## Nano-structured nickel trithiocarbonate complex supported on g-

## C<sub>3</sub>N<sub>4</sub> as an efficient electrocatalyst for urea electro-oxidation

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Figure S1. FT-IR spectra of  $g-C_3N_4$  and 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub>. Inset shows the range between 850-675 cm<sup>-1</sup>.



Figure S2. FT-IR spectra of ttc, Ni-ttc, and Ni-ttc-d.



**Figure S3.** Diffused reflectance spectroscopy (DRS) of (a) Ni-ttc, Ni-ttc-d,  $g-C_3N_4$ , and 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub> and (b) Ni-ttc-d in 200-1500 nm range.



Figure S4. Nitrogen adsorption/desorption isotherm of (a) 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub> and (b) g-C<sub>3</sub>N<sub>4</sub>.



Figure S5. XPS survey spectrum of (a) Ni-ttc and (b) Ni-ttc-d



Figure S6. XPS survey spectra of 35-Ni-ttc-d/g- $C_3N_4$ 



Figure S7. TEM-EDX elemental mapping of 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub>.



Figure S8. CV recorded in  $N_2$ -saturated 1 M KOH solution and 0.33 M urea at 50 mVs<sup>-1</sup> scan rate



Figure S9. Nyquist plots of g-C<sub>3</sub>N<sub>4</sub> and Ni-ttc-d



**Figure S10**. Cyclic voltammograms of (a) 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub>, (b) Ni-ttc-d, and (c) Ni-ttc in 1 M KOH solution at different scan rates. The plot of  $\Delta j vs$ . scan rates for (d) 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub>, (e) Ni-ttc-d, and (f) Ni-ttc.



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Sample	BET Surface area $(m^2 g^{-1})$
35-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	4.335
g-C <sub>3</sub> N <sub>4</sub>	4.726

**Figure S11**. Repeatability test of 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub> for UOR. **Table S1**. Multi point BET Surface area of 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub> and g-C<sub>3</sub>N<sub>4</sub>

## Calculation for the electrochemical active surface area (ECSA) from Faradic region

The electrochemical surface area (ECSA) the catalysts were calculated using the relation as given below

$$ECSA = Q/(mq)$$

Where, Q represents the charge required for the anodic oxidation (Ni<sup>2+</sup>/Ni<sup>3+</sup>) in CV, m is the catalyst loading on the GCE surface (~0.4 mg cm<sup>-2</sup>) and q is the specific charge related to the formation of Ni(OH)<sub>2</sub> monolayer and it is 257  $\mu$ C cm<sup>-2</sup>.<sup>1, 2</sup>

**Table S2.** Integrated charge and ECSA calculated for Ni-ttc, Ni-ttc-d, 5-Ni-ttc-d/g-C $_3N_4$ , 15-Ni-ttc-d/g-C $_3N_4$ , 35-Ni-ttc-d/g-C $_3N_4$  and 50-Ni-ttc-d/g-C $_3N_4$ .

Sample	Q (mC cm <sup>-2</sup> )	$ECSA(m^{2}/g)$
Ni-ttc	0.066	0.6425
Ni-ttc-d	0.139	1.36
5-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	0.89	8.75
15-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	2.8	27.25
35-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	4.59	44.72
50-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	3.98	38.75

Table S3. Tafel slope calculated for different samples

Sample	Tafel Slope (mV dec <sup>-1</sup> )
Ni-ttc	280
Ni-ttc-d	200
5-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	157
15-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	81
35-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	50
50-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	51

Table S4. R<sub>ct</sub> and R<sub>s</sub> values for different catalysts

Catalyst	$R_{\rm ct}$ (ohm)	$R_{\rm s}$ (ohm)
5-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	120	32
15-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	100	31
35-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	60	25
50-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	79	31

Calculation for the electrochemical active surface area (ECSA) from non-faradic region  $ECSA = C_{dl}/C_s$ 

where  $C_s$  is the specific capacitance of the material per unit area under identical electrolyte conditions and considered as 0.040 mF cm<sup>-2</sup> in 1 M KOH based on typical reported values

Table S5. ECSA calculated for 35-Ni-ttc-d/g-C<sub>3</sub>N<sub>4</sub>, Ni-ttc-d and Ni-ttc from non-Faradic region

Sample	Capacitance (mF cm <sup>-2</sup> )	ECSA (cm <sup>2</sup> )
Ni-ttc	2.38	4.165
Ni-ttc-d	2.82	4.935
35-Ni-ttc-d/g-C <sub>3</sub> N <sub>4</sub>	6.74	11.795

## References

1. R. K. Singh and A. Schechter, *Electrochim. Acta*, 2018, 278, 405-411.

 L. Wang, Z. Liu, S. Zhu, M. Shao, B. Yang, and J. G. Chen, ACS Appl. Mater. Interfaces, 2018, 10, 41338–41343.