Supplementary Information (SI) for Analytical Methods.

Supporting Information

Convenient in situ self-assembled formation of dual-functional Ag/MXene nanozymes for efficient chemiluminescence sensing

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EXPERIMENTAL SECTION

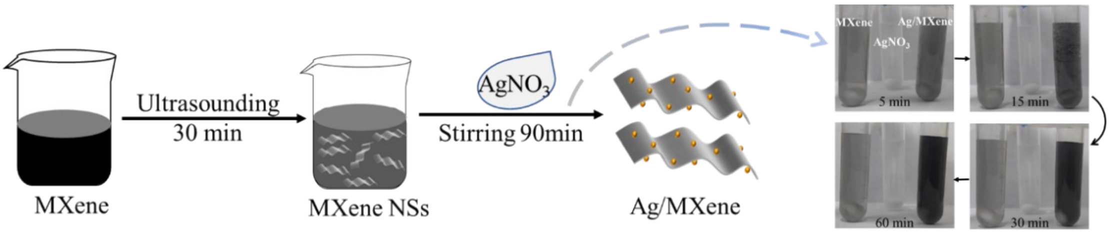
Reagents

Ti_3AlC_2 was purchased from Lai Zhou Kaiene Ceramic Material Co., LTD. HF, H_2O_2, NaOH were obtained from Xi long Chemical Co., LTD. Citric acid, glucose and urea were purchased from Shanghai Sinopharm Group Chemical Reagent Co., LTD. Luminol was purchased from Beijing Balingway Technology Co., LTD. Reduced Glutathione, Uricase (20 U/mg), Calcium acetate monohydrate ($C_4H_8CaO_5$), uric acid, cysteine, lysine, tryptophan, threonine, glutamic acid, arginine, alanine, valine, glycine, isoleucine, aspartic acid, leucine, methionine, hydroxylamine hydrochloride, thiourea, ascorbic acid were purchased from Shanghai Maclin Biochemical Technology Co., LTD. NaCl, KCl, $MgCl_2$, $AgNO_3$ were purchased from Shanghai Aladdin Biochemical Technology Co., LTD. All reagents are analytically pure and used directly. The ultrapure water used in all experiments was purified by Millipore Synergy System (18.24 $M\Omega\ cm^{-1}$, Milli-Q, Millipore).

Apparatus and Characterization

All chemiluminescence (CL) signals were collected using an ultra-weak luminescence analyzer (BPCL-1-TGC, Institute of Biophysics, Chinese Academy of Sciences, China). UV–vis absorption analysis was performed on the UV-vis spectrometer (TU-1901, Puxi General Instrument Co., Ltd., China Beijing). A scanning electron microscope (SEM, TESCAN MIRA LMS, Czech Republic) was used to characterize the formation of Ag/MXene. A transmission electron microscope (TEM, JEM-2010HR, JEOL, Japan), with a 200 kV acceleration voltage was used to measure the size. X-ray powder diffraction spectra (XRD) of the Ag/MXene powders were characterized by Cu Kα radiation (λ = 1.5418 Å) using an x-ray diffractometer (D8-FOCUS, Bruker, Karlsruhe, Germany) with diffraction angles ranging from 5° to 90°. The elemental valence states of Ag/MXene were determined by X-ray photoelectron spectroscopy (XPS) using Thermo Scientific K-Alpha (EscaLab250, Thermo Fisher Scientific, America). The 300E electron spin resonance (EPR) spectrometer (Bruker EMXplus, Germany) was employed to record the EPR spectra at room temperature.

Supplementary Figures



Scheme S1. Preparation process of Ag/MXene composites.

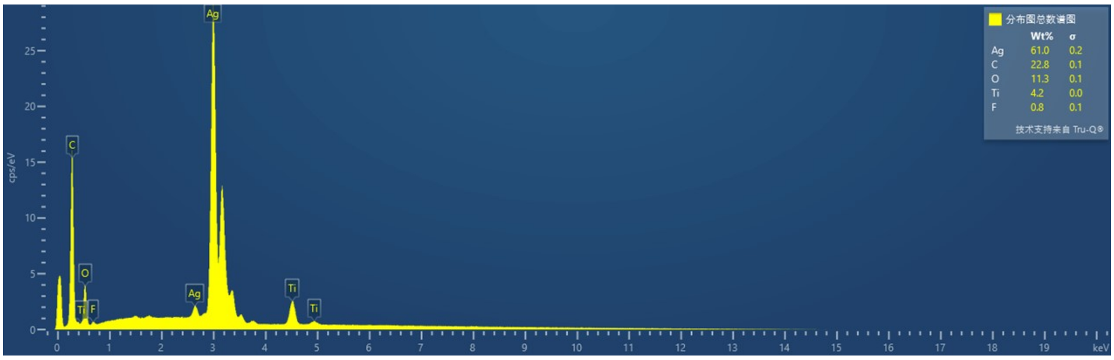


Figure S1. The EDS images of Ag/MXene composites.

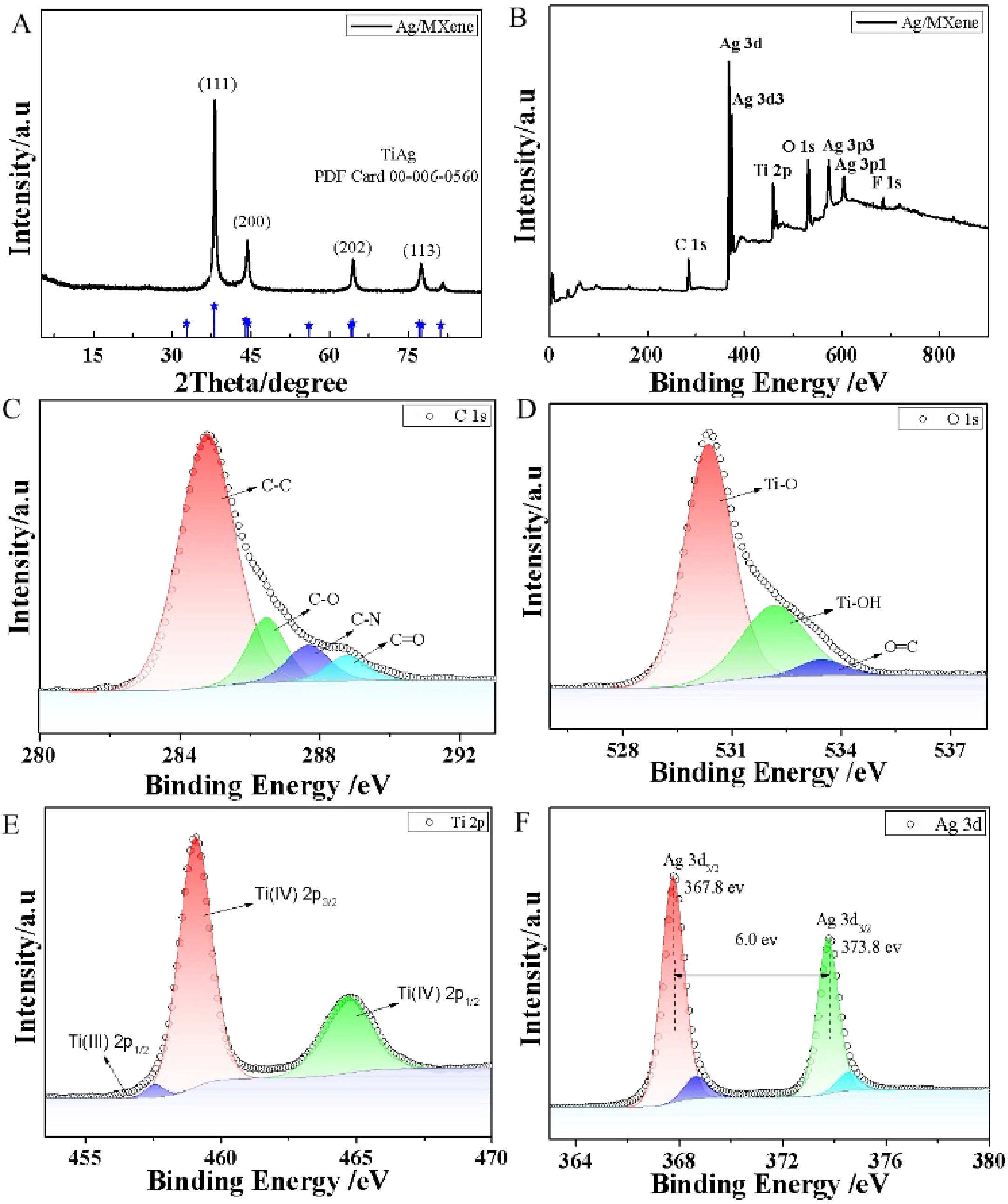


Figure S2. XRD image for Ag/MXene composites (A). Full-scan XPS spectrum of Ag/MXene (B). High-resolution XPS spectra of C 1s (C), O 1s (D), Ti 2p (E) and Ag 3d (F) for Ag/MXene.

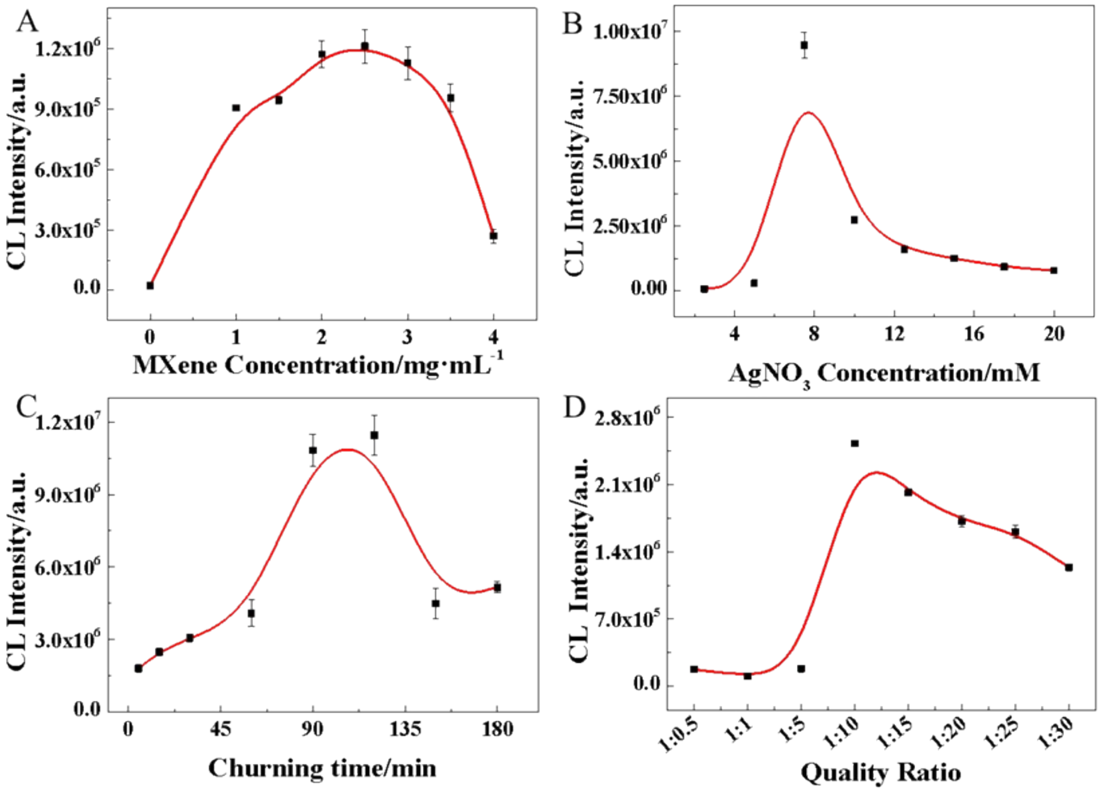


Figure S3. Optimization of Ag/MXene synthesis conditions, including MXene concentration (A), $AgNO_3$ concentration (B), the churning time (C) and the mixing volume ratio between MXene solution and $AgNO_3$ solution (D). Reaction condition: luminol, 1 μM, 50 μL; H_2O_2, 10 mM, 50 μL; Ag/MXene, 10 times diluted, 50 μL.

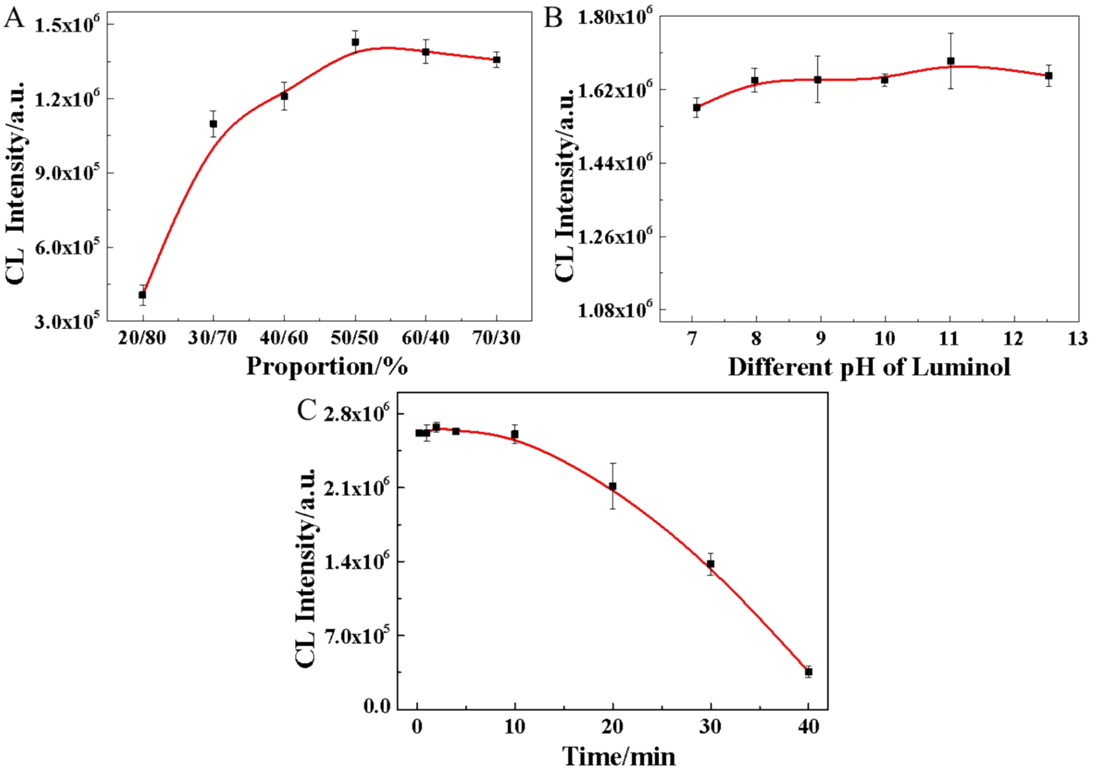
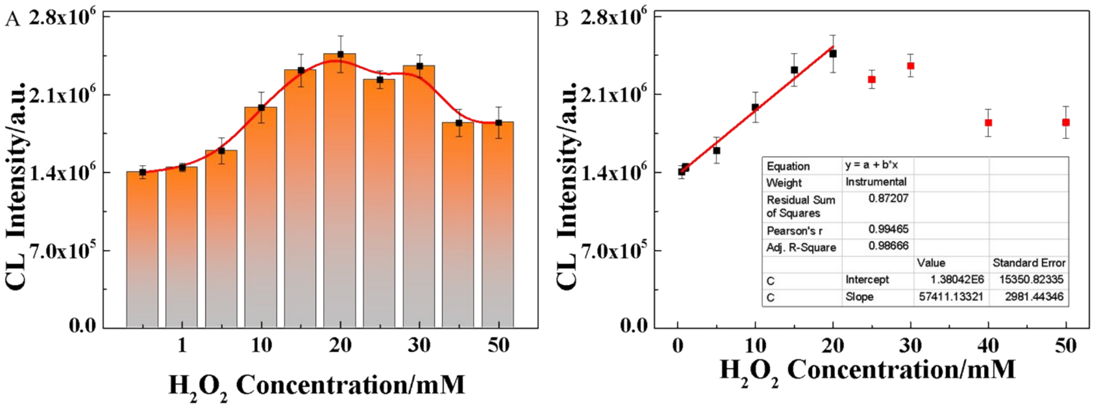


Figure S4. Ag/MXene-luminol-H_2O_2 measurement condition optimization, including substrates luminol and H_2O_2 volume ratio (A), pH of luminol (B) and luminol-H_2O_2 reaction time (C).



Equation	y = a + b*x		
Weight	Instrumental		
Residual Sum of Squares	0.87207		
Pearson's r	0.99465		
Adj. R-Square	0.98666		
		Value	Standard Error
C	Intercept	1.38042E6	15350.82335
C	Slope	57411.13321	2981.44346

Figure S5. Sensing analysis of H_2O_2 concentration by Ag/MXene composites in chemiluminescence luminol-H_2O_2 system. The error bars represented the standard deviation of three replicates.

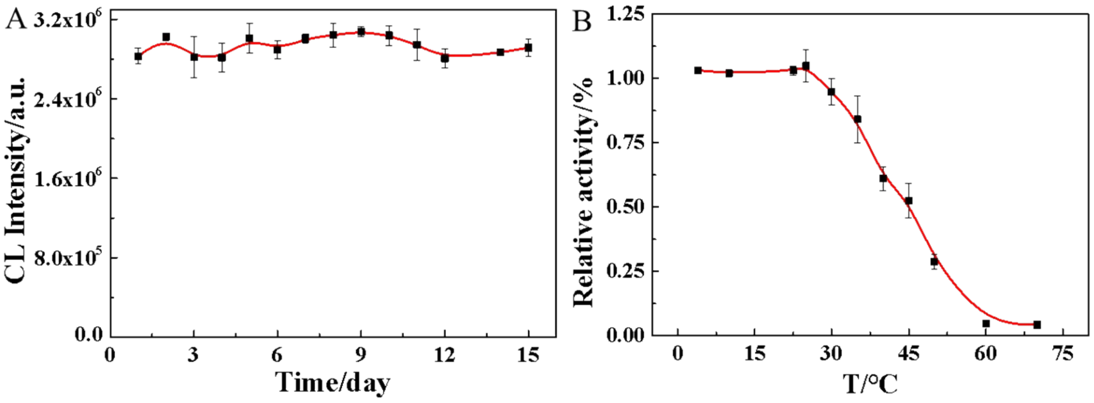


Figure S6. The time stability (A) and temperature stability (B) tests of Ag/MXene composites.

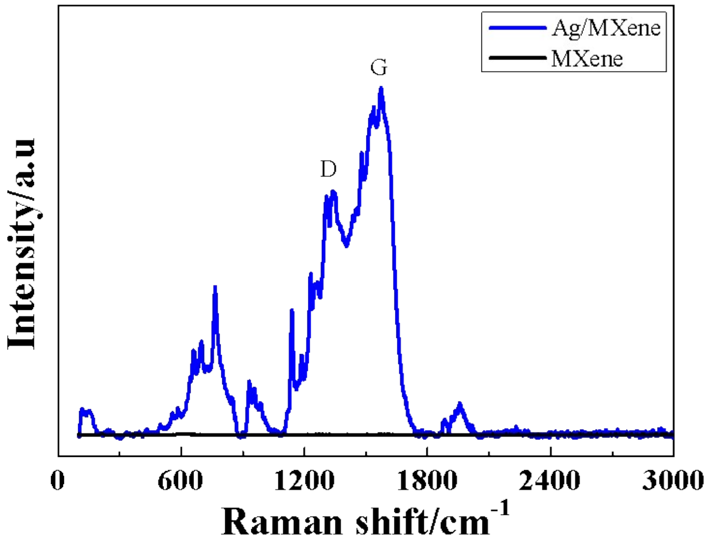


Figure S7. Raman spectra of Ag/MXene composites.

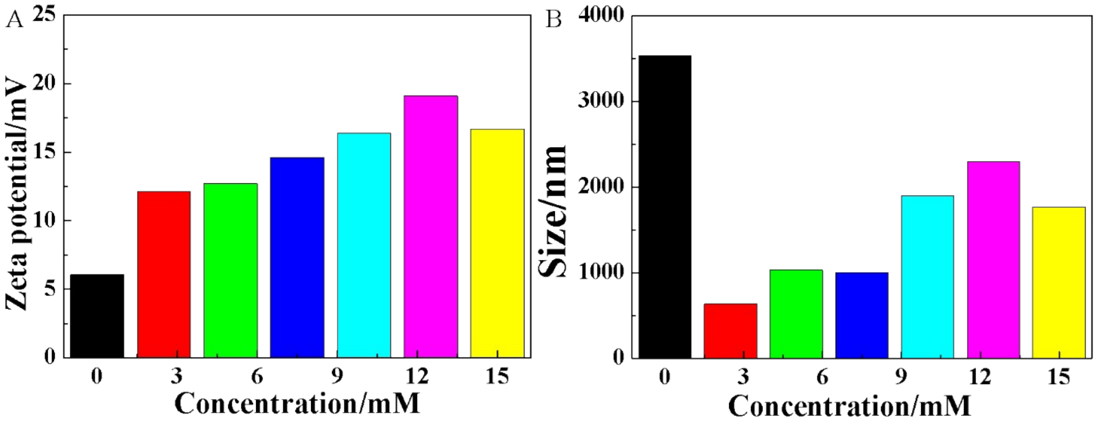


Figure S8. (A) Zeta potential and (B) particle size analysis between different concentrations of $AgNO_3$ and MXene.

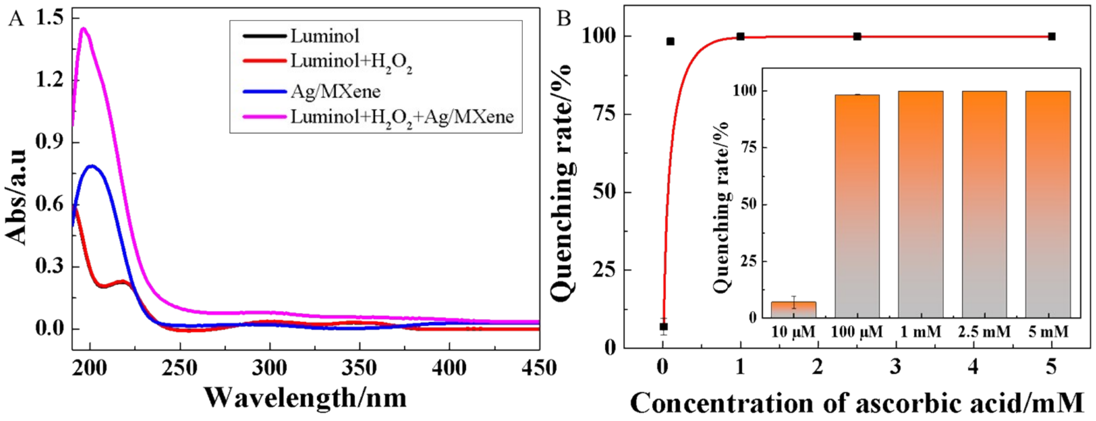


Figure S9. UV-vis absorption spectra of luminol, luminol-H_2O_2, Ag/MXene and Ag/MXene-luminol-H_2O_2 (A). Scavenging experiment of reactive oxygen species with ascorbic acid (B).

Table S1. Application with the proposed method to detect human Serum spiked with varied GSH amounts

samples	Spiked (μM)	Found (μM)	Recovery (%)	RSD (%, n=3)
1	0.5	0.56	10.68	1.94
	5	5.59	111.80	3.08
	10	10.68	106.80	5.55
2	0.5	0.48	95.72	1.56
	5	4.94	98.70	5.68
	10	10.42	104.20	5.28

Table S2. Application with the proposed method to detect human Serum spiked with varied UA amounts

samples	Spiked (μM)	Found (μM)	Recovery (%)	RSD (%, n=3)
1	10	10.49	104.90	1.88
	15	14.63	97.55	5.87
	20	19.52	97.59	5.12
2	10	10.6	106.00	4.07
	15	16.25	108.32	4.56
	20	21.67	108.34	1.23