Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

Layered double hydroxide nanoparticles embedded in biopolymer: A novel platform for electroanalytical determination of diazepam

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S. 1. Characterization of LDH nanoparticles

The powder X–ray diffraction (XRD) is a powerful technique for characterizing the structure of LDHs. The XRD pattern for Co–Al LDH was shown in Fig. S1 A. The XRD pattern of the synthesized LDH exhibits the characteristic diffraction peaks at 2θ=11.71°, 23.56°, 34.65°, 39.29°, 46.79°, which correspond to the reflections of (003), (006), (012), (015) and (018), respectively. It can be seen that Co–Al LDH exhibits the characteristic reflections of hydrotalcite–like compounds and no other crystalline phases are present, which is in agreement with the results reported by other researchers [1].

Fig. S1 B shows the FT–IR spectrum of Co–Al LDH. The broad band around 3444 cm^{-1} can be assigned to the stretching mode of hydroxyl groups of LDH layers and interlayer water molecules. The presence of water molecules is also responsible for the weak band at 1634 cm^{-1} (bending mode). A sharp and intense band at 1362 cm^{-1} is attributed to stretching vibration of interlayer carbonate ions. Other absorption bands below 800 cm^{-1} are due to the vibration of M–O and M–O–H in the brucite-like lattice [2, 3]. Therefore, XRD results and FTIR spectrum confirm that Co–Al LDH has been successfully synthesized.

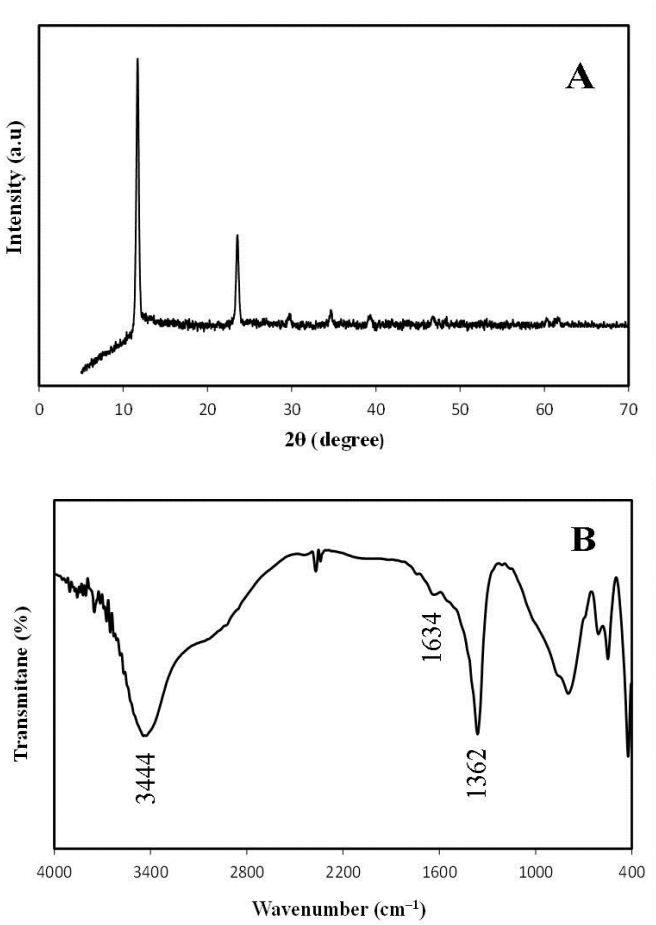

Fig. S1 (A) XRD pattern and (B) FT–IR spectrum of Co–Al LDH.

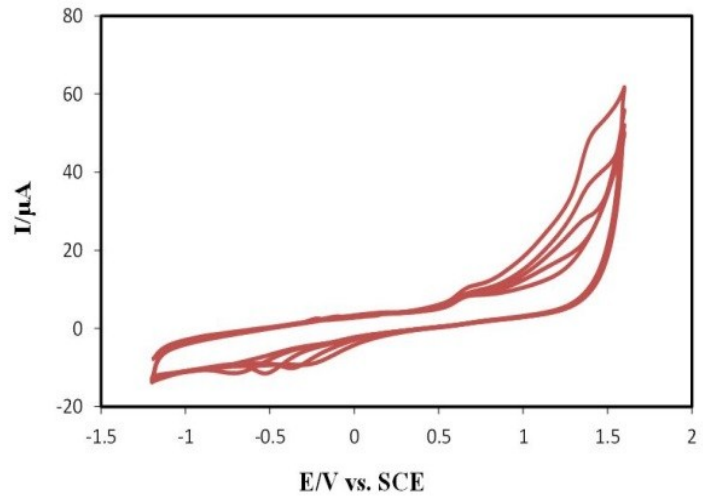

Fig. S2. CVs for the electropolymerization of Tyr in the potential range between –1.2 and +1.6 V. scan rate: 100 mV s^{-1}.

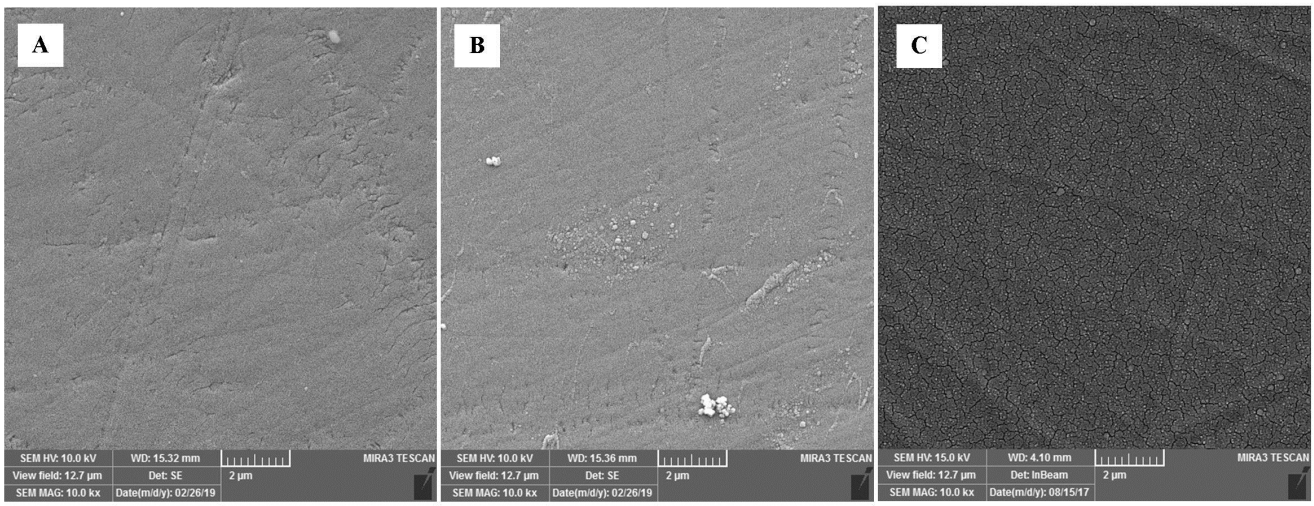

Fig. S3. FESEM images of poly(Tyr)/GCE fabricated under the different positive potential value for electropolymerization of Tyr on GCE (negative potential of –1.2 V) (A) +1.6 V (B) +1.8 V and (C) +2.5 V.

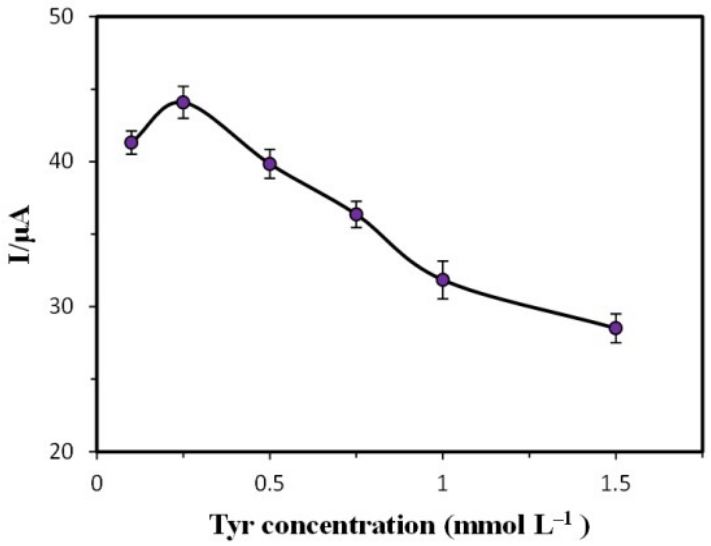

Fig. S4. Effect of Tyr concentration on the reduction peak current of DZ.

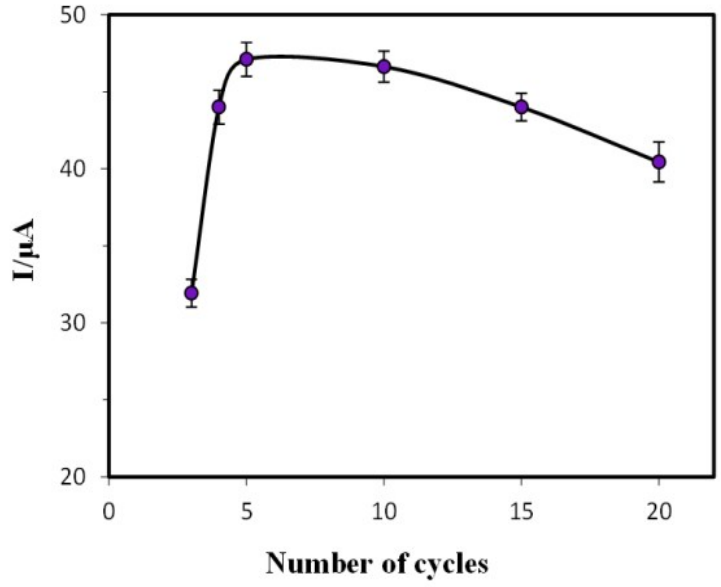

Fig. S5. Effect of the number of cycles of polymerization of Tyr on the reduction peak current of DZ.

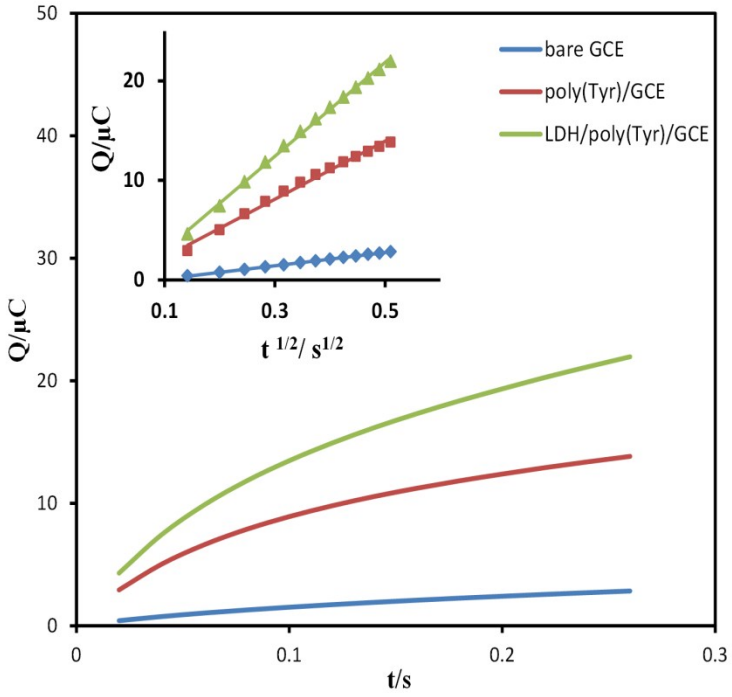

Fig. S6. Plot of Q–t curve of bare GCE, poly(Tyr)/GCE and LDH/poly(Tyr)/GCE in 0.1 mol L^{-1} KCl containing 1 mmol L^{-1} $K_3[Fe(CN)_6]$. Inset: Plot of Q–$t^{1/2}$ curve of bare GCE, poly(Tyr)/GCE and LDH/poly(Tyr)/GCE.

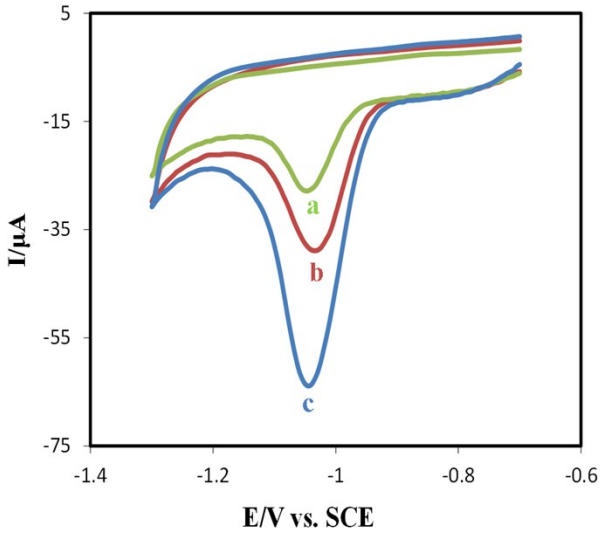

Fig. S7. CVs of 1.0×10^{-4} mol L^{-1} DZ on LDH/poly(Tyr)/GCE in 0.1 mol L^{-1} different supporting electrolytes (pH 6.0) (a) Britton Robinson buffer (b) acetate buffer (c) PBS ; Scan rate: 50 mV s^{-1}.

Table S1
Recovery test for DZ in human blood plasma samples.

Sample	Added (μM)	found (μM)	Recovery (%)	RSD (%)
Plasma 1	2.0	2.10	105	2.6
	4.0	3.94	98.5	1.8
Plasma 2	2.0	2.05	102.5	2.4
	4.0	4.20	105	3.1

References

1. S. Huang, G.-N. Zhu, C. Zhang, W. W. Tjiu, Y.-Y. Xia and T. Liu, *ACS Appl Mater Inter*, 2012, **4**, 2242-2249.
2. Z. P. Xu, G. Stevenson, C.-Q. Lu and G. Q. Lu, *J. Phys. Chem. B*, 2006, **110**, 16923-16929.
3. R. Amini and K. Asadpour-Zeynali, *New J. Chem.*, 2018, **42**, 2140-2148.