov[1]

[1] *Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences, bl.11, Acad. G. Bonchev str., 1113 Sofia, Bulgaria*

[2]*Institute of Organic Chemistry with Centre of Phytochemistry, Bulgarian Academy of Sciences, bl. 9, Acad. G. Bonchev str., 1113 Sofia, Bulgaria*

[3]*Institute of Mineralogy and Crystallography, Bulgarian Academy of Sciences, Acad. G. Bonchev Str., bl. 107, 1113 Sofia, Bulgaria*

The hydriding and dehydriding properties of 95 wt.% MgH₂ - 5 wt. % C composites, synthesized by ball milling under argon with two different carbon additives (activated carbon derived from apricot stones - AS and from polyolefin wax - POW), were studied. Both samples showed no significant difference in the hydrogen absorption kinetics and reached absorption capacity of 5.8 wt.% for 95 wt.% MgH₂b - 5 wt. % AS and 5.4 wt . % for 95 wt.% MgH₂ - 5 wt. % POW at 573K and 1MPa after 60 min of hydriding. Faster desorption rate at 623 K and 0.15 MPa was demonstrated by the composite containing activated carbon derived from apricot stones. Morphology and phase composition of the composites were investigated by TEM and XRD.

INTRODUCTION

Important factor in the use of hydrogen as a clean fuel is the development of safe and effective materials for its storage. One of the most promising hydrogen storage materials is magnesium, mostly because of its high theoretical hydrogen storage capacity (7.6 wt.%), abundance and low cost. Its practical application, however, is hindered by the slow hydrogen sorption kinetics and high hydriding/dehydriding temperatures. Improvement of the hydrogen storage properties of magnesium is achieved by the method of high-energy ball milling using different types of additives which act as a catalyst during the processes of hydriding/ dehydriding. According to many authors, carbon containing additives improve the hydrogen sorption properties of magnesium [1-19]. It has been proven that carbon protects magnesium from oxidation and acts as process controlling agent during ball milling due to its anti-stacking properties [1, 4, 12-14, 19]. In addition, carbon containing additives reduce hydrogen storage temperature and improve the hydrogen sorption kinetics of magnesium based materials [2, 4, 5, 12, 13, 19].

Various carbon containing additives as graphite [1-6, 8-15, 17, 18], activated carbon [1, 6, 20-22], carbon nanomaterial [5, 16], nano diamonds [16],

diamonds [17, 18] carbon from anthracite coal [19], carbon fibers and nanofibers [1, 18], nanotubes [1, 6, 7, 14,17], carbon black [6, 14-16], amorphous carbon soot [16] and amorphous carbon [15, 17] are used as additives to magnesium and MgH₂.

All carbon materials used by Lillo-Rodenas *et al.* [1] decrease the decomposition temperature of MgH₂ and the best results are demonstrated by the mixtures with carbon nanofibres and multi-walled carbon nanotubes. Huot *et al.* [2] reported that graphite additive significantly intensifies the reactive milling synthesis of MgH₂ and improves the hydrogen desorption kinetics. Improvement of hydrogen sorption properties in ball milled MgH₂, using expanded natural graphite and compression in order to form compacted disks, has been observed by Chaise *et al.* [3]. Wu *et al.* [6, 7] found that considerable improvement of the hydrogen storage properties of Mg can be achieved upon mechanical milling of MgH₂ with carbon additives compared with non-carbon materials. Purified single-walled carbon nanotubes (SWNTs) show the most pronounced catalytic effect on the hydrogen storage properties of Mg. The hydrogen absorption capacity and hydriding kinetics of the composites are dependent on the added amount of SWNTs, as well as on the milling time [6, 7]. Several papers by Imamura *et al*. and Montone *et al*. have presented results for the Mg-graphite nanocomposites, prepared by ball milling with different organic additives [8–11]. They suggested the possibility of the formation of new sites for hydrogen storage other

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* To whom all correspondence should be sent:
E-mail egeorg@svr.igic.bas.bg

than those due to the magnesium, able to reversibly store hydrogen. Materials synthesized by reactive mechanical milling of magnesium powder, a mixture of Mg-graphite flakes and MgH_2- graphite flakes for different duration, were investigated by Fuster *et al.* [12, 13]. The composite prepared from a mixture of magnesium powder and graphite shows better sorption properties in comparison with a material where graphite is added to MgH_2 already synthesized and mixed by milling. When the additive is homogeneously distributed within the bulk, graphite catalytic activity is more pronounced.

Among the composites with different carbon additives, magnesium with carbon black revealed the best hydriding properties - low temperature of hydriding and relatively fast hydriding kinetics. Spassov *et al.* [16] suggested that the improved sorption properties of this composite are due to the finer particles and the better contact between carbon and MgH_2/Mg particles compared to the other two materials studied. Hydrogen sorption by Mg-based composites with addition of different carbon additives during reactive ball milling was investigated by Rud *et al.* [17]. The use of these types of additives led to a significant increase in hydrogen sorption rate. The formation of MgH_2 during reactive ball milling has been faster for the carbon added materials than for the pure magnesium. Awad *et al.* [18] investigated the desorption reaction of MgH_2 catalyzed by different forms of carbon under microwave irradiation. Rising carbon content, microwave power and milling time resulted in enhancement of dehydrogenation rate.

Despite the numerous results published on addition of carbon to magnesium, the mechanism of carbon additive influence on hydrogen absorption/ desorption properties of magnesium is not very clear. The effect of graphite and activated carbons derived from low-cost agricultural by-products on the hydrogen sorption properties of Mg and Mg_2Ni was investigated previously by us and results were published in several papers [4, 20-23]. It was established that the presence of carbon containing additives in the Mg-based composites has positive influence on their hydrogen sorption properties, leading to an enhanced hydriding rate and higher absorption capacity compared to pure magnesium. The aim of the present work was to continue the investigation of the role of carbon containing additives derived from non-expensive and waste products on the hydrogen sorption kinetics of magnesium. Activated carbons derived from apricot stones and from polyolefin wax (a waste product from polyethylene production) were used as additives to MgH_2 and the hydrogen sorption properties of the composites obtained by ball milling under argon were studied.

EXPERIMENTAL

One of the activated carbons was prepared from apricot stones by steam pyrolysis. Detailed description of the preparation procedure of activated carbon derived from apricot stones can be found in [24]. As for the other activated carbon, it was synthesized from polyolefin wax, a waste product of low-pressure polyethylene production from Burgas petroleum plant, Bulgaria. The polyolefin wax sample was heated to 383 K until melting. Concentrated sulfuric acid was added dropwise under continuous stirring, and the temperature was increased up to 433 K. The obtained solid product was washed with water, dried at 423 K and carbonized at 873 K. POW carbonizate was subjected to water steam activation at 1073 K for 1 h to obtain activated carbon. Textural characterization was carried out by measuring the N_2 adsorption isotherms at 77 K using Quantachrome NovaWin2 porosimeter. The isotherms were used to calculate the specific surface area S_{BET} and the total pore volume Vt. The pore size distribution, micropore and mesopore volumes were obtained by applying the DFT model to the N_2 adsorption data, assuming a slit-shaped pore geometry. The carbon samples were analyzed using the FTIR spectrometer Bruker IFS 113V with resolution of 1 cm^{-1} and 64 scans. The samples were mixed with potassium bromide (1:1000) and the mixture was pressed into pellets to be used in the analysis.

Composites of MgH_2 (98 % purity, purchased from Alfa Aesar) and 5 wt.% activated carbons, were obtained by ball milling in a planetary mono mill Fritsch Pulverisette 6. The milling was performed under Ar atmosphere using stainless steel balls (diameter 10 mm) and vial (volume ca. 75 cm^3), the balls to sample weight ratio being 10:1. The rotation speed was 200 rpm and the grinding duration 30 min. Messer GmbH argon and hydrogen gases with purity of 99.998% and 99.999%, respectively, were used in the experiments. Further in the article, the composites will be denoted as: MgH_2-AS: 95 wt.% MgH_2 - 5 wt.% activated carbon derived from apricot stones; MgH_2-POW: MgH_2 - 5 wt.% activated carbon derived from POW.

The phase composition of the starting compounds, the initial, hydrided and dehydrided composites was controlled with the use of a Bruker D8 Advance X-ray diffractometer (CuK$_\alpha$ radiation).

Additional characterization of the samples was performed by transmission electron microscopy (TEM). The TEM studies were performed on a JEOL

2100 instrument at an accelerating voltage of 200 kV. The specimens were prepared by grinding and dispersing in ethanol followed by an ultrasonic treatment for 6 min. The suspensions were dripped on standard holey carbon/Cu grids.

The hydrogen absorption-desorption properties of the composites were determined by volumetric or Sievert's type apparatus. Hydrogen absorption proceeded at temperatures of 573 K and 473 K and a pressure P = 1 MPa. Desorption was carried out at 573 K and 623 K and P = 0.15 MPa.

RESULTS AND DISCUSSION

The XRD patterns of the composites 95 wt.% MgH_2 - 5 wt.% AS and 95 wt.% MgH_2 - 5 wt.% POW after ball milling are shown in Fig. 1.
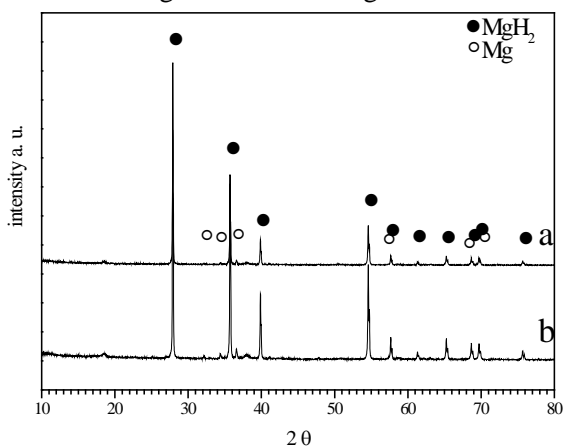

Fig. 1. X-ray diffraction pattern of the composites a) 95 wt. % MgH_2 - 5 wt. % activated carbon from apricot stones and b) 95 wt. % MgH_2 - 5 wt. % activated carbon from polyolefin wax, obtained by ball milling.

Stable β-MgH_2 was detected as the main phase. Small amount of magnesium and no MgO was detected. Probably, even after only 30 min of ball milling some part of MgH_2 was decomposed, because overpressure in the ball milling jar was observed when opened after ball milling. For such a short milling time, e.g. 30 min and at these milling parameters it is not unexpected that no metastable γ-MgH_2 was formed.

The hydrogen absorption curves at 573K and 1 MPa are presented in Fig. 2. Both composites showed very close and overlapping curves in the first 15 min of hydriding. Addition of activated carbon derived from apricot stones resulted in higher absorption capacity of 5.8 wt%. The decrease in the absorption temperature to 473 K led to a slower rate of hydriding and after 1 hour both samples reached an absorption capacity of about 1 wt.%. If we compare our previous result for the composite based on Mg and the same additive, *viz.* activated carbon derived from apricot stones [20, 21], it is obvious

that the 95 wt% Mg-5 wt% AS has slightly better hydrogen absorption kinetics and higher absorption capacity. Same effect on the hydrogen absorption properties was observed by Fuster *et al.* [13].

Obviously, the ball milling conditions of their experiments are very different from these used by us, but probably better contact with carbon containing additives can be achieved when the starting material is magnesium. With such short milling time, as applied in this study, no drastic difference in the rates of hydriding of the samples based on magnesium and magnesium hydride can be expected, as can be seen in Fig. 2.
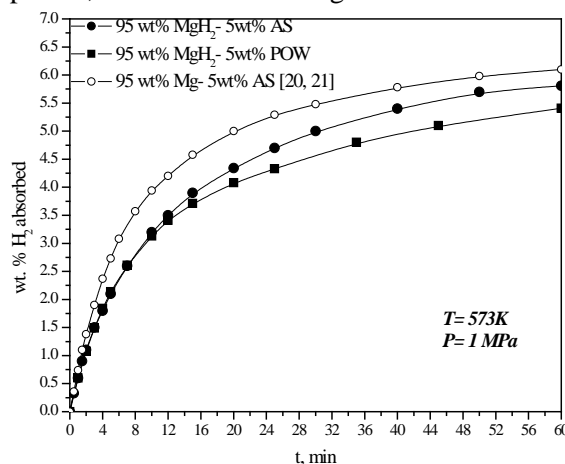

Fig. 2. Kinetic curves of hydrogen absorption by the composites 95 wt. % MgH_2 - 5 wt. % activated carbon from apricot stones and 95 wt. % MgH_2 - 5 wt. % activated carbon from polyolefin wax at P = 1 MPa and T= 573 K.
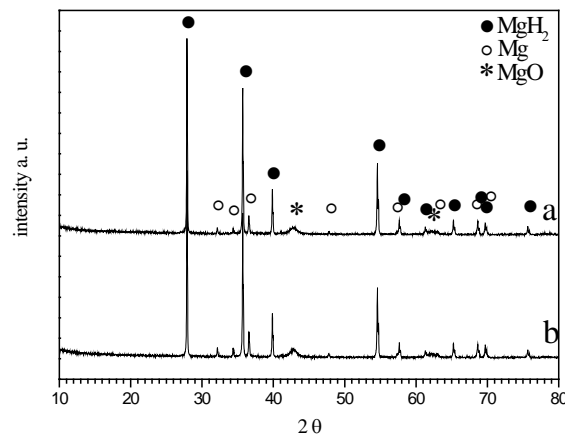

Fig. 3. X-ray diffraction pattern of the composites a) 95 wt. % MgH_2 - 5 wt. % activated carbon from apricot stones and b) 95 wt. % MgH_2 - 5 wt. % activated carbon from polyolefin wax obtained after hydriding at 573 K and 1 MPa and ten cycles.

The XRD patterns after ten cycles of hydriding/dehydriding and hydriding at 573K and 1 MPa are shown in Fig.3. β-MgH_2 was detected as the main phase and the diffraction patterns of both composites did not show substantial difference from these observed for ball milled samples (Fig. 1). Only presence of a minor quantity of MgO was registered.

The appearance of MgO could be explained by the higher surface area obtained and the sensitivity to oxidation after cycling and hydriding.
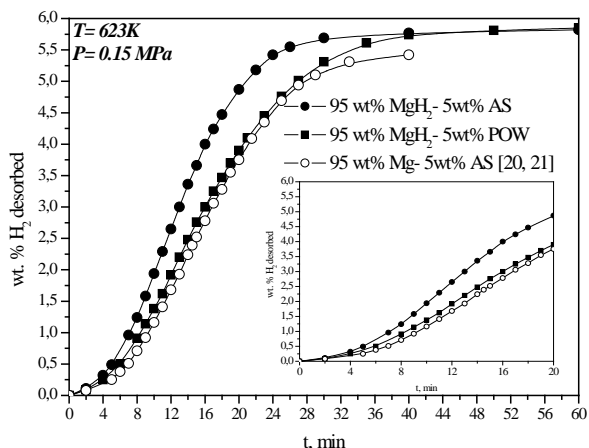

Fig. 4. Kinetic curves of hydrogen desorption from the composites 95 wt. % MgH$_2$ - 5 wt. % activated carbon from apricot stones and 95 wt. % MgH$_2$ - 5 wt. % activated carbon from polyolefin wax at P = 0.15 MPa and T= 623 K.
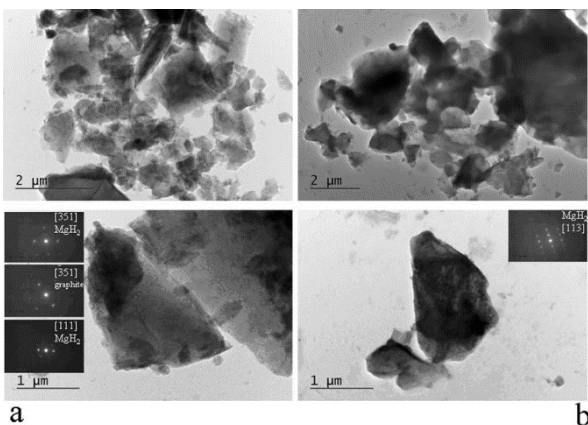

Fig. 5. TEM bright field micrograph and electron diffraction pattern of the composites a) 95 wt. % MgH$_2$ - 5 wt. % activated carbon from apricot stones and b) 95 wt. % MgH$_2$ - 5 wt. % activated carbon from polyolefin wax obtained by ball milling.

The fastest desorption rate at 623 K and 0.15 MPa was demonstrated by the composite containing activated carbon derived from apricot stones (Fig. 4). During the first few minutes of desorption all samples showed overlapping curves as it can be seen from the inset graph in Fig. 4. Desorption at a lower temperature of 573 K was not observed. The desorption rate of MgH$_2$-AS was faster than that published before for Mg-AS [20, 21]. This could be due to the use of MgH$_2$, which has a higher surface area and is more brittle material.
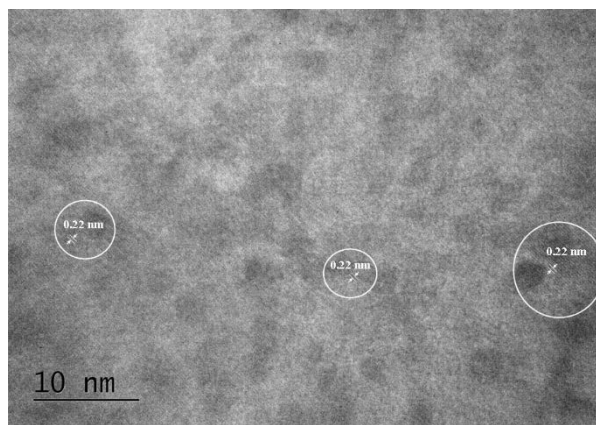

Fig. 6. HTREM image of the composite 95 wt. % MgH$_2$ - 5 wt. % activated carbon from polyolefin wax obtained by ball milling.

For the activated carbons derived from apricot stones and polyolefin wax the same values of specific surface area were measured, but they differ in some other characteristics. Activated carbon derived from apricot stones has a higher carbon content and a higher micropore volume than activated carbon derived from polyolefin wax (Table 1). The results from IR spectroscopy (figure not shown) demonstrated the presence of oxygen-containing surface groups for both activated carbons [20, 27]. It was shown in the literature that the electrochemical hydrogen storage is favoured in carbon materials with a well developed porosity and a low content of surface oxygen complexes [25]. The functional groups on the surface of the activated carbons, as well as the porosity have a strong effect on the adsorption properties. According to Su *et al.* [26], adsorption capacity of activated carbon for non-polar molecules such as hydrogen, increases with the increase in the amount of surface acidic groups. Additionally, Gergova *et al.* [24] established that a higher amount of surface chemical groups containing oxygen or another heteroatom in activated carbons derived from agricultural by-products, ensures higher concentrations of active sites. Both activated carbons have well developed pore structure and are suitable for the adsorption of small molecules. The activated carbon derived from apricot stones has a higher carbon content and this can probably explain the better hydrogen adsorption/desorption properties as slightly increased hydrogen absorption capacity and faster desorption for MgH$_2$-AS than for MgH$_2$-POW.

Table 1. Chemical characterization, BET surface area and pore volume of the activated carbons, determined by N$_2$ adsorption

Type of activated carbon	S_{BET} m^2/g	Ash^{mf} wt. %	V_{tot} ^{a} cm^3/g	V^b_{micro} cm^3/g	V^b_{meso} cm^3/g	C^c wt. %	H^c wt.%	S^d wt.%	O^e wt. %

Activated carbon from apricot stones [21, 24]	800	1.10	0.50	0.36	0.04	95.1	1.3	0.7	1.9
Activated carbon from polyolefin wax	800	0.11	0.60	0.27	0.14	87.4	3.5	0.5	8.6

mf – moisture-free sample; a - evaluated at relative pressures of 0.99; b - evaluated by DFT method applied to N_2 adsorption data using slit-shaped pore model; c - data from combustion elemental analyzer; d - data from Eshka method; e - determined by difference; V_{tot} - total pore volume; V_{micro} - volume of micropores and V_{meso} - volume of mesopores; the chemical composition results are for moisture-free samples

TEM, SAED and HRTEM images of the composites after ball milling are presented in Figs. 4 a, b and Fig 5. The samples consisted of agglomerates and two principal groups of small and large particles. The particle size of the ball milled powders was varied from less than 1 μm to several μm. The composite containing activated carbon derived from apricot stones had finer particles and a separation between them was more clearly observed. MgH_2 well defined hexagonal shaped particles were not visible in contrast to the study of Chi Xue Zhou et al. [19]. Probable reason for that was the formation of mixed areas of magnesium hydride and carbon. The single crystal electron diffraction patterns revealed the presence of tetragonal MgH_2, Mg and graphite. The interplanar distance of the lattice fringes of about 0.22 nm belongs to (200) planes of β-MgH_2. Other lattice fringes that were observed are these of MgO - $d_{200} = 0.21$ nm, Mg - $d_{100} = 0.28$ nm and graphite - $d_{002} = 0.34$ nm and $d_{100} = 0.21$ nm. XRD patterns in Fig. 1 show no presence of MgO in both samples after ball milling, but HTREM detected this phase. Probably MgO content in ball milled composites is below the detection limits of XRD analyses. Only for the sample containing activated carbon derived from apricot stones, mixed areas on the surface containing MgH_2 and graphite were observed by SAED and HTREM. For the sample containing POW, no graphite was detected.

The influence of the carbon additive morphology on its catalytic activity concerning magnesium sorption properties was studied by Fuster et al. [13]. They used graphite in form of powder or flakes as an additive to Mg and MgH_2. Sorption rates were practically the same for both samples and they implied that the catalytic activity of graphite does not rely upon its initial microstructure or morphology [13]. In our experiments, as it is seen in Figs. 2 and 4, the effect of magnesium on the hydrogen absorbing properties depends on the starting material - Mg or MgH_2 and also on the type of activated carbon. It was shown that there is no substantial difference in absorption kinetics, but the effect of the additive was more pronounced in the kinetics of desorption. These two additives of activated carbon have different micropore volume, carbon and oxygen content, which play a role in the absorption of hydrogen.

CONCLUSIONS

Hydrogen sorption properties of 95 wt% MgH_2-5 wt% activated carbon derived from apricot stones-AS or polyolefin wax-POW synthesized by high energy ball milling in argon medium were studied. Probably, because of the short time of ball milling, no formation of γMgH_2 was observed. After 60 min of hydriding at a temperature of 573 K and a pressure of 1 MPa, the composites showed no substantial difference in hydrogen absorption rate and capacity; the one containing AS reached 5.8 wt% versus 5.4 wt% for the second one containing activated carbon from POW.

More pronounced positive effect on the hydrogen desorption of MgH_2 was demonstrated by the activated carbon derived from apricot stones. It seems likely that for the investigated composites, after ball milling the activated carbon prepared from apricot stones had better contact with MgH_2, its addition also resulted in finer particles with less agglomerates compared to activated carbon derived from polyolefin wax. Carbon containing additive with high pore volume and higher carbon content has a more favorable effect on the hydrogen sorption characteristics of magnesium.

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ЕФЕКТ НА АКТИВНИ ВЪГЛЕНИ, ПОЛУЧЕНИ ОТ КАЙСИЕВИ КОСТИЛКИ ИЛИ ПОЛИОЛЕФИНОВ ВОСЪК ВЪРХУ СОРБЦИОННИТЕ СВОЙСТВА ПО ОТНОШЕНИЕ НА ВОДОРОД НА MgH₂

Е. Григорова[1], М. Христов[1], Б. Цинцарски[2], И. Стойчева[2], Д. Нихтянова[1,3], П. Марков[1]

[1] Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences, bl.11, Acad. G. Bonchev str., 1113 Sofia, Bulgaria
[2] Institute of Organic Chemistry, Bulgarian Academy of Sciences, bl. 9, Acad. G. Bonchev str., 1113 Sofia, Bulgaria
[3] Institute of Mineralogy and Crystallography, Bulgarian Academy of Sciences, Acad. G. Bonchev Str., bl. 107, 1113 Sofia, Bulgaria

(Резюме)

Свойствата на хидриране и дехидриране на композити със състав- 95 мас.% MgH₂ - 5 мас. % С, синтезирани с помощта на механоактивиране в атмосфера от аргон с две различни въглерод- съдържащи добавки са изследвани (активен въглен получен от кайсиеви костилки- AS и полиолефинов восък- POW). Двете проби не показват съществена разлика в кинетиката на абсорбция на водород и достигат абсорбционен капацитет от 5.8 мас.% за 95 мас.% MgH₂- 5 мас. % AS и 5.4 мас . % за 95 мас.% MgH₂- 5 мас. % POW при 573 К и 1 MPa след 60 мин. хидриране. По- висока скорост на десорбция при 623 К и 0.15 MPa показа композита съдържащ активен въглен получен от кайсиеви костилки. Морфологията и фазовият състав на образците са изследвани чрез ТЕМ и XRD.