

Water splitting in PEM electrolysis with Ebonex supported catalysts

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A series of mono- and bi-metallic compositions, containing Pt, Co and Pt-Co in various ratios, supported on Ebonex were prepared using the borohydride wet chemical reduction method. It was found that PtCo/Ebonex shows enhanced efficiency toward oxygen evolution in PEM water electrolysis compared to pure metallic Pt. The activity changes in the order: Pt₃Co₂/Ebonex > Pt₃Co₂ > Pt₃Co/Ebonex > Pt/Ebonex > Co/Ebonex. The effect was explained by the formation of solid solution between the metallic components combined with hyper-hypo-d-electron metal-support interactions. These effects, together with the high stability of the supporting material against oxidation, give reason to consider Ebonex as alternative support for the anodic catalytic materials used in PEM water electrolysis.

Key words: PEM water electrolysis, Pt, Co, Ebonex.

INTRODUCTION

Polymer Electrolyte Membrane Water Electrolysers (PEMWE) are the newest development in the electrolyser technology with high potential for further improvements and with main advantages, compared to conventional alkaline water electrolysis such as no use of fluid electrolyte, lower energy parasitic losses and very high purity of the produced hydrogen [1]. However, this technology is still expensive since the used catalysts are predominantly Pt and Pt alloys. The main source of energy dissipation during the electrochemical splitting of water is the oxygen evolution reaction (OER). Therefore, the synthesis of highly active and cost efficient OER catalysts is of great importance for the effective functioning of PEMWEs.

One way to reduce the cost of the catalysts is to alloy Pt with other less expensive transition metals (Fe, Co, Ni, Cu, Mo, Sn, Pd, Ir) [2–14]. Alloying with base metals can reduce the amount of Pt and at the same time increase the stability of the catalyst impeding the Pt surface mobility and thus, preventing the particles growth and formation of clusters [9, 15]. Another way is the development of composite catalysts with increased activity through realization of synergetic effects with the catalytic substrate [9, 16, 17]. The main advantage of the most used carbon support is its very high surface area combined with excellent electroconductivity. Its big disadvantage is the lack of stability at high anodic potentials.

Recently, Magneli phase titanium oxides with general formula Ti_nO_{2n-1} known under the trade name Ebonex (Atraverda Inc., UK) have been investigated as a substitute support material of carbon [18–20]. Ebonex has a unique combination of electrical conductivity approaching that of a metal and high corrosion resistance approaching that of ceramics. The primary role of Ebonex is to suppress the agglomeration of catalyst particles during the synthesis. In addition, interelectronic and/or interionic interactions with the metallic components, affecting the catalytic efficiency of the composite material are expected since its hypo-d-electron character suggest an ability to interact with metals such as Pt, Ni, Co.

The method of wet chemical reduction with NaBH₄ is a well known technique for synthesis of nanoscale metal particles, metal nanowires and similar materials with various chemical compositions. The most important prerequisite for nanoparticles synthesised by the borohydride reduction process is the knowledge of and the control over the kinetic parameters determining their nucleation and growth. The nucleation rate and growth are tools for control of those nanoparticles in size, shape, structure and properties, that provides a large choice for their most successful applications [21].

Our previous investigations showed that Pt-Co/Ebonex catalyst prepared by the borohydride reduction method facilitates essentially the oxygen evolution reaction in alkaline aqueous solutions compared to both the supported Pt/Ebonex and unsupported Pt-Co catalysts [7].

The aim of this research is to investigate the catalytic efficiency of mixed Pt-Co catalyst dis-

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persed on the interactive support of Ebonex in regard to oxygen evolution in PEM water electrolysis.

EXPERIMENTAL

Synthesis of the catalysts. A series of mono- and bi-metallic compositions, containing Pt, Co and Pt-Co in various ratios, was prepared using the borohydride wet chemical reduction (BH) method. The synthesis of the catalyst nanoparticles and their deposition upon Ebonex required first synthesizing of platinum and cobalt ethylenediamine complexes serving as metal precursors according to a previously developed preparation procedure [7, 22, 23]. In a second stage, the prepared aqueous solutions of the precursors (single- and mixed-metal complexes) were impregnated on the surface of the support and then reduced by addition of sodium borohydride solution under continuous stirring at 40°C for several minutes. The procedure was carried out with different ratios of precursors in order to obtain the desired composition (Table 1).

Table 1. Pt:Co ratio, Pt cell parameter and crystal size of the synthesized mono and bimetallic materials

Sample	Initial composition, Pt:Co ratio, at.%	Pt cell parameter, Å	Crystal size, nm
Pt/Ebonex	1:0	3.9191 ± 0.0018	4.2 ± 0.7
Pt3Co/Ebonex	3:1	3.9050 ± 0.0037	3.9 ± 0.6
Pt3Co2/Ebonex	3:2	3.8783 ± 0.0149	2.1 ± 0.4
Pt3Co2	3:2	3.8539 ± 0.0002	2.3 ± 0.2
Co/Ebonex	0:1	amorphous	

Characterization of the synthesized electrocatalysts. The phase composition, morphology and surface structure of the catalysts under study were investigated by X-Ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). XRD spectra were recorded by X-ray diffractometer Philips APD-15. The diffraction data were collected at a constant rate of $0.02^\circ \cdot s^{-1}$ over an angle range of $2\theta = 10-90^\circ$. The average particle size (in our case it coincides with crystallite size) was calculated from the broadening of the XRD peaks using the Scherrer's equation [24]. The XPS of the samples were recorded with an ESCALAB MK II (VG Scenific, England) electron spectrometer. The photoelectrons were excited with a twin anode X-ray source using Mg $K\alpha$ ($h\nu = 1253.6$ eV) radiation. C1s photoelectron line at 285.0 eV was used as a reference for calibration.

Laboratory PEM cell and test procedure. The electrochemical tests were performed on membrane electrode assemblies (MEAs), using a Nafion 117

membrane (Alfa Aesar) as an electrolyte. The catalysts under study were investigated as electrodes for the oxygen evolution reaction, while a commercial E-TEK catalyst containing 20% Pt on carbon support was used to prepare both the hydrogen (HE) and the reference electrodes (RE). The electrodes have a complex multilayer structure, consisting of gas diffusion, backing, and catalytic layers. The backing layer was made of a mixture of carbon particles (Shawinigan Acetylene Black) and 30% of PTFE suspension, deposited on thin carbon cloth, serving as a gas diffusion layer. The catalytic layer was spread upon the backing one as an ink (catalyst particles mixed with diluted Nafion ionomer) by several steps. After each step the electrode was dried for 30 min at 80°C and weighted, using an analytical microbalance Boeco, Germany. The procedure was repeated until a metal loading of $5 \text{ mg}\cdot\text{cm}^{-2}$ was reached. Then the electrodes were hot pressed onto the PEM electrolyte forming the test MEA. The hot pressing was performed stepwise in the regime of gradual temperature and pressure increase starting from 50°C and $6 \text{ kg}\cdot\text{cm}^{-2}$ and ending with 120°C and $12 \text{ kg}\cdot\text{cm}^{-2}$, respectively.

The PEM electrochemical cell used for testing of the prepared catalysts is presented in Fig. 1. It consists of two gas compartments, where the hydrogen and oxygen evolution take place, separated by the MEA.

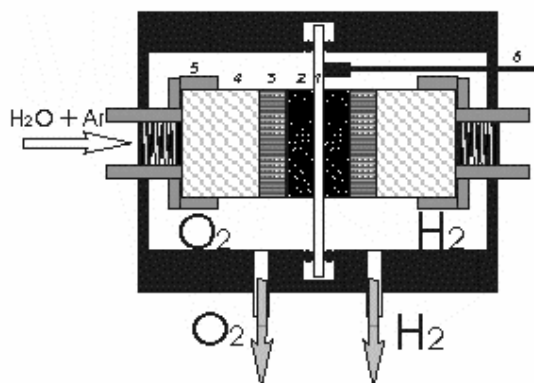


Fig. 1. Scheme of the test PEM electrolytic cell: 1) Nafion membrane; 2) catalytic layer; 3) carbon cloth; 4) foamed Ni plate; 5) PTFE rings; 6) reference electrode.

The HE and RE are located in one compartment so that during the operation, the former ensures the atmosphere required for the stable potential of the latter. The complete design of the model test PEM electrolyser includes also graphite cloths, which ensure a good electronic contact and foamed nickel plates, serving as current collectors. The whole stack is fastened on both sides by metal springs and is insulated from the stainless steel housing of the cell by PTFE rings.

The CV curves were recorded at room (20°C) temperature, using a potential scanning rate of 20 $\text{mV}\cdot\text{s}^{-1}$. The steady state polarisation tests were run in galvanostatic mode at operating temperature of 80°C. All electrochemical measurements were carried out with a commercial Galvanostat/Potentiostat POS 2 Bank Elektronik, Germany.

RESULTS AND DISCUSSION

Fig. 2 represents the XRD spectra of the synthesized mono and bimetallic materials and in Table 1 the results obtained by X-ray diffraction analysis are summarized.

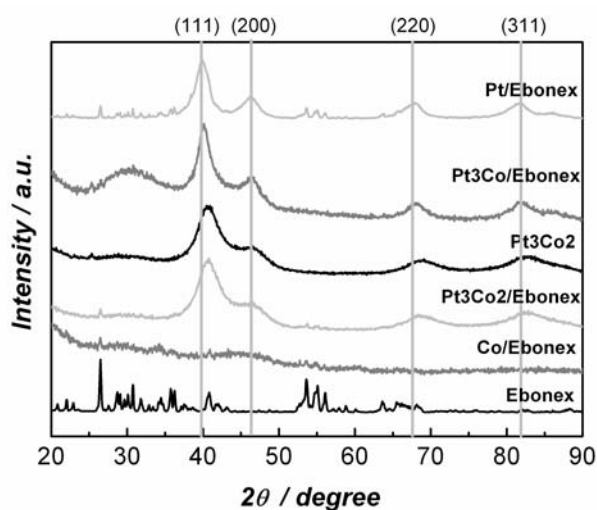


Fig. 2. XRD spectra of the studied Pt and Pt-Co catalysts.

The patterns show that the monometallic Co sample is amorphous, while the rest of the samples have Pt peaks with varying half-width and 2θ position, reflecting the differences in the size of the metal crystallites and the crystal lattice parameters. The calculated parameters of the crystal unit cell for the Pt/Ebonex sample correspond to pure Pt. For all other samples in the series the parameters of the lattice are lower. This is an indication of inclusion of Co inside the Pt unit cell and formation of solid metal solution. In addition, there is a trend to decrease the crystal size, respectively increase in the catalyst specific surface area with the increase in the Co content. The particle diameters calculated from full width of the half-peak maximum are in the range 4.2 to 2.1 nm.

The smallest crystal size has the catalyst Pt3Co2 deposited on Ebonex, suggesting an increased surface area and enhanced activity in accordance with other authors who reported higher catalytic activity of Pt-based binary electrocatalysts (Pt-Co, Pt-Cr, Pt-Ni) [3, 8, 25, 26]. The data of unsupported sample (Pt3Co2) are not significantly different from those deposited on Ebonex.

The electrochemical investigation of the catalytic activity was performed by the method of cyclic voltammetry (CV) and steady state polarization. The CV gives some qualitative information about the electrochemical activity and the nature of the processes occurring on the catalyst surface. CV curves of MEAs containing Pt3Co2/Ebonex catalyst or unsupported nanoparticles at the same ratio, Pt3Co2 at the anode are presented in Fig. 3. The latter curve was included in order to follow the influence of the substrate on the catalyst performance.

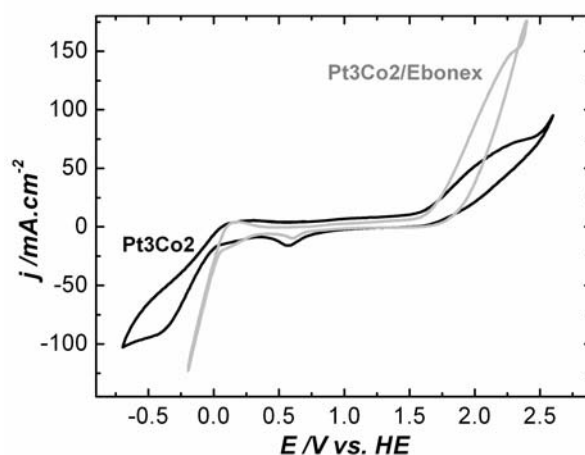


Fig. 3. Cyclic voltammetric curves of Pt3Co2/Ebonex and unsupported Pt3Co2 nanoparticles at 20°C and scanning rate of 20 $\text{mV}\cdot\text{s}^{-1}$.

The observed current peaks corresponding to the formation of oxygen coverage on the surface (Pt-O, Pt-OH) are not very well depicted on both CV curves shown. They are spread in a broad potential range. Both hydrogen and oxygen evolution reactions start at lower overpotentials, when the Pt3Co2 nanoparticles are deposited on Ebonex. The effect is more pronounced for the OER, which commences at potential of about 1.5 V.

The main results from the steady state electrochemical characterisation of the prepared catalysts, incorporated in MEAs are summarised in Fig. 4. The galvanostatic polarization curves of the partial electrode reaction (OER) involved in the electrolytic water splitting are recorded at typical PEMWE operation temperature of 80°C. For comparison in Fig. 4 data are included from polarisation experiments performed on MEAs with different Pt:Co ratios deposited of Ebonex as well as unsupported Pt3Co2 nanoparticles at the anode and a constant total metal loading of 5 $\text{mg}\cdot\text{cm}^{-2}$.

It can be seen that the monometallic Co/Ebonex sample shows an insignificant catalytic action, while all Pt-Co catalysts, supported on Ebonex have higher catalytic effect compared to the Pt/Ebonex monometallic sample and unsupported catalyst. This

demonstrates the positive influence of the cobalt inclusions on the activity of Pt, observed also by other authors [4, 7, 8] and secondly the Ebonex support [7, 9, 16, 20, 27].

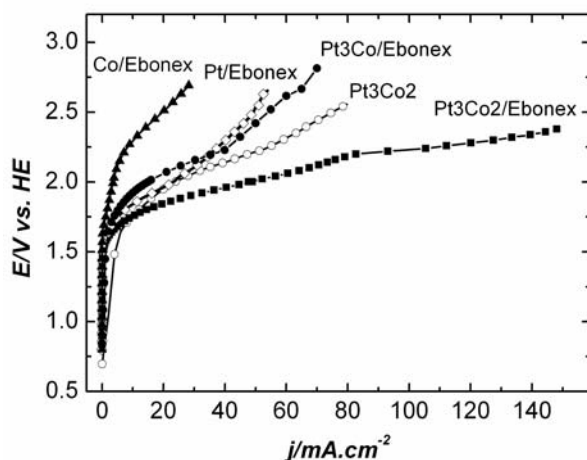


Fig. 4. Galvanostatic polarisation curve of Pt-Co catalysts with varying metal ratios at 80°C.

The best catalytic activity for oxygen evolution reaction is exhibited by the sample 3Pt2Co/Ebonex (sample with the lowest crystal size). It has higher efficiency relative to all other compositions, including Pt/Ebonex. Despite the similarity in the cells parameters the activity on Ebonex supported catalyst is higher. The activity changes in the order: Pt3Co2/Ebonex > Pt3Co2 > Pt3Co/Ebonex > Pt/Ebonex > Co/Ebonex. The combination of elements from the left side of the transition series in the Periodic Table, having empty or half-filled vacant semi-d-orbitals (hypo-d-electronic metal) with metals of the right half of the transitions series, having internally paired d-electrons (hyper-d-electronic metals) is already a proven approach to obtain efficient catalyst with decreased noble metal loadings. Such composite catalysts exhibit a well pronounced synergism toward OER and HER [7, 16, 27]. By dispersion (grafting) of transition metal intermetallic phases on the active centres of selected catalytic supports, it is possible to achieve a strong synergetic effect known as strong metal-support interaction (SMSI). In this way, it is possible to decrease the amount of the catalysts for a unit of electrode surface and to increase essentially the catalytic activity of the composite catalyst [19, 26–28].

Having these facts in mind, the positive effect of Ebonex support could be explained by formation of solid solution between the metallic components (registered in XRD spectra) combined with hyper-hypo-d-electron metal-support interaction between the metal components and the substrate.

In order to verify this assumption the surface state of the as-prepared Pt/Ebonex, Pt3Co2/Ebonex and Pt3Co2 catalysts has been studied further by photoelectron spectroscopy. The results obtained are represented in Fig. 5 and summarised in Table 2.

The results shown in Fig. 5 and Table 2 indicate that the Pt 4f spectrum (Fig. 5a) can be deconvoluted with three doublet components corresponding to 3-valence states of the platinum [29]. The metallic state Pt⁰ is dominant for all samples. Small amount of Pt(OH)₂ and PtO₂ are present too.

The Co 2p core level spectrum is characterized by two components due to Co 2p_{3/2} and Co 2p_{1/2}, and shake-up satellites. The Co 2p_{3/2} binding energies of Co²⁺ and Co³⁺ are very close and this makes determination of the oxidation states uncertain. But BE separation of the satellite signals from the Co 2p_{3/2}-Co 2p_{1/2} photo lines, as well as spin orbital splitting are dependent on the chemical state of cobalt. The high spin Co²⁺ compounds such as CoO (780 eV), Co(OH)₂ (780–781 eV) exhibit strong satellite lines, which are located at about 5–6 eV above the main line, in contrast to Co³⁺ that exhibits a weak satellite shifted to higher binding energies (Co₃O₄, CoOOH) [29–31]. The separation of the Co 2p_{3/2}-2p_{1/2} spin-orbit components is larger by about 1 eV for the high spin Co²⁺ compared to the low spin Co³⁺ valence states. The splitting is usually 16.0 eV for Co²⁺ and 15.0 eV for Co³⁺ and Co⁰. The spectrum of metallic cobalt does not contain shake-up satellite and gives rise at 778 eV Co 2p_{3/2}.

Fig. 5b shows deconvoluted Co2p spectra for catalysts Pt-Co 3:2. Three components can be distinguished. In accordance with the above data the component with lowest BE corresponds to metallic cobalt (Table 2). The appearance of both components at ~781 and ~785.6 eV and the value 15.7–15.8 eV for spin-orbital splitting means that cobalt is present in the second valence state. The intensity proportion between Co⁰ and Co²⁺ indicates that only 34–35% are in the metallic state. Namely, this part of cobalt incorporates into platinum cell. Taking into account the XRD patterns, the remaining larger part of the cobalt is in the amorphous state.

Additionally, from the fact, that the O1s spectra are situated around 532 eV (Fig. 5c), which corresponds to metal–OH bonds, one can conclude that the Co²⁺ and Pt²⁺ states are in the hydroxide form i.e. Co(OH)₂ and Pt(OH)₂ respectively. The low energy O1s component at about 530 eV due to the oxygen in the metal–O–metal bond and high energy component (533.5–534 eV) can be assigned to adsorbed H₂O and/or C=O bonds [29, 32].

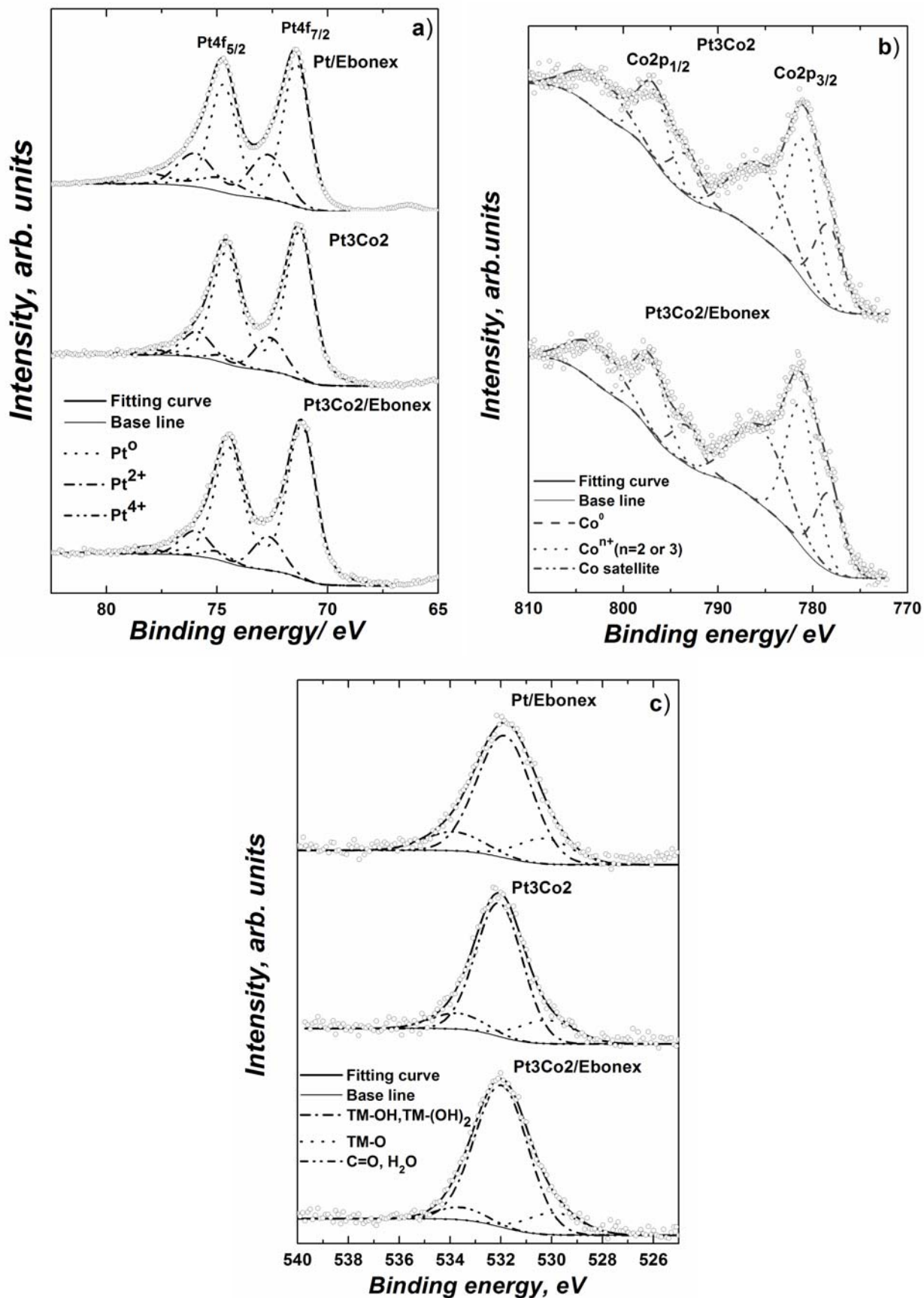


Fig. 5. XPS spectra of Pt-containing catalysts: a) Pt 4f, b) Co2p, c) O1s.

Table 2. Summarized data from XPS spectra.

XPS lines		Pt4f _{7/2}			Co2p _{3/2}				O1s			
Sample	Chemical species	Pt ⁰	Pt ²⁺ PtO Pt(OH) ₂	Pt ⁴⁺ PtO ₂ Pt(OH) ₄	Co ⁰	Co ²⁺ Co(OH) ₂ , CoO		Satellite	TM–O	TM(OH) _x	H ₂ O, C=O	
						spin orbital splitting	spin orbital splitting		(TM = Pt, Ti, Co)			
Pt/Ebonex	BE eV	71.42	72.70	74.76					530.27	531.86	533.85	
	%	65.47	25.37	9.16					15.33	72.57	12.11	
Pt3Co2	BE eV	71.28	72.61	74.61	778.36	14.99	781.08	15.68	785.61	530.19	532.06	533.71
	%	78.93	17.45	3.62	35.30		64.70			15.65	74.26	10.09
Pt3Co2/ Ebonex	BE eV	71.17	72.68	74.96	778.28	15.03	781.15	15.84	785.60	529.90	531.97	533.58
	%	79.73	16.43	3.84	34.30		65.70			12.68	80.72	6.60

The results imply the formation of solid solution of both metals and verify to a great extent the expected formation of surface bonds between the metallic components of the catalyst and the supporting material.

In order to check the stability of the synthesized catalysts the development of the electrode potential with time was followed under galvanostatic conditions at current density of 100 mA·cm⁻² for period of 24 h. These results show a stable catalytic property of the Pt-Co/Ebonex catalyst. The measured initial electrode potential was 1.72–1.75 V. It varied slightly during the first 8 hours of the test and after reaching a value of about 1.6 V, no further changes were observed (Fig. 6).

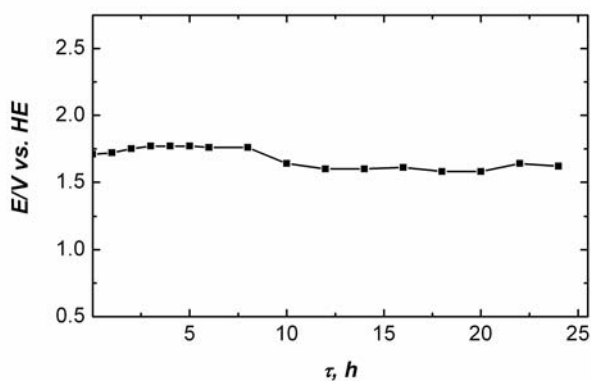


Fig. 6. Long-term performance of Pt3Co2/Ebonex recorded in a galvanostatic mode at 80°C and current density 100 mA·cm⁻².

CONCLUSIONS

The results demonstrate that Pt-Co/Ebonex catalysts, obtained by the borohydride method of wet chemical reduction possess enhanced efficiency toward oxygen evolution compared to pure Pt. The effect is explained by the formation of solid solution between the metallic components combined with hyper-hypo-d-electron metal-support interactions.

These effects together with the high stability of the supportive material against oxidation, give reason to consider Ebonex as alternative support for the anodic catalytic materials for PEM water electrolysis.

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РАЗЛАГАНЕ НА ВОДА ЧРЕЗ ПЕМ ЕЛЕКТРОЛИЗА С КАТАЛИЗАТОРИ ВЪРХУ НОСИТЕЛ ЕБОНЕКС

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Серия от моно- и биметални състави, съдържащи Pt, Co и Pt-Co в различни съотношения, отложени върху Ебонекс, са получени по метода на мократа борхидридна редукция. Установено е, че PtCo/Ebonex показва висока ефективност по отношение на отделянето на кислород в ПЕМ водната електролиза в сравнение с чистата метална Pt. Активността се променя в реда: Pt₃Co₂/Ebonex > Pt₃Co₂ > Pt₃Co/Ebonex > Pt/Ebonex > Co/Ebonex. Резултатът е обяснен с образуването на твърд разтвор между металните компоненти, комбиниран с хипер-хипод-електронни взаимодействия между метала и носителя. Тези ефекти, заедно с високата стабилност на носителя срещу окисляване, дават основание Ебонекс да се разглежда като алтернативен носител за анодни каталитични материали, използвани в ПЕМ водната електролиза.