

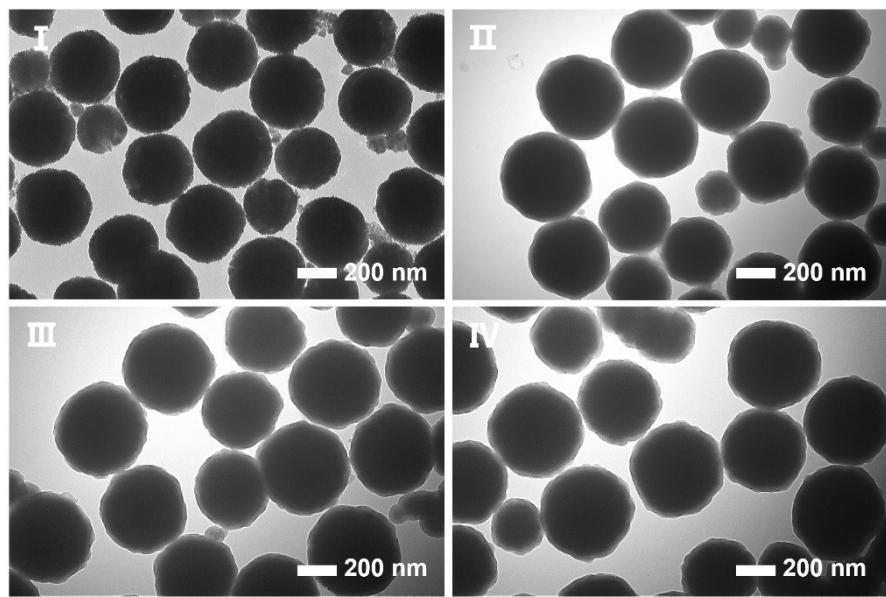
# Supporting Information

## **Chemiluminescence Immunosensor for Biomarker Detection Based on Boronic Acid-Modified Magnetic Composite Microspheres**

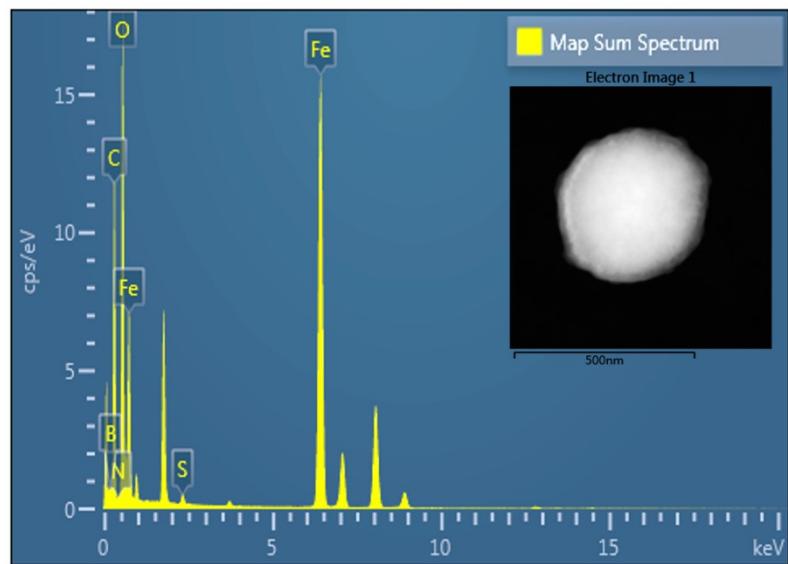
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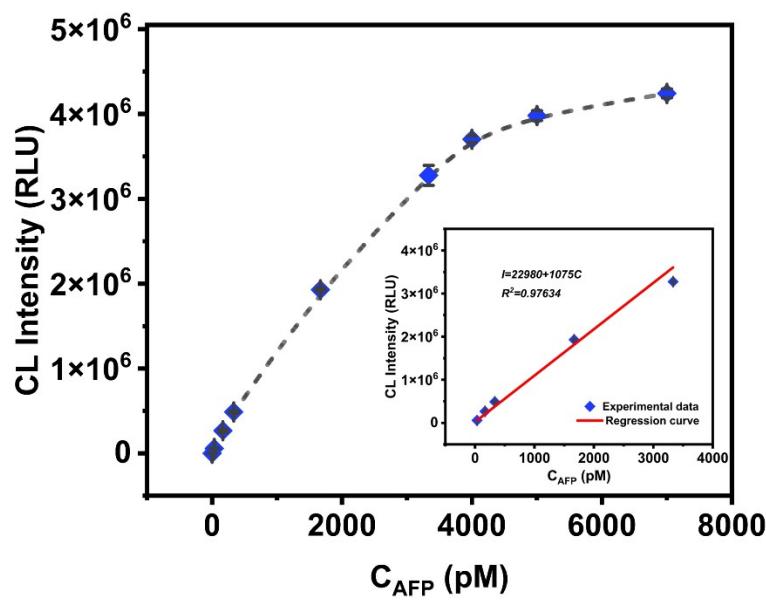
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**Fig. S1** TEM images of (I) MSP, (II) MSP@PGMA, (III) MSP@PGMA-SH and (IV) MSP@PGMA-PBA (scale bar: 200 nm).



**Fig. S2** SEM-EDS spectrum of MSP@PGMA-PBA microspheres.



**Fig. S3** The relationship between the CL intensity and AFP concentration in serum, the inset illustrating their linear relationship.

**Table S1.** The elemental content of Fe, S and B in MSP@PGMA-PBA microspheres.

Sample	Test element	Element content (mg/kg)
MSP@PGMA-PBA	Fe	530076
	S	5553
	B	1211

**Table S2.** The chemiluminescence intensity of MSP@PGMA-PBA based immunosensor upon repeated testing of 20 times on blank samples without AFP.

Number of tests	RLU
1	686
2	674
3	672
4	679

5	667
6	698
7	699
8	672
9	676
10	680
11	682
12	684
13	686
14	688
15	690
16	692
17	694
18	696
19	698
20	672

**Table S3.** Performance comparison of the as-prepared biosensor with other sensors.

Substrate materials	Method	Type of target	Linear range	LOD	Detection time	Ref
MS	Chemiluminescence	miRNA-21	0.01-10000 pM	60 fM	90 min	1
G4 DNAzyme	Chemiluminescence	ATP	2-2000 μM	84.3 nM	>75 min	2
CdTe-Ru@SiO <sub>2</sub>	Electrochemistry	ATP	10-5000 nM	6.68 nM	90 min	3
Au NPs	Electrochemistry	ATP	30-500 nM	10 nM	370 min	4
AuNPs/U-GDY-CP	Self-powered electrochemical	miRNA-155	0.1-10000 fM	0.15 fM	130 min	5
SGDY/AuNPs-CP	Self-powered electrochemical	miRNA-141	1-10000 fM	3.0 fM	overnight	6
AuNPs/GDY-CP	Self-powered electrochemical	miRNA-21	0.1-100000 fM	0.034 fM	140 min	7
U-GDY/ AuNPs--	Self-powered	miRNA-141	0.5-10000 fM	0.14 fM	180 min	8

CP	electrochemical					
AuNPs/U-GOD-CP	Self-powered electrochemical	miRNA-21	0.1–10000 fM	0.04 fM	overnight	9
MSP@PGMA-PBA	chemiluminescence	AFP	167-6667 pM	8 fM	90 min	This work

\*MS: magnetic microspheres; G4: quadruplex; GDY: graphdiyne; U-GDY: ultra-thin graphdiyne; SGDY: sandwich graphdiyne; GOD: glucose-oxidase; CP: carbon paper; ATP: adenosine triphosphate

**Table S4.** Performance comparison of the MSP@PGMA-PBA with other magnetic bead system.

Sample	Carboxyl magnetic bead	Tosyl magnetic bead	Our system
Coupling principle	Amidation reaction between carboxyl and amino	Amidation reaction between p-toluenesulfonyl chloride and amino/sulphydryl	Phenylborate-catechol complexation
Group activation treatment	Need (RT/0.5-2h)	No need	No need
Catalyst/reaction promoter	EDC/EDC-NHS (participatory response) (RT/0.5-2h)	Ammonium sulfate (not involved in the reaction) (37°C/24h)	No need
Coupling temperature	RT	37 °C	37 °C
Coupling time	1-3h	18-24h	1-2h
Coupling pH	5 ~ 6.5	7 ~ 9.5	7.5 ~ 8
Blocking temperature	RT	37 °C	37 °C
Blocking time	1-3h	6-10h	1-3h

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