SYNTHESIS OF HIGHLY NITROGEN DOPED CARBON NANOSTRUCTURES

IITM Technology Available for Licensing

PROBLEM STATEMENT

- **Nitrogen doped carbon nanostructures** are promising for **energy applications due to their high surface area**, electrical conductivity, mechanical strength, thermal and chemical stability.
- **Synthesis methods include polymerizing** solid precursors at **450-700°C** and chemical vapor deposition at **950-1050°C.**
- In-situ preparation methods include high-temperature pyrolysis and pyrolysis at **600-1100°C.**
- The **study proposes** a **method for synthesizing highly nitrogen doped** carbon nanotubes and graphitic carbon nitride g-C_3N_4 simultaneously, **without the need for hazardous ammonia gas or liquid injection.**

TECHNOLOGY CATEGORY/ MARKET

Technology: Synthesis of highly nitrogen doped carbon nanostructures
Category: Micro & Nano Technologies
Industry: Advanced material
Application: Clean energy
Market: The global market size was valued at **USD 70.72 billion in 2022** and is projected to reach **USD 115.80 billion by 2030,** growing at a **CAGR of 6.54% from 2023 to 2030.**

INTELLECTUAL PROPERTY

IITM IDF Ref. 1411,Patent No: IN 325804

TRL (Technology Readiness Level)

TRL- 3, Experimental proof of concept

Research Lab

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TECHNOLOGY

Fig 1 shows a Apparatus for the synthesis of nitrogen doped carbon nanotubes and g-C_3N_4.

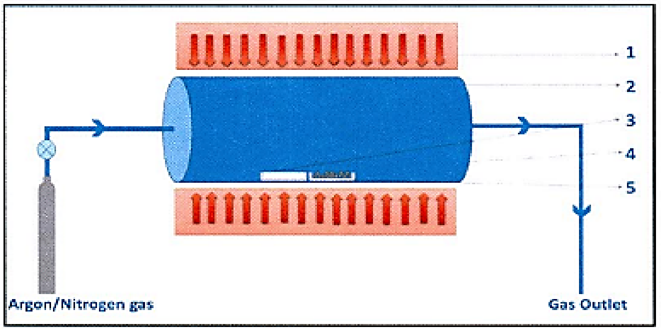
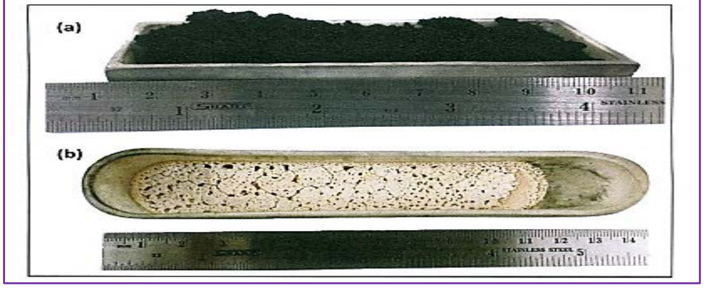


Fig 2 shows a photograph of the prepared (a) N-CNTs and (b) g-C_3N_4.



Process for Synthesis

Place precursor (melamine), catalyst ($MmNi_3$) in furnace
↓
Heat precursor, catalyst in inert atm @ ~650-750°C
↓
Polymerize precursor to g-C_3N_4, evolves NH_3, C_nH_{2n+2}
↓
NH_3, C_nH_{2n+2} react on surface of catalyst to grow N-CNTs
↓
Dissolve catalyst + N-CNTs in conc. acid to obtain pure N-CNTs

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FIG. 3 shows a 2-stage process for synthesis of N-CNTs from a nontoxic solid precursor.

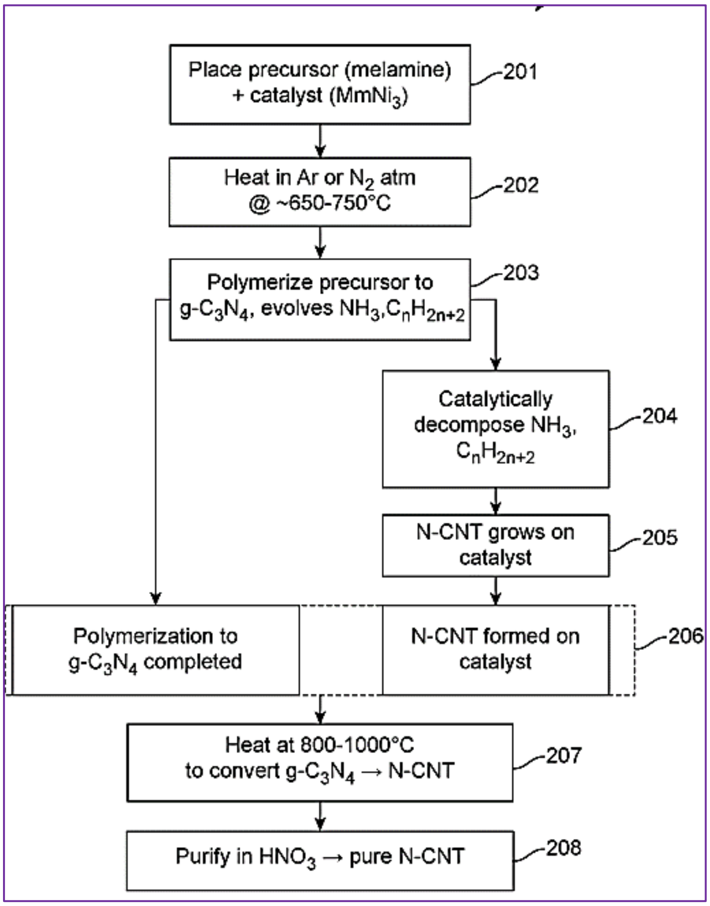


FIG. 5 shows the X-ray diffraction pattern for g-C_3N_4

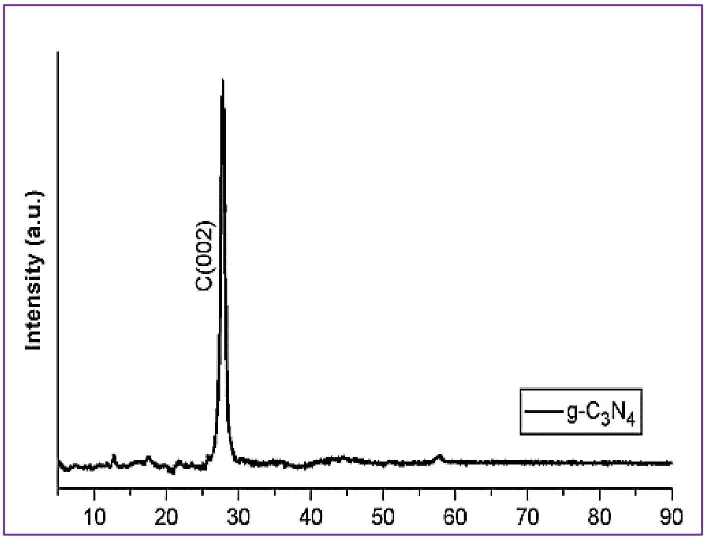
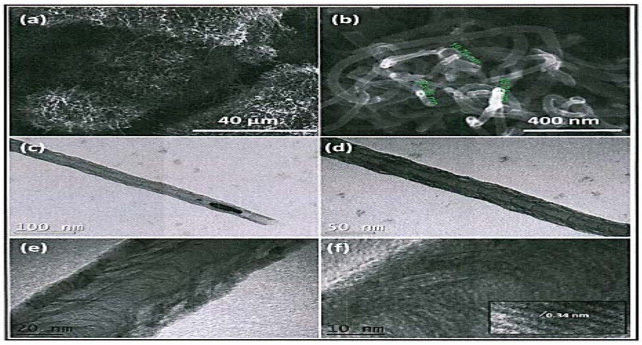


FIG. 4 shows the SEM and TEM images of as-grown and purified N-CNTs.



Key Features / Value Proposition

"Recovering Pure N-CNTs"

- Dissolving catalyst in concentrated nitric acid medium.

Precursor Selection Method

- Selects from nontoxic nitrogenous materials.
- Examples: melamine, dicyandiamide, thiourea, urea, guanidine hydrochloride.

Catalyst Method Overview

- Utilizes metal nanoparticles.
- Catalyzes metal alloys, oxides, hydroxides.

Nanoparticle Selection Method

- Selects nanoparticles from metals (Fe, Ni, Co, Mo).
- Combines metal alloys (Fe-Sn, Fe-Mo, Fe-Ni, ZrMnNi0.5Fe0.5, ZrMnFeNiCo).
- Combines metal oxides (Fe304, NiO).
- Combines metal hydroxides (Co(OH)2 or Ni(OH)2).

Synthesizing N-CNTs

- Uniform diameter with 20 nm outer diameter.
- Variance within 10%.

Synthesized N-CNTs: Bamboo-like Structure

- d spacing of 0.34 nm.
- Highly nitrogenized with 12 ± 3% N.
- Currently amended method.

Heating g-C_3N_4 to Decompose to Ammonia and Hydrocarbons

- Heats g-C_3N_4 to 800-1000 °C.
- Ammonia and hydrocarbons react to form N-CNTs on catalyst surface.

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