Bimetallic Pd(II)/Fe(II)-Mediated Self-Assembly of Three-Dimensional Hybrid Multilayers with a Terpyridine-Contained Poly(vinylpyridine) Derivative as a Linker on Substrate Surface

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Figure S1. ¹H NMR spectra of PVP, TPyBr and PVPTPy.



Figure S2. FTIR spectra of (a) TPyBr, (b) PVP and (c) PVPTPy.



Figure S3. Absorption spectra for the (Pd)Fe-PVPTPy multilayers on the quartz substrate surface (**E**, Scheme 2). From bottom to up: BPy-Pd modified quartz, BPy-Pd-PVPTPy, and then one to three layers of (BPy-Pd)/(Fe-PVPTPy)_{1~3} multilayers.



Figure S4. Absorption spectra for the LBL assembly of Fe-PVP. Black line, the quartz plate covered by one layer of PVP (denoted as Q-PVP); Red line, Q-PVP covered by the first deposition of Fe/PVP layer; Green line, Q-PVP covered by twice deposition of Fe/PVP layer.



Figure S5. AFM images for the gold substrates covered by (A) the initial layer of PySH, denoted as AuSPy, as well as the LBL multilayers of (B) AuSPy(Pd/Fe-PVPTPy), (C) AuSPy(Pd/Fe-PVPTPy)₃, and (D) AuSPy(Pd/Fe-PVPTPy)₅.



Figure S6. Emission spectra for (A) TPyBr and (B) PVPTPy in the 0.085 mmol/L methanol solutions after addition of different concentrations of $Fe(BF_4)_2$. Inserted: Concentration of $Fe(BF_4)_2$ in the solutions.

	C(1s)	N(1s)	O(1s)	Cl(2p)	Pd(3d)	Fe(2p)	B(1s)	F(1s)
BPy modified (B)	284.8 (47.28%)	400.0 (1.99%)	532.6 (50.49%)	198.1 (0.24%)				
Pd-PVPTPy (D)	284.8 (53.53%)	400.1 (3.69%)	532.2 (39.27%)	198.2 (1.88%)	337.4,343.0 (1.63%)			
Pd/Fe-PVPTPy (F)	284.8 (44.95%)	400.1 (2.42%)	532.3 (49.06%)	197.8 (0.62%)	338.0,343.4 (1.32%)	708.7 (0.18%)	188.0 (1.37%)	685.1 (0.08%)

Table S1.Deconvolution of XPS Peaks.