



# ELECTROCHEMICAL FIXATION AND CONVERSION OF NITROGEN INTO AMMONIA BY $ZnMn_2O_4$ SPINEL DERIVED FROM SPENT BATTERY

## IITM Technology Available for Licensing

### Problem Statement

- In instance, the development of efficient catalyst for electrochemical synthesis of ammonia is a **great challenge** because of the rate of **ammonia formation and conversion efficiency** is **not satisfactory** due to competing hydrogen evolution reaction.
- Various catalysts have been explored for electrochemical ammonia synthesis by NRR,
- but most of the reported catalyst suffers in the selectivity for NRR.
- Hence, there is a need to address the issues.

### Technology Category/ Market

**Technology:** Preparation of  $ZnMn_2O_4$  through hydrothermal synthesis; **Industry & Application:** Material Science, Catalysts; **Market:** The global **zinc oxide** market is projected at a **CAGR** of **5.7%** during **2024-2030**

### Technology

- Present invention describes a **method of electrochemical fixation and conversion of nitrogen into ammonia by spinel  $ZnMn_2O_4$  derived from spent primary zinc carbon batteries.**
- Said method comprises a few steps explained hereinbelow:

#### Step 1

i)preparing spinel  $ZnMn_2O_4$  from primary battery waste by hydrothermal route;

#### Step 2

ii)preparing  $ZnMn_2O_4$  ink by ultrasonically dispersing 10 mg of  $ZnMn_2O_4$  from step (i) 5 into a solution containing 1 mL of dimethylformamide (DMF) and 10 $\mu$ L of 1 wt.% PVDF binder;

#### Step 3

iii)drop-casting the obtained ink from step (ii) onto polished glassy carbon electrode (GCE);

- Further, the derived  $ZnMn_2O_4$  from primary battery waste acts as an **electrocatalyst** to increase the selectivity of the **nitrogen reduction reaction (NRR)** to yield ammonia at **low negative potential (<- 0.6 V)** by **suppressing** the **hydrogen evolution reaction**.
- The spinel  $ZnMn_2O_4$  derived from primary **zinc carbon batteries** prepared by a simple thermal route with calcination & filtration.

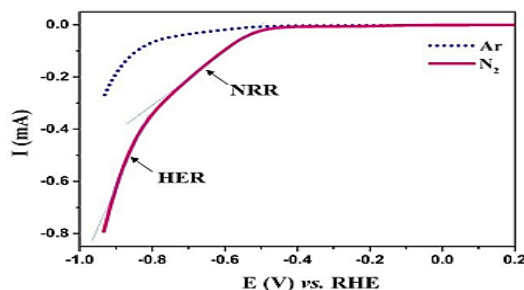


Fig.1 depicts LSV curves of the  $ZnMn_2O_4$  catalyst on GCE measured in Ar and  $N_2$  saturated 0.05 M  $H_2SO_4$  (100 mV s<sup>-1</sup>)

### Intellectual Property

IITM IDF Ref. 2211; Patent No:502600;

TRL (Technology Readiness Level)

TRL-4, Proof of concept tested in Lab;

### Research Lab

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**Key Features / Value Proposition**

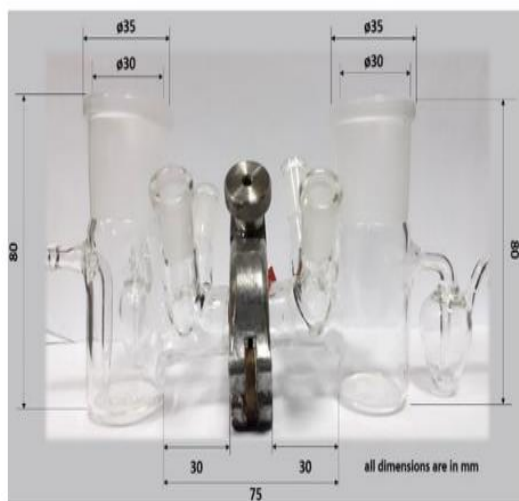
**❖ *Technical Perspective:***

- ❖  $ZnMn_2O_4$  derived from the primary battery waste divulged to be a promising electrocatalyst for NRR, **achieving an  $NH_3$  yield rate of  $13.5 \mu g h^{-1} mg_{cat}^{-1}$**  and with conversion efficiency of  **$\sim 51\%$  at  $-0.5 V$  vs RHE**.
- ❖  $ZnMn_2O_4$  electrocatalyst derived from spent zinc-carbon battery is **highly active and selective for  $N_2$  fixation**.
- ❖ Further, the **residue** collected by centrifugation is **dried at  $\sim 80^\circ C$  for 16h**. Later, it is **calcinated at  $400^\circ C$  for 6h** with a **20 heating/cooling rate of ( $5^\circ/min$ )** to obtain  $ZnMn_2O_4$ , wherein  **$570 \mu g/cm^2$  of  $ZnMn_2O_4$**  is coated on glassy carbon electrode.
- ❖ Obtained  $ZnMn_2O_4$  are **mesoporous in structure**.
- ❖ Catalyst employs a dissociative method to produce **electrochemical ammonia** by nitrogen reduction.
- ❖  $ZnMn_2O_4$  can effectively suppress the **hydrogen evolution reaction** to increase the selectivity of the NRR at **low negative potential region ( $< -0.6 V$ )**.

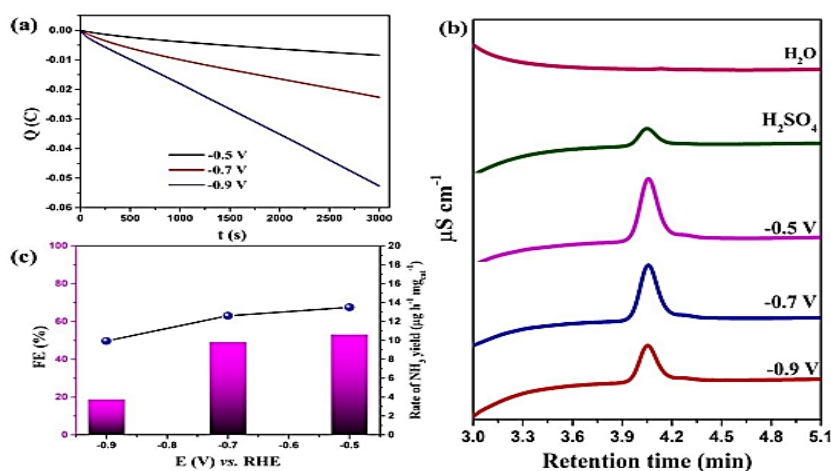
**❖ *Industrial Perspective:***

- ❖ The proposed process is **eco-friendly** and provides **efficient e-waste management** by obtaining  $ZnMn_2O_4$  derived from the primary battery waste.
- ❖ Applicable in the field of **Material Science**.

**Image**



**FIG 2: Illustrates H-cell showing dimensions in mm used for chronoamperometry study;**



**FIG. 3 shows (a) Q vs t plot of the  $ZnMn_2O_4$  catalyst on GCE in  $N_2$ -saturated  $0.05 M H_2SO_4$  at various potentials; (b) IC of post electrolyte solutions and blank; (c)  $NH_3$  yield rates and Faradaic efficiencies of the  $ZnMn_2O_4$  catalyst on the GCE measured in  $N_2$ -saturated  $0.05 M H_2SO_4$  electrolyte at different potentials.**

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