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Pyridinium Based Carboxyl Functionalized Porphyrin: An Easy Gateway to Afford Substituted Benzyl Aryl Ethers

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2.3 Synthesis Route of pyridinium-based carboxyl-functionalised porphyrin photocatalyst

2.3.1 Preparation of 4-((pyridin-4-ylimino) methyl)benzaldehyde (1A)



Scheme. S1 Preparation of (1A)

Separately dissolve terephthalaldehyde (0.68 g, 5 mmol) and p-aminopyridine (0.475 g, 5 mmol) in a beaker with 10 mL THF, then transfer to a 50 mL RB flask. The solution was

refluxed for 45 hours with constant stirring. We subsequent stirred the resulting liquid nonstop for 26 hours to acquire the necessary yellow precipitate. The solid product was wash away with aqueous ethanol, earlier acetone (3 x 10 mL), and dried in a hot air oven. [1] The greater compound (1A) was well-known by 1H NMR. (SI Fig. S12, Pg.14) ¹³C NMR, (SI Fig. S13, Pg.14), FT-IR (SI Fig. S14, Pg.15) and HR-MS analysis (SI Fig. S15, Pg.15), (1.337 g) (Scheme 1). (Yield = 62%).

400 MHz, DMSO-d6: δ 10.02 (s, 1H), 8.07 (s, 1H), 7.97 (d, J = 8.00 Hz, 2H), 7.79 (d, J = 8.00 Hz, 2H), 7.35 (d, J = 6.80 Hz, 2H), 6.61 (d, J = 6.00 Hz, 2H).

100 MHz, DMSO-d₆: δ 193.54, 156.06, 151.18, 148.97, 140.21, 136.59, 130.46, 127.85, 109.47 ppm.

FT-IR (cm⁻¹): 1709.34, 1612.67, 1507.53, 1195.17.

2.3.2 Preparation of precursor of photocatalyst 1-(carboxymethyl)-4-((4-formylbenzylidene)amino)pyridin-1-ium chloride (1B)



Scheme. S2 Preparation of (1B)

The resultant intermediate 1A (1.051 g, 5 mmol) was carefully transferred into a 50 mL RB flask containing 10 mL chloroform and kept at freezing temperature with constant agitation. The resulting solution was progressively combined with chloroacetic acid (0.5825 g, 5 mmol) and refluxed for 42 hours. After the reaction was complete, the solvent was evaporated under decreased pressure to produce a viscous orange liquid. To remove unreacted substrates, the sticky ionic liquid precursor 1B was rinsed with acetone (2 x 10 mL) before being dried in the oven.^[2] The creation of compound was (1B) confirmed by ¹H NMR (SI Fig. S16, Pg.16) ¹³C NMR, (SI Fig. S17, Pg.16), FT-IR (SI Fig. S18, Pg.17) and HR-MS analysis (SI Fig. S19, Pg.17), Yielding 53.21% (0.878 g) (Scheme 2).

400 MHz, DMSO-d6: δ 10.14 (s, 1H), 8.43 (s, 1H), 8.21 (d, J = 8.00 Hz, 2H), 8.13 (d, J = 6.80 Hz, 2H), 8.07 (d, J = 9.60 Hz, 2H), 6.85 (d, J = 7.20 Hz, 2H), 5.04 (s, 2H). 100 MHz, DMSO-d6: δ 193.59, 174.54, 169.32, 160.32, 159.39, 144.41, 140.21, 140.07, 130.47, 109.20, 59.97.

2.3.3 Preparation of Porphyrin based photocatalyst (PCFPc) (C)

Using previously published material, we attempted to build a PCFPc photocatalyst. [2] Precursor 1B (1.3070 g, 4 mmol) was diluted in 5 mL acetic acid and transferred to a 100-mL RBF. Pyrrole (0.2683 g, 4 mmol) was mixed with 5 mL of acetic acid in a dropping funnel. The pyrrole solution was gradually added to the RBF while being continuously agitated, and the entire mixture heated up for 35–45 minutes. Shortly after the process of reaction finished, the resultant mixture was constantly agitated at a comfortable temperature for a period of 12 hours. The resultant substance was afterwards rinsed several times utilizing water until it tested negative on blue litmus paper as a medium, then with methanol and acetone (3 x 10 mL). To achieve the PCFPc photocatalyst (C), this dark brown powder form had been dried in an oven using hot air at 60°C during the requisite time. The formation of was demonstrated by several

spectral scrutiny (C) such as ¹H NMR (SI Fig. S1), Solid ¹³C NMR (Fig.S2) Comparative FT-IR of precursor 1B and photocatalyst (Manuscript Fig. 2, Pg.5), Hammett acidity (Manuscript Fig. 3, Pg.6), BET (Manuscript Fig. 4, Pg.7) UV- Visible Spectrum and Fluorescence Emission Spectrum (Manuscript Fig. 5, Pg.8), Powder XRD (Manuscript Fig. 6, Pg.9), Electrochemical cyclic voltammetry (Fig. S7), SEM and EDAX (Fig. 8), XPS (Fig. 9), (HRMS(M+1)= 1266. Yield 47.03% (2.756 g) (Scheme 1).



Fig. S1. ¹H NMR Spectrum of PCFPc photocatalyst



Fig. S2. ¹³C NMR Spectrum of PCFPc photocatalyst



Fig. S3. CV of PCFPc photocatalyst

Table S.2

Electrochemical onset potentials and electronic energy levels of PCFPc Photocatalyst.

	1		0,			
Compound	E_{ox}^{onset}/V	E ^{onset} /V	E _{HOMO} /eV	E _{LUMO} /eV	$\mathbf{E}_{g}^{ec}/\mathbf{V}$	
PCFPc	1.18	-0.86	6.223	4.213	2.01	

 $E_{HOMO} = - [E_{ox \ vs. Fc} + 5.043] \text{ eV}, E_{LUMO} = - [E_{red \ vs. Fc} + 5.043] \text{ eV}, E_{g}^{ec}/V = E_{LUMO} - E_{HOMO}$



Fig. S4. a. SEM images of PCFPc photocatalyst b. EDAX of PCFPc photocatalyst



Fig. S5. a) XPS Survey of PCFPc Photocatalyst b) High resolve XPS Spectrum of C1s of PCFPc photocatalyst c) High resolve XPS Spectrum of O1s of PCFPc photocatalyst d) High-resolve XPS Spectrum of N1s of PCFPc photocatalyst e) High-resolve XPS Spectrum of Cl2p of PCFPc photocatalyst.



Fig. S6. Lab-made photoreactor (Outer View and Inner View)

Fig. S7. DEPT-135 NMR Spectrum of 4-((4-nitrobenzyl)oxy)benzaldehyde

Fig. S8. FT-IR Spectrum of 4-((4-nitrobenzyl)oxy)benzaldehyde

Fig. S9. HR-MS Spectrum of 4-((4-nitrobenzyl)oxy)benzaldehyde

Fig. S10. HR-MS Spectrum of Tempo

Fig. S11. ¹H NMR spectrum of (E)-4-((pyridine-4-ylimino) methyl) benzaldehyde (1A)

Fig. S12. ¹³C NMR spectrum of (E)-4-((pyridine-4-ylimino) methyl) benzaldehyde (1A)

Fig. S13. FT-IR spectrum of (E)-4-((pyridine-4-ylimino) methyl) benzaldehyde (1A)

Fig. S14. HR-MS spectrum of (E)-4-((pyridine-4-ylimino) methyl) benzaldehyde (1A

Fig. S15. ¹H NMR spectrum of 4-((4-formylbenzylidene)amino)-1-sulfopyridin-1-ium chloride(1B)

Fig.S16. ¹³C NMR spectrum of 4-((4-formylbenzylidene)amino)-1-sulfopyridin-1-ium chloride(1B)

Fig. S17. FT-IR spectrum 1-(carboxymethyl)-4-((4-formylbenzylidene)amino)pyridin-1-ium chloride (1B)

Fig. S18. HR-MS spectrum of 1-(carboxymethyl)-4-((4-formylbenzylidene)amino)pyridin-1ium chloride (1B)

Fig. S19. ¹H NMR spectrum of 4-((4-nitrobenzyl)oxy)benzaldehyde

Fig. S20. ¹³C NMR spectrum of 4-((4-nitrobenzyl)oxy)benzaldehyde

Fig. S21. ¹H NMR spectrum of 1-nitro-4-((4-nitrobenzyl)oxy)benzene

Fig S22. ¹³C NMR spectrum of 1-nitro-4-((4-nitrobenzyl)oxy)benzene

Fig. S23. ¹H NMR spectrum of 1-nitro-3-((4-nitrobenzyl)oxy)benzene

Fig. S24. DEPT-135 spectrum of 1-nitro-3-((4-nitrobenzyl)oxy)benzene

Fig. S25. ¹H NMR spectrum of 1-methoxy-4-((4-nitrobenzyl)oxy)benzene

Fig. S26. ¹³C NMR spectrum of 1-methoxy-4-((4-nitrobenzyl)oxy)benzene

Fig. S27. ¹H NMR spectrum of 4-((4-nitrobenzyl)oxy)aniline

Fig. S28. ¹³C NMR spectrum of 4-((4-nitrobenzyl)oxy)aniline

Fig. S29. ¹H NMR spectrum of 1-methoxy-4-((4-nitrophenoxy)methyl)benzene

Fig. S30. ¹³C NMR spectrum of 1-methoxy-4-((4-nitrophenoxy)methyl)benzene

Fig. S31. ¹H NMR spectrum of 1-((4-methoxybenzyl)oxy)-3-nitrobenzene

Fig. S32 ¹³C NMR spectrum of 1-((4-methoxybenzyl)oxy)-3-nitrobenzene

Fig. S33. ¹H NMR spectrum of 4-((4-methoxybenzyl)oxy)benzonitrile

Fig. S34. ¹³C NMR spectrum of 4-((4-methoxybenzyl)oxy)benzonitrile

Fig. S35. ¹H NMR spectrum of 1-methoxy-4-((4-methoxybenzyl)oxy)benzene

Fig. S36. ¹³C NMR spectrum of 1-methoxy-4-((4-methoxybenzyl)oxy)benzene

Fig. S37. ¹H NMR spectrum of 4-((4-chlorobenzyl)oxy)benzaldehyde

Fig. S38. ¹³C NMR spectrum of 4-((4-chlorobenzyl)oxy)benzaldehyde

Fig. S39. ¹H NMR spectrum of 4-((4-chlorobenzyl)oxy)benzonitrile

Fig. S40. ¹H NMR spectrum of 1-chloro-4-((4-nitrophenoxy)methyl)benzene

Fig. S41. ¹³C NMR spectrum of 1-chloro-4-((4-nitrophenoxy)methyl)benzene

Fig. S42. ¹H NMR spectrum of 1-chloro-4-((4-methoxyphenoxy)methyl)benzene

Fig. S43. ¹³C NMR spectrum of 1-chloro-4-((4-methoxyphenoxy)methyl)benzene

Fig. S44. DEPT-135 spectrum of 1-chloro-4-((4-methoxyphenoxy)methyl)benzene

Fig. S45. ¹H NMR spectrum of 4-(benzyloxy)benzaldehyde

Fig. S46. ¹NMR spectrum of 4-(benzyloxy)benzaldehyde

Fig. S47. DEPT-135 spectrum of 4-(benzyloxy)benzaldehyde

Fig. S48. ¹H NMR spectrum of 4-(benzyloxy)benzaldehyde

Fig. S49. ¹H NMR spectrum of 1-(benzyloxy)-4-nitrobenzene

Fig. S50. ¹H NMR spectrum of 1-(benzyloxy)-4-mithoxybenzene

Fig. S51. ¹³C NMR spectrum of 1-(benzyloxy)-4-mithoxybe

Fig. S52. ¹H NMR spectrum of 1-(benzyloxy)-3-mithoxybenzene

Fig. S53. ¹³C NMR spectrum of 1-(benzyloxy)-3-mithoxybenzene

Fig. S54. ¹H NMR spectrum of 4-(benzyloxy)aniline

Fig. S55. ¹³C NMR spectrum of 4-(benzyloxy)aniline

Fig. S56. ¹H NMR spectrum of (benzyloxy)benzene

Fig. S57. ¹³C NMR spectrum of (benzyloxy)benzene

Fig. S58. Recyclability FT-IR Spectrum of PCFPc photocatalyst

Table S3. Spectral data

Sr.	Code	Product	Spectral data
No			
1	2a	CHO O ₂ N	400 MHz, CDCl ₃ : δ 9.84 (s, 1H), 8.20 (d, J = 8.80 Hz, 2H), 7.80 (d, J = 8.80 Hz, 2H), 7.55 (d, J = 8.80 Hz, 2H), 7.02 (d, J = 8.80 Hz, 2H), 5.20 (s, 2H).
		(Manuscript Fig.3,4, Pg. 11)	¹³ C NMR 100 MHz, CDCl ₃ : δ190.70, 162.94, 147.80, 143.29, 132.10, 130.65, 127.68, 123.99, 115.10, 68.84 ppm.
2	2b	O ₂ N CHO	400 MHz, CDCl ₃ : δ 9.75 (s, 1H), 8.12 (d, J = 8.40 Hz, 2H), 7.71 (d, J = 8.80 Hz, 2H), 7.46 (d, J = 8.80 Hz, 2H), 6.93 (d, J = 8.40 Hz, 2H), 5.11 (s, 2H). ¹³ C NMR 100 MHz, CDCl ₃ : δ191.20, 163.44, 148.31, 143.80, 132.61, 131.15, 128.18, 124.50, 115.60, 69.35 ppm.
3	2c	O ₂ N NO ₂	400 MHz, CDCl3: δ 8.15 (d, J = 8.68 Hz, 2H), 8.12 (d, J = 9.00 Hz, 2H), 7.47 (d, J = 7.12 Hz, 2H), 7.45 (d, J = 9.68 Hz, 2H), 4.77(s, 2H). 100 ¹³ C NMR MHz, CDCl ₃ : δ 161.21, 148.16, 141.41, 129.61, 127.01, 124.96, 123.76, 64.03 ppm.
4	2d	O ₂ N NO ₂	400 MHz, CDCl ₃ : δ 8.26 (s, 1H), 8.06 (d, J = 8.00 Hz, 2H), 7.74 (d, J = 7.20 Hz, 2H), 7.35 (t, J = 8.00 Hz, 3H), 7.19 (d, J = 8.80 Hz, 2H), 4.51 (s, 2H).
5	2e	O ₂ N	400 MHz, CDCl ₃ : δ 8.10 (d, J = 6.80 Hz, 2H), 7.45 (d, J = 7.20 Hz, 2H), 7.21 (d, J = 8.80 Hz, 2H), 6.82 (d, J = 8.40 Hz, 2H), 4.50 (s, 2H), 3.74 (s, 3H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 160.82, 148.80, 147.01, 145.02, 129.06, 126.94, 123.59, 116.48, 63.63, 60.66 ppm.
6	2f		400 MHz, CDCl3: δ 8.03 (d, J = 6.32 Hz, 2H), 7.49 (d, J = 9.12 Hz, 2H), 7.35 (d, J =

		NH ₂	7.16 Hz, 2H), 7.25 (d, J = 6.76 Hz, 2H), 5.25 (s, 2H), 4.56(s, 2H).
		0 ₂ N	100 MHz, DMSO-d ₆ : δ 166.08, 142.99, 136.61, 133.81, 130.56, 128.48, 127.07, 126.91, 63.50 ppm.
7	2g	NO ₂	400 MHz, CDCl ₃ : δ 8.13 (d, J = 8.40 Hz, 2H), 7.45 (d, J = 8.80 Hz, 2H), 7.29 (d, J = 8.80 Hz, 2H), 6.70 (d, J = 9.20 Hz, 2H), 4.75 (s, 2H), 3.70 (s, 3H).
			¹³ C NMR 100 MHz, CDCl ₃ : δ 158.69,148.20, 147.30, 132.24, 127.00, 123.74, 115.73, 112.82, 64.01, 55.45 ppm.
8	2h	NO ₂	400 MHz, CDCl3: δ 8.14 (d, J = 8.72 Hz, 2H), 7.45 (d, J = 8.80 Hz, 2H), 7.06 (t, J = 7.96 Hz, 1H), 7.01 (s, 1H), 6.98 (d, J = 6.08 Hz, 1H), 6.74 (d, J = 6.16 Hz, 2H), 4.76 (s, 2H), 3.71 (s, 3H).
			¹³ C NMR 100 MHz, CDCl ₃ : δ 160.36, 148.19, 147.30, 130.55, 127.00, 123.78, 122.83, 117.16, 113.07, 64.01, 55.45 ppm.
9	2i	O CN	400 MHz, CDCl ₃ : δ 8.10 (d, J = 7.20 Hz, 2H), 7.43 (d, J = 8.00 Hz, 2H), 7.01 (d, J = 8.00 Hz, 2H), 6.55 (d, J = 8.40 Hz, 2H), 4.70 (s, 2H), 3.52 (s, 3H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 169.94, 147 20 145 29 142 51 127 20 125 25
			147.30, 143.28, 143.51, 127.30, 125.25, 121.84, 115.14, 114.71, 61.79, 58.89 ppm.
10	2j		400 MHz, CDCl ₃ : δ 7.35 (d, J = 8.80 Hz, 2H), 7.24 (d, J = 8.40 Hz, 2H), 6.85 (d, J = 8.80 Hz, 2H), 6.76 (d, J = 8.80 Hz, 2H), 4.54 (s, 2H), 3.76 (s, 3H), 3.73 (s, 3H).
			¹³ C NMR 100 MHz, CDCl ₃ : δ 158.91 158.57, 146.42, 141.26, 133.30, 128.59, 124.91, 113.78, 64.35, 60.59, 55.17 ppm.
11	2k	СНО	400 MHz, CDCl3: δ 9.89 (s, 1H), 8.12 (d, J = 8.64 Hz, 2H), 7.75 (d, J = 8.44 Hz, 2H), 7.46 (d, J = 6.64 Hz, 2H), 7.44 (d, J = 6.68 Hz, 2H), 4.76 (s, 2H).
			¹³ C NMR 100 MHz, CDCl ₃ : δ 191.07, 162.84, 148.34, 141.03, 134.66, 130.95, 129.48, 127.00, 123.71, 63.95 ppm.

12	21	CI CN	400 MHz, CDCl ₃ : δ 7.94 (d, J = 8.40 Hz, 2H), 7.29 (d, J = 8.00 Hz, 2H), 6.97 (d, J = 8.80 Hz, 2H), 6.62 (d, J = 8.80 Hz, 2H), 4.64 (s, 2H).
13	2m	CI NO2	400 MHz, CDCl3: δ 7.38 (d, J = 6.80 Hz, 2H), 7.34 (d, J = 7.20 Hz, 2H), 7.19 (d, J = 8.80 Hz, 2H), 6.79 (d, J = 8.80 Hz, 2H), 4.70 (s, 2H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 161.10, 145.42, 140.93, 132.03, 128.58, 127.04,
			116.87, 110.29, 65.23 ppm.
14	2n		400 MHz, CDCl3: δ 7.23 (d, J = 8.40 Hz, 2H), 7.12 (d, J = 8.80 Hz, 2H), 6.84 (d, J = 8.40 Hz, 2H), 6.74 (d, J = 8.80 Hz, 2H), 4.55 (s, 2H), 3.75 (s, 3H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 157.54,
			153.67, 131.23, 127.78, 127.28, 123.05, 115.29, 112.39, 63.03, 53.70 ppm
15	20	СНО	400 MHz, CDCl3: δ 9.81 (s, 1H), 7.76 (d, J = 8.72 Hz, 2H), 7.35 (t, J = 8.12 Hz, 3H), 7.28 (d, J = 6.76 Hz, 2H), 7.00 (d, J = 8.72 Hz, 2H), 5.07 (s, 2H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 190.83, 163.75, 135.96, 132.03, 130.14, 128.76, 128.36, 127.51, 115, 17, 70.20 ppm
16	2p	СНО	$\begin{array}{l} 400 \text{ MHz, CDCl3: } \delta 9.73 \text{ (s, 1H), 7.68 (d, J)} \\ = 8.80 \text{ Hz, 2H), 7.26 (t, J = 8.40 \text{ Hz, 3H),} \\ 7.20 \text{ (d, J = 6.80 \text{ Hz, 2H), 6.92 (d, J = 8.80 \text{ Hz, 2H), 5.00 (s, 2H).}} \end{array}$
17	2q	NO ₂	400 MHz, CDCl3: δ 7.85 (d, J = 6.40 Hz, 2H), 7.50 (d, J = 7.60 Hz, 2H), 7.45 (t, J = 7.20 Hz, 3H), 7.22 (d, J = -6.40 Hz, 2H), 4.77 (s, 2H).
18	2r		400 MHz, CDCl3: δ 7.63 (t, J = 6.64 Hz, 3H), 7.57 (d, J = 6.76 Hz, 2H), 7.37 (d, J = 8.80 Hz, 2H), 6.85 (d, J = 8.72 Hz, 2H), 4.90 (s, 2H), 3.63 (s, 3H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 161.23, 144.99, 141.01, 129.15, 128.58, 127.06, 123.22, 116.49, 65.06, 60.63 ppm.

19	2s		400 MHz, CDCl3: δ 7.39 (d, J = 10.68 Hz, 2H), 7.34 (t, J = 8.60 Hz, 2H), 7.19 (t, J = 7.80 Hz, 2H), 7.13 (s, 1H), 6.89 (d, J = 8.04 Hz, 2H), 4.68 (s, 2H), 3.81 (s, 3H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 164.77, 158.72, 139.34, 128.94, 126.84, 125.36, 122.15, 115.55, 111.44, 63.32, 53.74 ppm.
20	2t	NH ₂	 400 MHz, CDCl3: δ 7.29 (d, J = 8.40 Hz, 2H), 7.22 (t, J = 8.80 Hz, 3H), 7.01 (d, J = 8.40 Hz, 2H), 6.51 (d, J = 8.40 Hz, 2H), 5.28 (s, 2H), 4.59 (s, 2H). ¹³C NMR 100 MHz, CDCl₃: δ 161.68, 145.17, 141.18, 136.10, 129.20, 127.16, 123.25, 116.79, 64.77 ppm.
21	2u		400 MHz, CDCl3: δ 7.62 (t, J = 6.40 Hz, 2H), 7.15 (t, J = 7.60 Hz, 1H), 7.08 (d, J = 7.20 Hz, 2H), 6.96 (t, J = 7.60 Hz, 3H), 6.90 (d, J = 6.00 Hz, 2H), 4.90 (s, 2H). ¹³ C NMR 100 MHz, CDCl ₃ : δ 166.49, 136.06, 133.75, 133.05, 130.21, 129.72, 128.61, 128.18, 66.72 ppm.

Fig. S59. Leaching test of PCFPc photocatalyst

Fig. S60. HR-MS Spectrum of of PCFPc photocatalyst

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