

MULTISTEP MODIFICATION OF CHITOSAN BASED ON GLYCIDYLMETHACRYLATE AND ETHYLENEDIAMINE –SYNTHESIS, CHARACTERIZATION AND CATION REMOVAL

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Chitosan is a biopolymer obtained by deacetylation of chitin. Recently, various research groups demonstrated interest in chitosan modification due to variety of its applications, such as encapsulating agent for the sustained release of drugs, support for immobilization of enzymes and chelating agent for toxic metal removal from industrial effluents [1]. The present investigation based on a multistep modification of chitosan by glycidylmethacrylate and ethylenediamine. A sample of 8.0 g of chitosan was suspended in 200 cm³ of water, followed by the slow addition of 5.3 cm³ of glycidylmethacrylate at 353 K, under mechanical stirring for 2 h. The final product was filtered, washed with water and ethanol. This product was coded as (C-gly). In a further step, 4.0 g of (C-gly) was reacted with 1.67 cm³ of ethylenediamine in ethanol under reflux with mechanical stirring for 72 h. The material obtained from this reaction was filtered, washed with ethanol and coded as (C-glyed)

The IR spectra for chitosan and modified chitosan are shown in Fig. 1. The band at 3435 cm⁻¹ in (a), (b) and (c) is due to OH and NH stretching. Glycidylmethacrylate modified chitosan (C-gly), shows bands at 1719 and 1635 cm⁻¹, which are due to C=O and C=C stretching of glycidylmethacrylate moiety Fig (1-b). The band at 1635 cm⁻¹ disappeared when glycidylmethacrylate modified chitosan reacted with ethylenediamine, confirm the reaction of ethylenediamine with vinyl carbon of (C-gly) Fig (1-c).

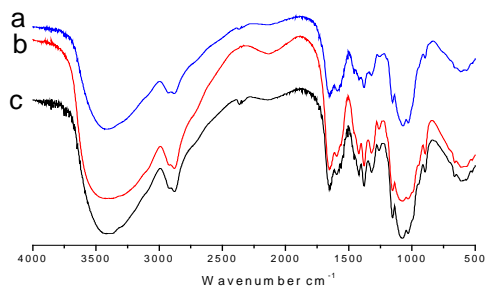


Fig 1: IR spectra of chitosan (a) glycidylmethacrylate modified chitosan (b) and ethylenediamine modified chitosan (c)

Solid state ¹³C NMR spectra of chitosan biopolymer and modified chitosan (C-gly and C-glyed) are shown in Fig. 2. Glycidylmethacrylate modified chitosan (C-gly) shows peaks at 175, 137 and 128 ppm, which are attributed to the carbon of C=O and C=C bonds of glycidylmethacrylate moiety Fig. (2b). The reaction of ethylenediamine with glycidylmethacrylate modified chitosan was confirmed by the disappearance of peaks at 137 and 128 ppm Fig (2c).

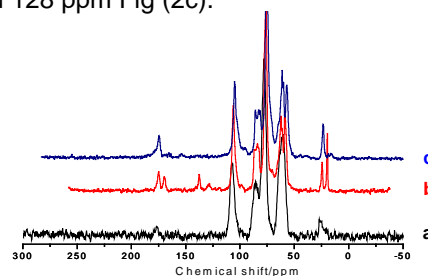


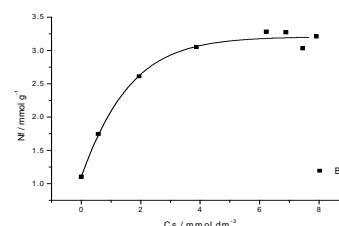
Fig 2: ¹³C NMR of chitosan, (a) glycidylmethacrylate modified (b) and ethylenediamine modified chitosan (c)

CHN analysis of chitosan and its derivatives (C-gly and C-glyed) are summarized in Table 1.

Table 1. Percentage of carbon (C), nitrogen (N) and their molar ratio in chitosan (C) and chitosan derivatives (mmol g⁻¹)

Sample	C	N	C/N
C	33.86	5.27	6.42
C-gly	36.69	4.21	8.71
C-glyed	33.97	4.57	7.43

The investigation demonstrated the successful modification of chitosan, which have the ability to copper removal from aqueous solution Fig. 3.



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