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Kaluža, Luděk
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Highly loaded carbon black supported Pt catalysts for fuel cells

L. Kaluža, M. Zdražil, D. Gulková, Z. Vít, O. Šolcová, K. Soukup, L. Maixnerová
Institute of Chemical Process Fundamentals of the ASCR, v.v.i., Prague, Rozvojová 135,
16502, Czech Republic, tel. +420 220390270, e-mail: kaluza@icpf.cas.cz.

Fulfilling of increasingly stringent environmental restrictions imposed on transportation drives the research on alternative energy resources such as hydrogen and fuel cells. Carbon supported Pt represents conventional catalyst in membrane electrode assembly, MEA. The aim of this work was to elucidate on the methods of Pt deposition on carbon black to achieve high loadings of Pt of about 60 wt.% in highly dispersed form. The carbon black supports were characterized by nitrogen physisorption. Platinum was deposited on the supports: (i) from true solutions of H_2PtCl_6 , $\text{Pt}(\text{C}_5\text{H}_7\text{O}_2)_2$, $\text{Pt}(\text{NH}_3)_4(\text{NO}_3)_2$, or $\text{Pt}(\text{NH}_3)_2(\text{NO}_2)_2$, and (ii) from fine dispersions of $\text{Pt}(\text{C}_5\text{H}_7\text{O}_2)_2$, PtO_2 , $\text{Pt}(\text{NH}_3)_4(\text{OH})_2$. Atomic adsorption spectroscopy, AAS, and scanning electron microscopy, SEM, were used for elemental analysis and determination of Pt content. The deposited Pt species were dried, calcined or reduced and were subjected to temperature programmed reduction, TPR. After degassing at increased temperature, the Pt dispersion was determined by hydrogen chemisorption at $-60\text{ }^\circ\text{C}$.

It was acquired that the used carbon black supports exhibited features of N_2 adsorption-desorption isotherms of type IV. The samples XC72R, XC72 (Vulcan, Cabot Corp.), and ENSACO 290G (Timcal Graphite & Carbon Ltd.) exhibited low surface area, $S(\text{BET})$, of about $226\text{-}236\text{ m}^2\text{g}^{-1}$ and low volume of micropores, $V(\text{Micro})$, of $55\text{-}65\text{ mm}^3\text{g}^{-1}$. In contrast, the supports TIMCAL research grade PT-P and ENSACO 350G (Timcal Graphite & Carbon Ltd.) exhibited about 2- and 4-fold higher $S(\text{BET})$ and $V(\text{Micro})$; they possessed also more developed microporous systems. Moreover, the sample PT-P clearly exhibited highly developed mesoporous system with maximum on the pore-size distribution curve at pore-size radius 7 nm, which was quite unique among the studied supports.

TPR revealed that PtO_2 , H_2PtCl_6 , $\text{Pt}(\text{C}_5\text{H}_7\text{O}_2)_2$, $\text{Pt}(\text{NH}_3)_4(\text{NO}_3)_2$, and $\text{Pt}(\text{NH}_3)_2(\text{NO}_2)_2$ deposited on carbon blacks and dried in rotary vacuum evaporator at $95\text{ }^\circ\text{C}$ were reduced to metallic Pt at 0, 70, 120, 140, 150 $^\circ\text{C}$, respectively. Hydrogen chemisorption experiments clearly showed that Pt dispersion in the catalysts mainly depended on the Pt precursor and activation conditions. Specific interaction of the Pt precursor with support surface was considered necessary for formation of the desired 4 nm Pt particles. This interaction might be partly considered as support corrosion and might lead to desirable new edge defects on the support surface, which promotes high Pt dispersion (4-6 nm particles). On one hand, the corrosion caused by H_2PtCl_6 or $\text{Pt}(\text{NH}_3)_2(\text{NO}_2)_2$ was mild and desirable. On the other hand, the corrosion observed while treating the $\text{Pt}(\text{NH}_3)_4(\text{NO}_3)_2$ containing samples was strong and undesirable. Bearing in mind the undesirable corrosion effects, the deposition of colloidal form of PtO_2 or $\text{Pt}(\text{C}_5\text{H}_7\text{O}_2)_2$ seemed to be particularly promising because it represented relatively clean and gentle method of Pt deposition. This was manifested by the sharp reduction peaks but the tailored high Pt dispersion was not reached.

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