INDIAN INSTITUTE OF TECHNOLOGY GUWAHATI
SHORT ABSTRACT OF THESIS

Name of the Student	:	**Anirban Chowdhury**
Roll Number	:	**166107007**
Programme of Study	:	**Ph.D.**

Thesis Title: **Bioinspired Synthesis of Metal Oxides and Sulphide Electrocatalysts for CO_2 and N_2 Conversion to Formate and Ammonia**

Name of Thesis Supervisor(s)	:	**Animes K Golder & Nageswara Rao Peela**
Thesis Submitted to the Department/ ~~Center~~	:	**Chemical Engineering**
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Key words for description of Thesis Work	:	Bioinspired route; Template-free synthesis; Cu_2O catalysts; SnO_2 nanoparticles; 1D Bi_2S_3 nanorods; pristine Co_3O_4 nanodiscs; Cyclic voltammetry; Linear sweep voltammetry; Impedance spectroscopy; Chronoamperometry; Electrochemical CO_2 reduction; Electrochemical N_2 reduction; Formate formation; NH_3 production

SHORT ABSTRACT

This doctoral work focuses on the development of environmentally friendly processes for the synthesis of metal oxides and metal sulphide (nano)electrocatalysts. These catalysts are then utilized for the electrochemical reduction of CO_2 and N_2 to produce value-added chemicals in a semi-batch laboratory electrolyzer.

In the first part of the study, Cu_2O nanoparticles (NPs) were synthesized using bioactive compounds found in *Sechium edule* (chayote) fruit extract. The formation mechanisms of Cu_2O NPs and their stabilizing cap through metal-organic ligands were investigated. The Cu_2O NPs were then applied to fabricate a modified electrode, resulting in 50 fold reduction in charge transfer resistance. The Cu_2O NPs/TCP electrode exhibited high catalytic activity for CO_2 reduction to formate ($HCOO^-$) with a promising Faradaic efficiency (FE), 65.3% in 60 min. However, the stability of Cu_2O NPs and their degradation over time were also studied.

The second part of the study aimed to enhance the efficiency of CO_2 reduction by synthesizing SnO_2 NPs using bioanalytes from the *Sechium edule* fruit extract. The SnO_2 NPs, as well as their calcined counterpart, were tested for CO_2 reduction, and both showed activity in forming $HCOO^-$ as the sole product. The FE of the calcined SnO_2 NPs reached high values, from 75.6 to 84.0% due to the predominant crystal facet in the calcined NPs.

A template-free process was developed in the third part to synthesize 1D Bi_2S_3 nanorods (Bi_2S_3-NRs) using the same bio-extract. The length and diameter of the nanorods were controlled by capping them with ascorbic acid (AA), present in the bio-extract. The AA capping prevented the oxidation of Bi_2S_3-

NRs during synthesis and led to a significant increase in the FE of $HCOO^-$ formation. The maximum FE was found to be 92.3%.

Lastly, pristine Co_3O_4 nanodiscs (NDs) were synthesized using commercially available gallic acid in an environmentally friendly process. The Co_3O_4 NDs exhibited distinct growth patterns and showed promising catalytic activity for electrochemical nitrogen reduction to produce NH_3. The Co_3O_4 NDs/TCP electrode demonstrated six times higher FE for NH_3 formation compared to the unmodified system. FE of NH_3 production of 2.6% was achieved with a yield rate of 17.2 μg h^{-1} $mg_{cat.}^{-1}$ at −1.25 V (vs. Ag/AgCl).

Overall, this doctoral work contributes to the development of efficient and environmentally benign electrocatalysts for CO_2 and N_2 reduction, with the potential to mitigate the adverse environmental impacts of fossil fuel consumption and CO_2 emissions.