

*Supplementary Information*

# Multilayered MoAlB@MBene using Mild Microwave-Assisted Etching and their Optical Properties

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## **Chemicals and Reagents Used**

Hydrochloric acid (HCl) with 37 % (v/v) concentration from Merk; Sodium hydroxide (NaOH, 0.1 M), 30 % (v/v) water solution of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) from chempur, double distilled water was used. Molybdenum boride (MoB) and aluminum (#325 mesh; 44 μm) powders were purchased from Alfa Aesar for MoAlB synthesis. All the chemicals were used without further purification.

## **Synthesis of MAB phase (MoAlB)**

The MoAlB MAB phase was synthesized using direct mixing of precursor powders i.e. Molybdenum boride (MoB) and aluminum (#325 mesh; 44 μm) powders. The MoB and aluminum powders were mixed in a ratio of 1:1.2. After mixing these two powders, it was pressed and heated in a Turbula T2F mixer for 3 h at 56 rpm. For mixing media 10 mm yttria-stabilized zirconia balls were used. After that, mixed powders were pressed into 12 g pellets. It was heated at 750 °C for 2 h, then 1500 °C for another 2 h, at a heating rate of 10 °C/min in a tube furnace under an argon atmosphere (with flow rate 100 standard cm<sup>3</sup>/min, SCCM). The obtained solid samples were ultimately cooled to room temperature and ground down to #325 mesh.

## **Detailed protocol for the etching of Al from the MoAlB phase**

### **MAB@MBene (Without pre-treatment)**

Initially, 250 mg of MoAlB was added slowly into 20 mL of 0.6 M HCl solution with constant stirring, followed by sonication for 2 minutes. After that 20 μl of H<sub>2</sub>O<sub>2</sub> (30%) solution was added dropwise and closed the lid of the Teflon container. Once the microwave is fitted, the reaction starts as per the designed program (Program: temperature: 150 °C, reaction time 120 min (40 min each in 3 cycles), and microwave power: 480W). After completion, the supernatant was navy blue, and the precipitate was black. After five washing using centrifugation (3000 RPM for 4 min), the reaction mixture pH becomes 6. The precipitate was collected and used further for characterization.

### **Base-MAB@MBene (With NaOH pre-treatment)**

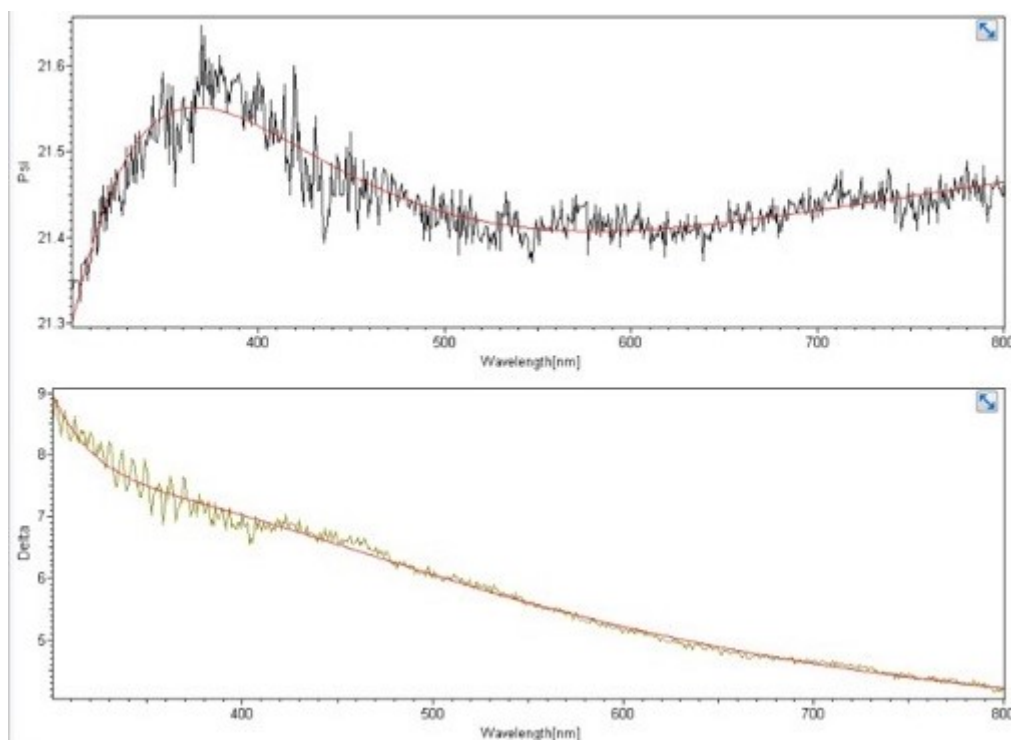
**Pretreatment:** Firstly, 250 mg of MoAlB was added into 0.1 M NaOH and stirred for 2h, then washed with distilled water till pH became 6.

**Microwave treatment:** The washed precipitate was slowly transferred into 20 mL of 0.6 M HCl with constant stirring, followed by sonication for 2 min. After that, 250  $\mu$ l of H<sub>2</sub>O<sub>2</sub> (30%) was added dropwise and closed the lid of the Teflon container. Once the microwave was fitted, the reaction started as per the designed program (Program: Temperature: 150 °C, reaction time 120 min (40 min each in 3 cycles), and microwave power 480 W). After completion, the supernatant was yellow, and the precipitate was black. After five washing using centrifugation (3000 RPM for 4 min), the reaction mixture pH becomes 6. The black precipitate was collected and used further for characterization.

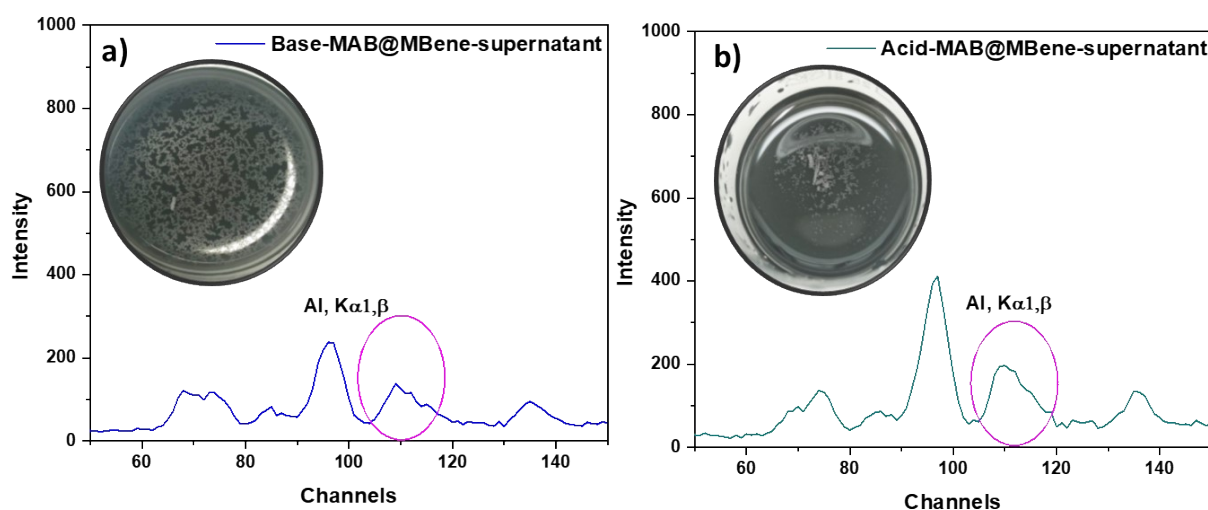
#### **Acid-MAB@MBene (With HCl pre-treatment)**

**Pretreatment:** Initially, 250 mg of MoAlB was stirred in 0.1 M HCl for 2h, then washed with distilled water till pH became 6.

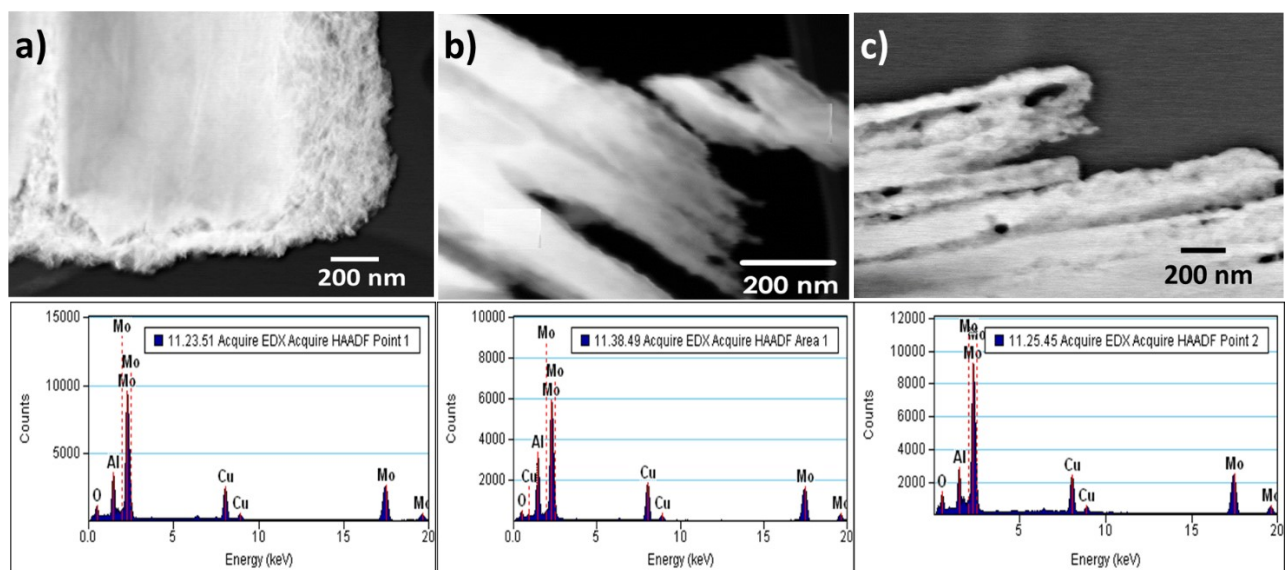
**Microwave treatment:** The washed precipitate was slowly transferred into 20 mL of 0.6 M HCl with constant stirring, followed by sonication for 2 min. After that, 250  $\mu$ l of H<sub>2</sub>O<sub>2</sub> (30%) solution was added dropwise and closed the lid of the Teflon container. Once the microwave was fitted, the reaction started as per the designed program (Program: Temperature: 150 °C, reaction time 120 min (40 min each in 3 cycles), and microwave power 480 W). After completion, the supernatant was Yellow, and the precipitate was black. After five washing using centrifugation (3000 RPM for 4 min), the reaction mixture pH becomes 6 (measured using pH Paper). The black precipitate was collected and used further for characterization.



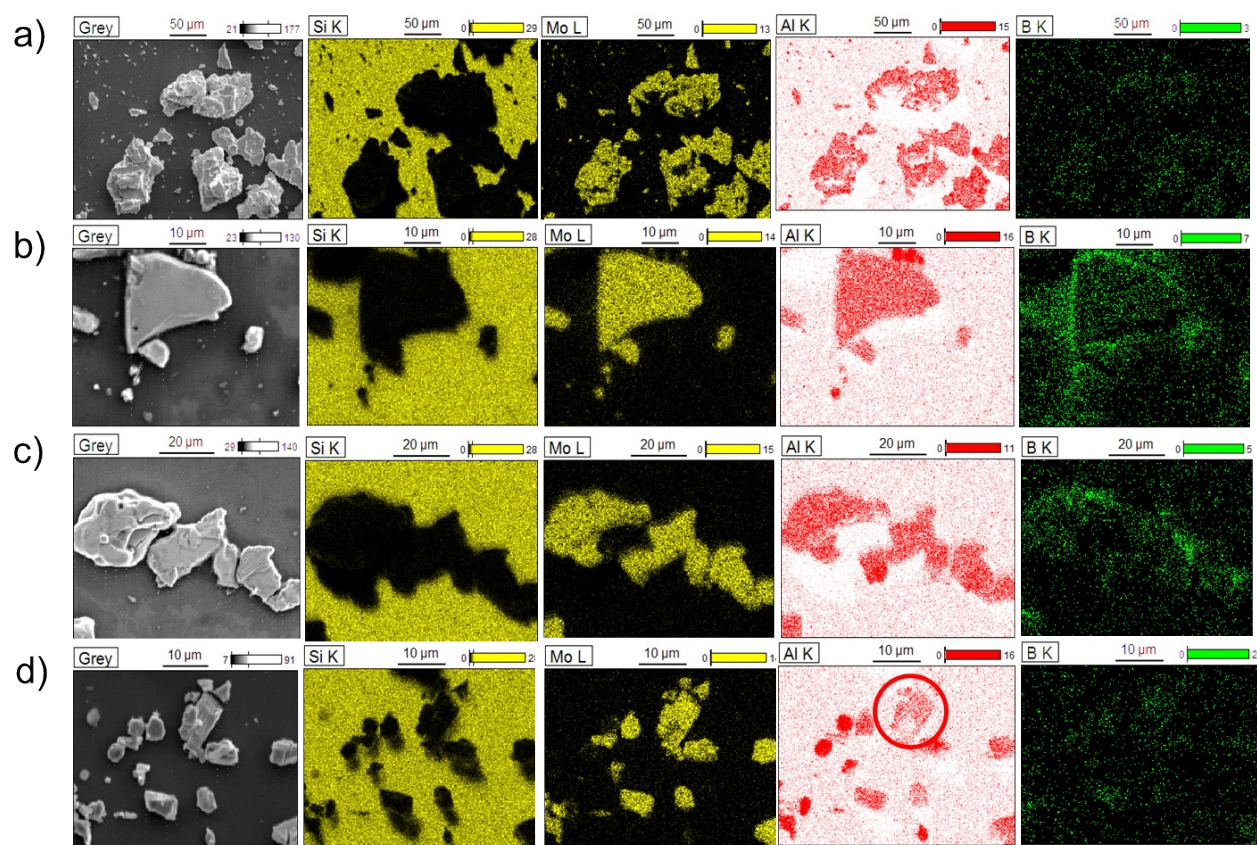
**Figure S1.** The  $\Psi$  and  $\Delta$  at the incident angle of  $45^\circ$  of the representative sample (MAB@MBene)



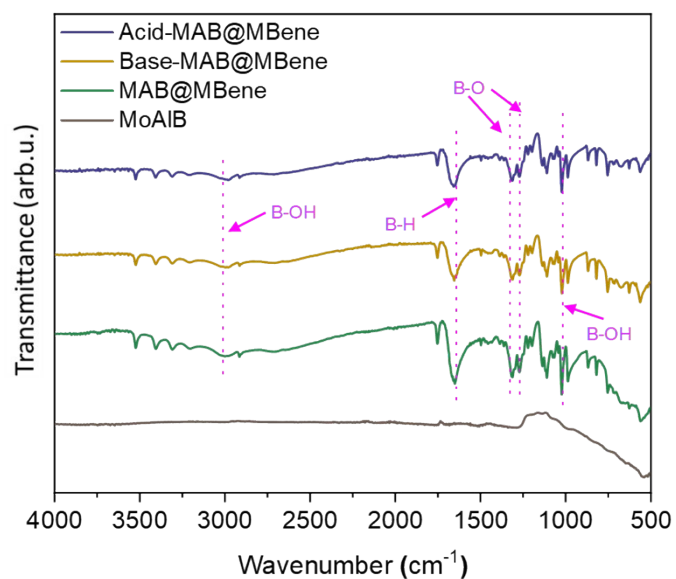
**Figure S2** XRF spectra of Al  $K\alpha$  peak for the supernatant after pre-treatment (a) 0.1 M NaOH and (b) 0.1 M HCl. Inset is the digital photograph of the respective reaction mixture.



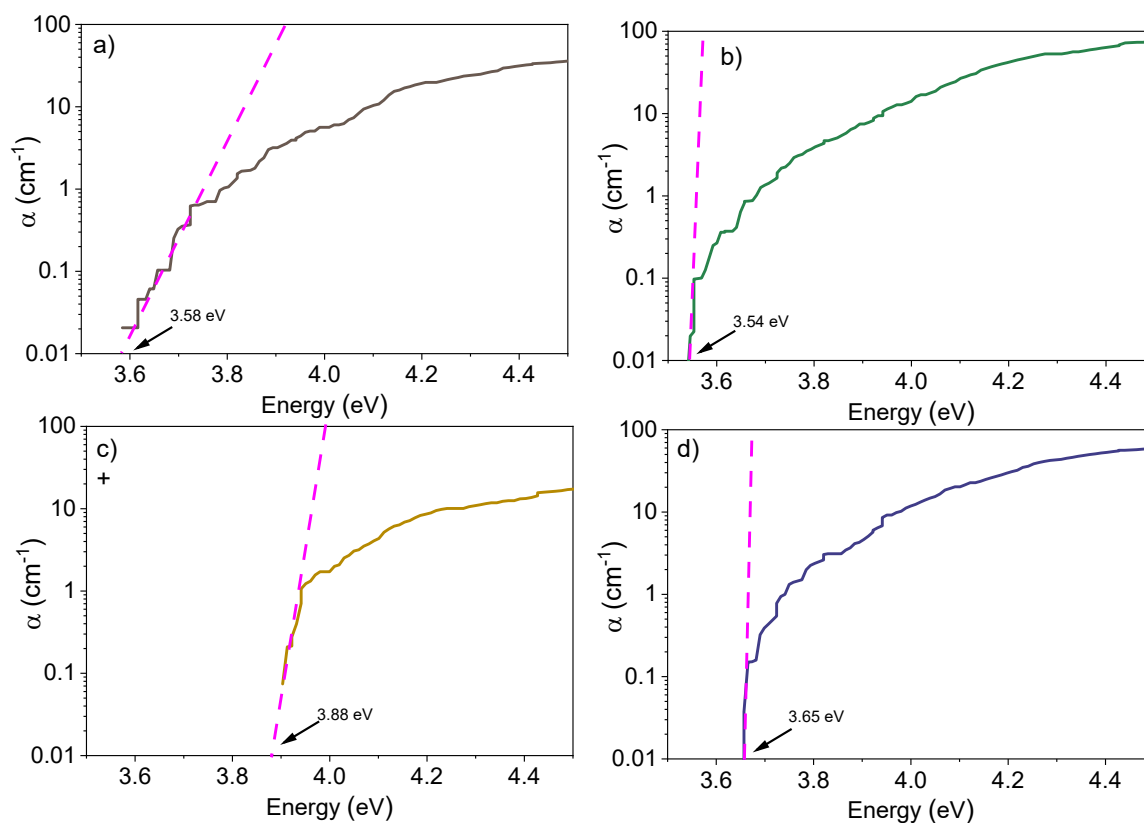
**Figure S3** TEM images and corresponding EDS of (a) MAB@MBene, (b) Base-MAB@MBene, and (c) Acid-MAB@MBene.



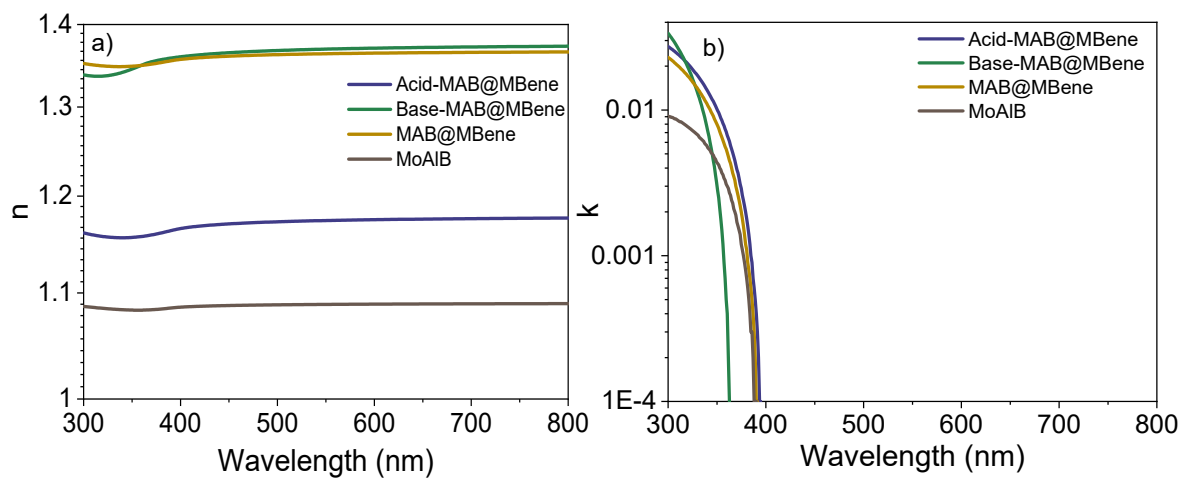
**Figure S4** SEM images and elemental mapping of (a) MoAlB, (b) MAB@MBene, (c) Base-MAB@MBene, and (d) Acid-MAB@MBene. The circled area is a highlight of the accordion-like structure



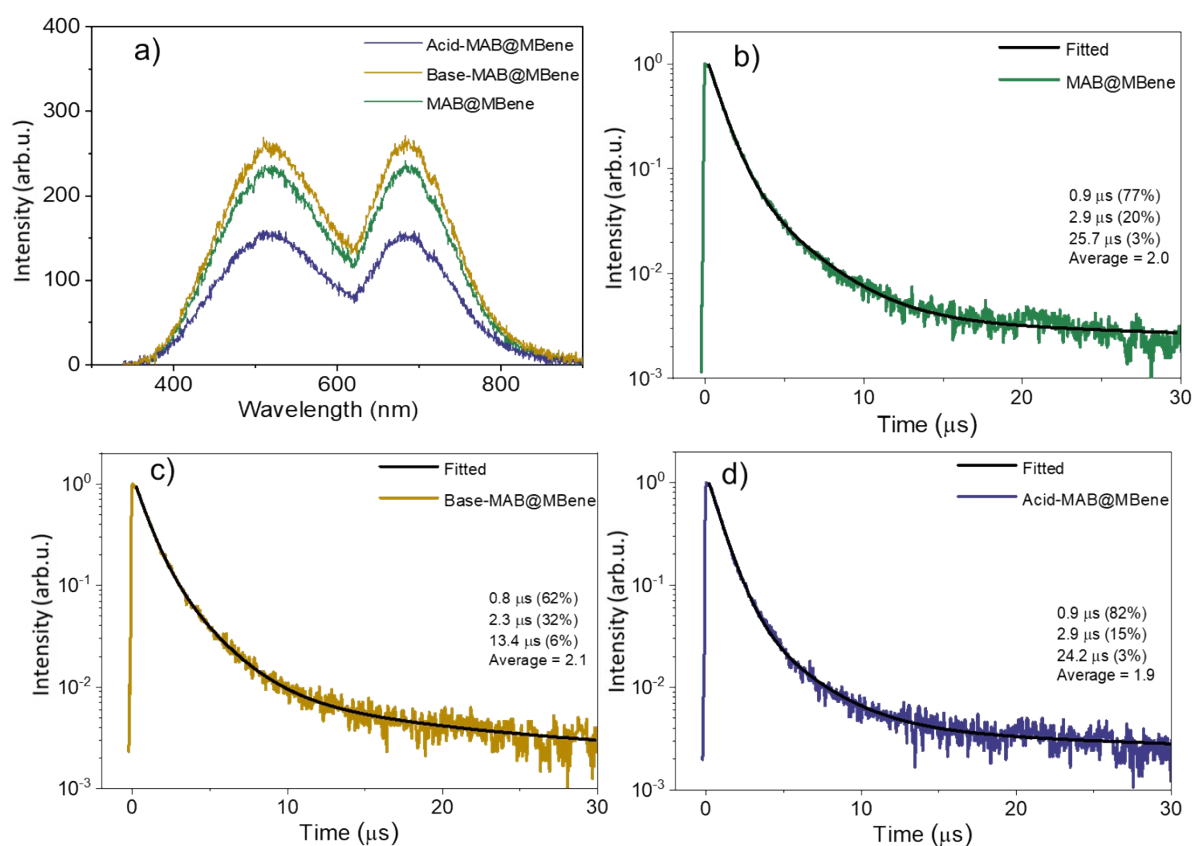
**Figure S5** FT-IR spectra of MoAIB, MAB@MBene, Base-MAB@MBene and Acid-MAB@MBene.



**Figure S6.** The estimated bandgap is based on the absorption spectra of (a) MoAIB, (b) MAB@MBene, (c) Base-MAB@MBene, and (d) Acid-MAB@MBene.



**Figure S7.** a) Refractive index ( $n$ ) and b) extinction coefficients ( $k$ ) of MoAIB, MAB@MBene, Base-MAB@MBene, and Acid-MAB@MBene.



**Figure S8** a) The photoluminescence measurements of MAB@MBene, Base-MAB@MBene and Acid-MAB@MBene. Steady-state and time-resolved photoluminescence measurements of

b) MAB@MBene, c) Base-MAB@MBene, and d) Acid-MAB@MBene with emission above 600 nm. All measurements were excited at 266 nm and room temperature.

**Table S1.** Results of the Elemental composition of the MoAlB sample using EDS

Element	Weight % [ $\pm$ Error]	Atom % [ $\pm$ Error]
B	1.25 $\pm$ 0.10	2.46 $\pm$ 0.19
Al	4.30 $\pm$ 0.06	4.87 $\pm$ 0.07
Si	77.05 $\pm$ 0.21	86.93 $\pm$ 0.23
Mo	17.40 $\pm$ 0.15	5.74 $\pm$ 0.05

Mo:Al:B atomic ratio 1: 0.85: 0.43

**Table S2.** Results of the Elemental composition of the MAB@MBene sample using EDS

Element	Weight % [ $\pm$ Error]	Atom % [ $\pm$ Error]
B	4.24 $\pm$ 0.12	8.53 $\pm$ 0.24
Al	3.93 $\pm$ 0.06	4.50 $\pm$ 0.07
Si	68.98 $\pm$ 0.19	79.30 $\pm$ 0.22
Mo	22.85 $\pm$ 0.22	7.67 $\pm$ 0.07

Mo:Al:B atomic ratio 1: 0.96: 1.11

**Table S3.** Results of the Elemental composition of the Base-MAB@MBene sample using EDS

Element	Weight % [ $\pm$ Error]	Atom % [ $\pm$ Error]
B	2.26 $\pm$ 0.11	4.82 $\pm$ 0.23
Al	2.11 $\pm$ 0.06	3.22 $\pm$ 0.08
Si	67.78 $\pm$ 0.19	82.04 $\pm$ 0.23
Mo	27.85 $\pm$ 0.18	9.92 $\pm$ 0.06

Mo:Al:B atomic ratio 1: 0.32: 0.48



**Table S4.** Results of the Elemental composition of the Acid-MAB@MBene sample using EDS

Element	Weight % [ $\pm$ Error]	Atom % [ $\pm$ Error]
B	2.75 $\pm$ 0.22	5.14 $\pm$ 0.42
Al	1.36 $\pm$ 0.14	2.52 $\pm$ 0.16
Si	83.92 $\pm$ 0.40	89.60 $\pm$ 0.42
Mo	11.97 $\pm$ 0.44	5.44 $\pm$ 0.14

Mo:Al:B atomic ratio 1: 0.46: 0.94

**Table S5. XPS peak component fitting analysis of MAB@MBene.**

Element	Element at%	Binding energy (eV)	Component name	Component at%
Mo 3d 5/2 (3d 3/2)	0.7	231.3 (234.5)	Mo <sub>2</sub> O <sub>5</sub>	0.5
		232.5 (235.7)	MoO <sub>3</sub>	0.2
Al 2p	0.1	74.2	Al <sub>2</sub> O <sub>3</sub>	0.1
B 1s	0.2	192.8	B <sub>2</sub> O <sub>3</sub>	0.2

**Table S6. XPS peak component fitting analysis of Base-MAB@MBene.**

Element	Element at%	Binding energy (eV)	Component name	Component at%
Mo 3d 5/2 (3d 3/2)	1.3	231.1 (234.3)	MoCl <sub>5</sub> /Mo <sub>2</sub> O <sub>5</sub>	0.5
		232.5 (235.8)	MoO <sub>3</sub>	0.8
Al 2p	0.5	74.5	Al <sub>2</sub> O <sub>3</sub>	0.1
		75.4	AlO(OH)	0.3
		76.4	AlCl <sub>3</sub>	0.1
B 1s	0.4	193.0	B <sub>2</sub> O <sub>3</sub>	0.4

**Table S7. XPS peak component fitting analysis of Acid-MAB@MBene.**

Element	Element at%	Binding energy (eV)	Component name	Component at%
Mo 3d 5/2 (3d 3/2)	0.9	230.7 (233.9)	MoO <sub>2</sub>	0.2
		232.1 (235.3)	MoO <sub>3</sub>	0.7
Al 2p	0.3	73.9	Al <sub>2</sub> O <sub>3</sub>	0.1
		76	AlCl <sub>3</sub>	0.2
B 1s	-	193.1	B <sub>2</sub> O <sub>3</sub>	-

**Table S8. Extracted parameters from the fitting of TRPL curves**

<b>Samples</b>	<b><math>\tau_1</math> (<math>\mu\text{s}</math>)</b>	<b><math>C_1</math> (%)</b>	<b><math>\tau_2</math> (<math>\mu\text{s}</math>)</b>	<b><math>C_2</math> (%)</b>	<b><math>\tau_3</math> (<math>\mu\text{s}</math>)</b>	<b><math>C_3</math> (%)</b>	<b><math>\tau_{\text{avg}}</math> (ns)</b>
MAB@MBene	0.9	77	2.9	20	25.7	3	2.0
Base-MAB@MBene	0.8	62	2.3	32	13.4	6	2.1
Acid-MAB@MBene	0.9	82	2.9	15	24.2	3	1.9

## Reference

- (1) Li, X.; Cui, H.; Zhang, R. First-Principles Study of the Electronic and Optical Properties of a New Metallic MoAlB. *Sci Rep* **2016**, 6 (1), 39790.