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## **Supplementary Data**

## For

Amperometric galectin-3 immunosensor based on gold nanoparticles functionalized graphitic carbon nitride nanosheets and core-shell Ti-MOF@COFs composites

Mehmet Lütfi Yola<sup>a\*</sup>, Necip Atar<sup>b</sup>

<sup>a</sup>Iskenderun Technical University, Faculty of Engineering and Natural Sciences, Department of Biomedical Engineering, Hatay, Turkey <sup>b</sup>Pamukkale University, Faculty of Engineering, Department of Chemical Engineering, Denizli, Turkey

\* To whom correspondence should be addressed:

E-mail: <u>mlutfi.yola@iste.edu.tr</u>

Tel.: +903266135600 Fax: +903266135613



**Fig. S1.** (A) UV-vis spectra of (a)  $g-C_3N_4$ , (b)  $g-C_3N_4$ @Au NPs composite, (B) Emission spectra (excitation at 360 nm) of (a)  $g-C_3N_4$ , (b)  $g-C_3N_4$ @Au NPs, (c) AuNPs, (C) FTIR spectra of (a)  $g-C_3N_4$ , (b)  $g-C_3N_4$ @Au NPs, (D) XRD pattern of  $g-C_3N_4$  and  $g-C_3N_4$ @Au NPs



**Fig. S2.** (A) XRD patterns of COFs, (B) Ti-MOF and Ti-MOF@COF composite, (C) FTIR spectra of COFs, Ti-MOF and Ti-MOF@COF composite



Fig. S3. (A) Survey XPS spectra of Ti-MOF@COF composite, (B) C1s, (C) N1s, (D) Ti2p binding energy spectra and (E)  $N_2$  adsorption-desorption of COFs, Ti-MOF and Ti-MOF@COF composite

# *Optimization for amperometric measurements pH effect of H*<sub>2</sub>*O*<sub>2</sub> *solution*

pH effect was investigated on immunosensor performance. The immunosensor response increased up to pH 7.0. Furthermore, highly acidic or alkaline medium damaged the immobilized protein. Hence, optimal pH was selected to be pH 7.0 (close to physiological pH) (Fig. S4A) (In the presence of 2.5 mM  $H_2O_2$ ).

#### Concentration effect of Ti-MOF@COF/anti-GL-3-Ab2 solution

The concentration of Ti-MOF@COFs/anti-GL-3-Ab<sub>2</sub> solution is important factor on the performance of amperometric immunosensor. The optimal and symmetrical peaks were observed up to 20.0 mg mL<sup>-1</sup>. Especially, after 20.0 mg mL<sup>-1</sup>, the optimal and symmetrical peaks decomposed and the signals decreased slightly or remained steady. Hence, the optimal concentration was selected as 20.0 mg mL<sup>-1</sup> (Fig. S4B) (In the presence of 2.5 mM H<sub>2</sub>O<sub>2</sub> in 0.1 M PBS, pH 7.0).

### *Immune reaction time effect*

When incubation time increased from 15 min to 45 min, peak current responses increase rapidly. After 45 min, immunosensor signals ( $\mu$ A) either remain constant or slightly diminish. Thus, optimal immune reaction time was selected to be 45 min (Fig. S4C) (In the presence of 2.5 mM H<sub>2</sub>O<sub>2</sub> in 0.1 M PBS, pH 7.0).

## Concentration effect of $H_2O_2$

In this study, different  $H_2O_2$  concentrations were tried for obtaining optimal immunosensor signals (Fig. S4D). When  $H_2O_2$  concentration gradually increased from 1.5 mM to 2.5 mM, the peak current gradually increased. After 2.5 mM  $H_2O_2$ , peak current decreased inversely. Due to overdose of  $H_2O_2$  catalyst, the catalytic reaction was inhibited and the overdose of  $H_2O_2$  catalyst affected the activity of the proteins. Thus, the optimal signals were obtained in 2.5 mM  $H_2O_2$  in 0.1 M PBS (pH 7.0).



**Fig. S4.** Effect of (A) pH, (B) Ti-MOF@COFs/anti-GL-3-Ab<sub>2</sub> solution concentration, (C) Immune reaction time, (D)  $H_2O_2$  concentration (antigen galectin-3 concentration: 0.001 ng mL<sup>-1</sup>, n = 6)

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Plasma sample	Added GL-3	Found GL-3	Recovery
	(ng mL <sup>-1</sup> )	(ng mL <sup>-1</sup> )	(%)
<sup>a</sup> Sample (1)	0.500	$0.504\pm0.002$	-
<sup>b</sup> Sample (2)	Sample(1) + 0.100	$0.604 \pm 0.001$	$100.67 \pm 0.07$
<sup>c</sup> Sample (3)	Sample(1) + 0.200	$0.703 \pm 0.001$	$100.43 \pm 0.03$
<sup>d</sup> Sample (4)	Sample(1) + 0.300	$0.798 \pm 0.004$	$99.75 \pm 0.06$

<b>Table S1.</b> The recovery of	fGL-3	(n=6)
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<sup>a</sup>containing 0.500 ng mL<sup>-1</sup> GL-3, 100.0 ng mL<sup>-1</sup> GLU, 100.0 ng mL<sup>-1</sup> DOP, 100.0 ng mL<sup>-1</sup> THR, 100.0 ng mL<sup>-1</sup> BSA

<sup>b</sup>containing 0.500 + 0.100 ng mL<sup>-1</sup> GL-3, 100.0 ng mL<sup>-1</sup> GLU, 100.0 ng mL<sup>-1</sup> DOP, 100.0 ng mL<sup>-1</sup> THR, 100.0 ng mL<sup>-1</sup> BSA

<sup>c</sup> containing 0.500 + 0.200 ng mL<sup>-1</sup> GL-3, 100.0 ng mL<sup>-1</sup> GLU, 100.0 ng mL<sup>-1</sup> DOP, 100.0 ng mL<sup>-1</sup> THR, 100.0 ng mL<sup>-1</sup> BSA

<sup>&</sup>lt;sup>d</sup>containing 0.500 + 0.300 ng mL<sup>-1</sup> GL-3, 100.0 ng mL<sup>-1</sup> GLU, 100.0 ng mL<sup>-1</sup> DOP, 100.0 ng mL<sup>-1</sup> THR, 100.0 ng mL<sup>-1</sup> BSA