A METHOD OF PREPARING CROSSLINKED CHITOSAN

IITM Technology Available for Licensing

Problem Statement

- Current methods for carboxymethylation of chitosan involve multiple stages and complex procedures, making them time-consuming and labor-intensive.
- Conventional carboxymethyl chitosan production results in non-crosslinked products, limiting their mechanical and functional properties.
- There is a need for a simpler, more sustainable, and cost-effective method to prepare crosslinked carboxymethyl chitosan.

TRL (Technology Readiness Level)

TRL - 4: Technology validated in lab scale.

Intellectual Property

- IITM IDF Ref. **2298**
- **IN 529908 - Patent Granted**

Technology Category/ Market

Category- Advanced Biomaterials, Green Technology

Applications - Biomedical Applications, Water Treatment

Industry- Water Treatment, Healthcare and Pharmaceuticals

Market - Chitosan Market is expected to grow at a **CAGR of 15.2%** during the forecast period 2024-2031.

Research Lab

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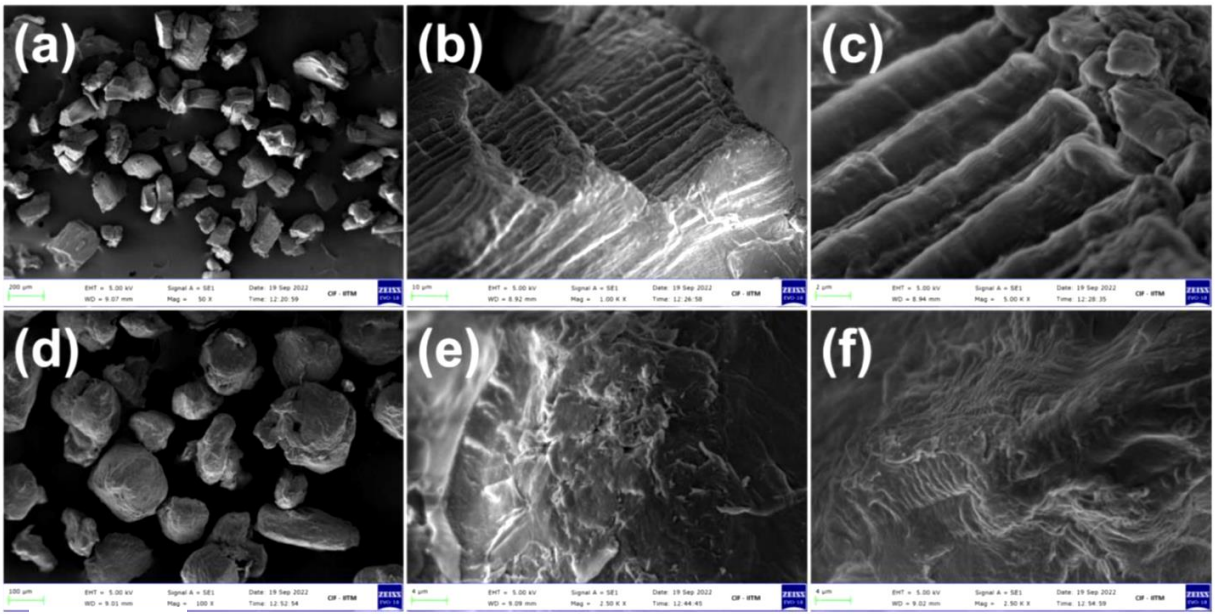

EM micrographs of (a) – (c) chitosan, and (d) – (f) crosslinked CMCh (molar ratio of glucosamine repeating units to MCAA of 1:3).

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Technology

The present invention provides a method of preparing cross-linked chitosan through a solvent-less carboxymethylation process.

1 **Solvent-Less Carboxymethylation**:

- The method involves mixing chitosan with halocarboxylic acid, heating the mixture to form crosslinked chitosan, followed by purification and drying, all without using solvents or mineral bases

2 **Reaction Conditions**:

- The reaction is carried out at 60-100°C for 6-10 hours, with the option of using a pestle and mortar or a twin-screw extruder, which allows for concurrent mixing and heating. The product is then purified by Soxhlet extraction and washing with an alkali solution.

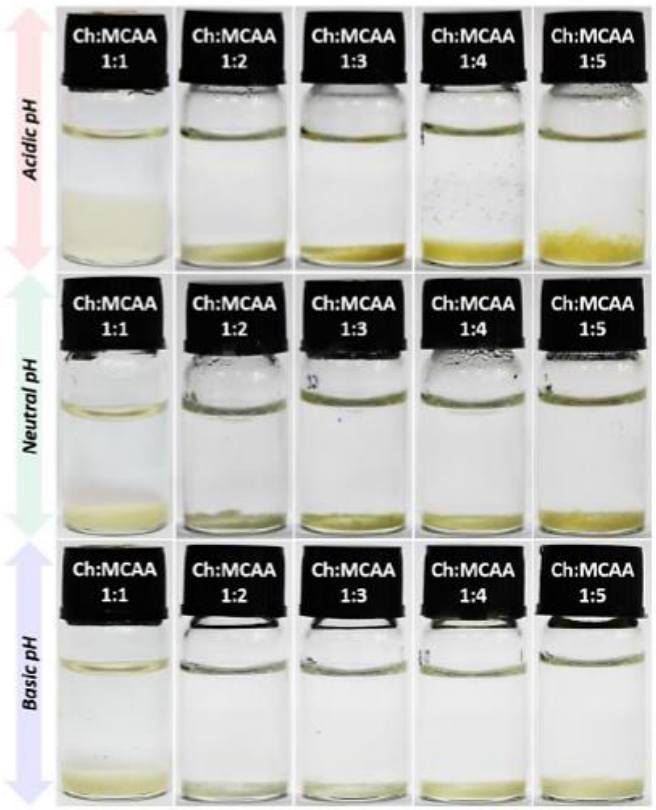

FIG. 1. Photographs show the insolubility of crosslinked CMCh in aqueous acidic, basic, and neutral solutions, even after heating at 60 °C for 24 hours.

Key Features / Value Proposition

a) Innovative Method

The invention presents a solvent-less carboxymethylation technique for crosslinking chitosan, using halocarboxylic acids and heating without solvents except for purification.

b) Procedure Overview

Chitosan and halocarboxylic acid are mixed, heated to 60-100 °C for 6-10 hours, then purified by Soxhlet extraction and rinsed with alkali solutions.

c) Twin-Screw Extruder Process

The process can be performed in a twin-screw extruder at 60-100 °C, with screw speeds of 10-200 rpm, ensuring concurrent mixing and heating, enhancing reaction control and efficiency.

d) Crosslinking Mechanism

N-carboxymethylation occurs under heat, with free amine groups in chitosan catalyzing the reaction, leading to electrostatic interactions and physical crosslinks between carboxylic acid and amine groups.

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