Proceeding of the 14th International Conference on QIR (Quality in Research)
Lombok, Indonesia, 10-13 August 2015
ISSN 1411-1284

Study of Different Operating Conditions during Carboxymethyl Chitosan Synthesis

Natalia Suseno[1,a*], Emma Savitri[1,b], and Karsono Samuel Padmawijaya[1,c]

[1]University of Surabaya, Raya Kalirungkut, Surabaya 60293 East Java, Indonesia

[a]suseno.natalia@yahoo.com, [b]savitri_ma@yahoo.com, [c]karsono_ksp@yahoo.co.id

Keywords: Chitosan; Carboxymethyl chitosan; Carboxymethylation; Degree of substitution; Swelling; Solubility

Abstract. Carboxymethyl chitosan (CMCh) was obtained under various conditions as a product of the etherification reaction between chitosan and monochloroacetic acid (MCA) in the presence of sodium hydroxide. Some variables used to synthesize carboxymethyl chitosan were of sodium hydroxide concentrations (10-50% w/v), etherification times (1-5hr), and temperatures (40-90^{o}C). The psysicochemical properties of the carboxymethyl chitosan were characterized through the estimation of degree of substitution (DS), swelling and solubility test, and FTIR analysis. There was an optimum condition for each variable based on the obtained highest DS, swelling degree and solubility. The highest value of DS being obtained when the carboxylation reaction conditions were performed at concentration of NaOH 30% (w/v), carboxymethylation time for 3 hours and carboxymethylation temperature at 60°C. Carboxymethyl chitosan (CMCh) has strong FTIR adsorption of carboxyl group in 1593.67 cm^{-1} and 1320.8 cm^{-1}

Introduction

Chitosan, is a cationic copolymer of glucosamine and N-acetylglucosamine, is a partially deacetylated derivative of a natural polysaccharide-chitin, which is one of the most abundant carbohydrates in nature and mostly derived from the exoskeleton of crustaceans. Chitosan has a unique set of useful characteristics such as biodegradability, biocompatibility, bioadhesivity and nontoxicity. These properties make chitosan and its derivative used in various field such as pharmaceutical, biomedicine, water treatment, cosmetics, agriculture, and food industry. Chitosan and derivative are also used as additives to cellulose fiber during the formation of paper in order to improve the mechanical and electrical properties of paper sheets [3]. However the applications of chitosan suffer severe limitations since it is insoluble in neutral or alkaline pH because of its very stable crystalline structure arising from strong hydrogen bonds. The solubility is observed only in acidic aqueous solutions below pH 6.5 (below the pKa of chitosan), at which a substantial fraction of its amino groups are ionized. It is generally soluble in acidic solutions such as those of acetic acid, lactic acid and dilute hydrochloric acid. The solubility of chitosan can be improved by depolymerization and its chemical modifications. Chitosan has three reactive amino, i.e. a primary hydroxyl group at C-6 and a secondary hydroxyl group at C-3 on each repeated unit, -and an amino group at C-2 on deacetylated unit (Fig. 1), which can be used for chemical modifications under mild reaction conditions to alter its properties. Many water-soluble derivatives have been prepared by quaternarization or by introducing hydrophilic groups like hydroxypropyl, dihydroxyethyl, hydroxyalkylamino, sulfate, carboxyalkyl groups as carboxymethyl, carboxyethyl, etc. in the macromolecular chain of chitosan. Compared with other water-soluble chitosan derivatives, Carboxymethyl Chitosan (CMC) has been widely studied because of its ease of synthesis, ampholytic character and possibilitises of ample applications, such as O-CMC, N-CMC, N,O-CMC) [1, 2]. Structures of chitosan (Ch) and chitosan derivatives showed in Fig. 1, which Chitosan is indicated as (R,R'=H); and derivatives chitosan such as *O*-CMC (R=CH_2COOH, R'=H); *N,O*-CMC(R, R'= CH_2COOH) [4].