



## Short communication

## The study of hydrogen sorption in palladium limited volume electrode from DEMA-TFO ionic liquid

M. Pająk<sup>a,b</sup>, K. Hubkowska<sup>a,\*</sup>, A. Czerwiński<sup>a,c</sup><sup>a</sup> University of Warsaw, Faculty of Chemistry, Pasteura 1, 02-093 Warsaw, Poland<sup>b</sup> University of Warsaw, Biological and Chemical Research Centre, Żwirki i Wigury 101, 02-089 Warsaw, Poland<sup>c</sup> Industrial Chemistry Research Institute, Rydygiera 8, 01-793 Warsaw, Poland

## ARTICLE INFO

## Keywords:

Hydrogen sorption  
 Ionic liquids  
 DEMA-TFO  
 Palladium limited volume electrode  
 Hydrogen pretreatment procedure (HPP)

## ABSTRACT

Hydrogen sorption into palladium limited volume electrode (Pd-LVE) was studied in protic ionic liquid – diethylmethylammonium triflate (DEMA-TFO) using cyclic voltammetry (CV), chronopotentiometry (CP) and chronoamperometry (CA) at constant temperature (298 K). Pd-LVE was obtained potentiostatically from the aqueous solution of PdCl<sub>2</sub>. The results were compared with those obtained in aqueous 0.5 M H<sub>2</sub>SO<sub>4</sub> solution. As the process of hydrogen sorption in ionic liquid medium is much less reversible than in acidic aqueous solution – the hydrogen pretreatment procedure (HPP) significantly increases the reversibility of this process at temperature of 298 K. The amount of absorbed hydrogen in DEMA-TFO is comparable with this in aqueous solution of acid and reaches the maximum value of H/Pd ca. 0.7. The hydrogen absorption isotherm was measured with the use of CV and CP techniques.

## 1. Introduction

The increasing energy consumption is commonly based on limited fossil fuel resources and it is necessary to move to the new forms of energy in the future. An interesting alternative is the use of the hydrogen which has the highest heating value of all chemical fuels but we have to find an efficient way of hydrogen storage. Over the last decades a large number of technologies have been developed and one of them is based on metal hydrides [1–3] that may reversibly store hydrogen by absorption mechanism. Among transition metals palladium-hydrogen system have been extensively studied and it is a model system for hydrogen-absorbing materials [4,5]. Palladium absorbs hydrogen under ambient conditions and it has a high absorption capacity. It forms palladium hydrides, PdH<sub>x</sub>, where values of x are up to 1. The sorption of hydrogen in palladium has been studied from the gas phase [6,7] or in aqueous solutions of acids and bases [8–11] and there are very few reports on the hydrogen sorption in ionic liquids. Recent reports of Meng et al. [12] and Martindale et al. [13] show that hydrogen absorption into palladium is possible from ionic liquids. Tremblay et al. [14] used electrochemical quartz crystal microbalance (EQCM) measurements and amperometry to determine the amount of hydrogen absorbed. H/Pd calculated from EQCM and CA measurements equals to 1 and 0.8 respectively. We decided to investigate this process using palladium limited volume electrode (Pd-LVE) [9,15] in selected protic

ionic liquid (PIL) – diethylmethylammonium triflate (DEMA-TFO). PILs are combinations of Brønsted acids and bases where Brønsted bases are acceptors of the protons from the Brønsted acids. Thereby they acts as proton-carriers [16] and can be a hydrogen source in absorption measurements. DEMA-TFO appears to be a suitable medium for such measurements, since Ejigu and Walsh [17] claims that the adsorption of monolayer of hydrogen atoms occurs at the Pt electrode during cycling in DEMA-TFO and it is not blocked by IL anions. Recently, there can be noticed an increase in interest in PILs due to their high synthetic and computational availability [18]. Thus, PILs seems to be alternative electrolytes in many areas of the chemical industries i.e. in fuel cells [16], thermoelectrochemical materials [19], or supercapacitors [20,21].

Pd thin film electrode is a very good model system to obtain preliminary results of hydrogen sorption in protic ionic liquids in the context of further research with the use of multicomponent alloys, since PILs and their mixtures with other solvents have been recently proposed as electrolytes in metal-hydride batteries [22].

In this paper the process of hydrogen sorption from DEMA-TFO ionic liquid in Pd-LVE is examined with the use of electrochemical techniques such as CV and CP. Tremblay et al. [14] have depicted the possibility of hydrogen sorption in Pd electrode in DEMA-TFO. This group stressed the influence of the temperature on the enhancement of reversibility and rate of the hydrogen sorption process. In our research

\* Corresponding author.

E-mail address: [khubkowska@chem.uw.edu.pl](mailto:khubkowska@chem.uw.edu.pl) (K. Hubkowska).

we present a comprehensive study of the electrochemical sorption of hydrogen in Pd electrode from DEMA-TFO at temperature of 298 K. Recently, we emphasized the crucial role of HPP in the examination of hydrogen sorption from strong basic medium electrolyte [11]. Therefore, we expected the significance of HPP also in the further study of hydrogen sorption in non-aqueous solutions. All the presented results are compared with those for 0.5 M sulfuric acid aqueous solution.

## 2. Experimental

All electrochemical experiments were carried out in diethylmethylammonium triflate (DEMA-TFO, IoLiTec, Germany) under Ar stream at constant (298 K) temperature controlled by Lauda Eco Re 630 thermostat. Water content (Karl-Fisher titration) equals to ca. 0.28% in case of ionic liquid before measurements and maximum ca. 1.6% for the one after 4 days experiment of continuous electrochemical hydrogen sorption involving HPP (CV, CA) and hydrogen sorption performed with the use of CV and CP techniques. Three-electrode system with a Pd-LVE working electrode, an Ag wire as a quasi-reference electrode and a Pt spiral as auxiliary electrode was used. The reference electrode was calibrated using 5 mM ferrocene,  $E(\text{Fc}^{0/+}) = 0.20 \text{ V}$  (vs Ag wire). Pd-LVE electrode (0.5  $\mu\text{m}$  thick, 2 mm high, except results in Fig. 1a containing different thicknesses) was obtained electrochemically on a gold wire (0.5 mm diameter) from the 0.11 M  $\text{PdCl}_2$  in 1 M HCl solution. The thicknesses of Pd electrodes were determined on the basis of the deposition charges (obtained from the integration of CA signals) and Faraday's law. Unless otherwise stated, all potentials were recalculated vs. RHE (potential measured with the use of Hydroflex electrode,

Gaskatel, GmbH, Germany). Freshly deposited Pd-LVE electrode was subjected to series of voltammetric and chronoamperometric cycles – hydrogen pretreatment procedure (HPP) – similarly to the procedure used in [11]: ca. 30 CV cycles in the potential range  $-1.05 \div 0.95 \text{ V}$  (scan rate  $1 \text{ mVs}^{-1}$ ) alternating with CA steps (10 CA steps on every CV cycle, summarily ca. 300 CA steps: the potential of hydrogen absorption/desorption in CA:  $-1.05 \text{ V}/0.95 \text{ V}$ ). The termination of HPP was indicated when repeatable CV course was obtained. All electrochemical measurements were performed using CH Instruments potentiostat, model 760D. Hydrogen capacity (expresses as the hydrogen to palladium atomic ratio – H/Pd) was calculated on the basis of hydrogen oxidation charge (from the integration of CV signal), palladium deposition charge and Faraday's law [23].

## 3. Results and discussion

Fig. 1a shows CV curves for the Pd-LVE electrodes of different thicknesses after HPP. What is clearly visible, the magnitude of oxidation and reduction signals increase with the increase of electrode thickness. Also, it is noteworthy that there are no other signals in this potential region. Hydrogen absorption is the process which occurs in the bulk of the palladium, hence the rise of oxidation/reduction signals (for CVs measured in the same potential region) for electrodes of various thicknesses implies that the nature of those signals is associated with hydrogen absorption (reduction) and desorption (oxidation).

Analysis of CV curves in Fig. 1a and b enables to state that the hydrogen absorption/desorption peaks after HPP in DEMA-TFO are sharp and well developed in contrast to poorly defined signals before

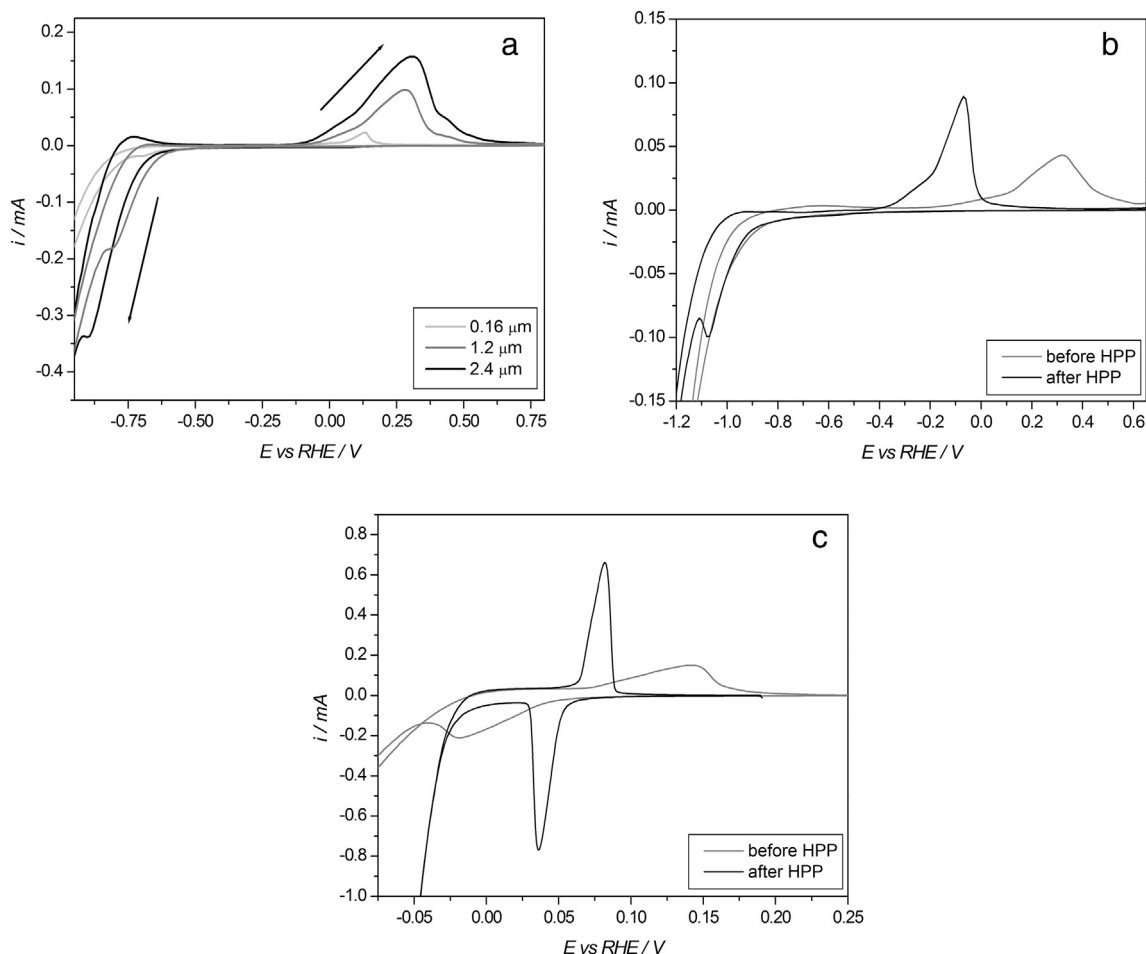


Fig. 1. a) Cyclic voltammetry curves (CVs) of Pd electrodes of different thicknesses registered after HPP in DEMA-TFO; b) CVs of Pd-LVE in DEMA-TFO; c) CVs of Pd-LVE in 0.5 M  $\text{H}_2\text{SO}_4$ . Scan rate:  $1 \text{ mVs}^{-1}$ .

Download English Version:

<https://daneshyari.com/en/article/9951804>

Download Persian Version:

<https://daneshyari.com/article/9951804>

[Daneshyari.com](https://daneshyari.com)