PC 18 - Preparation and Properties of Carboxymethylchitosan

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The limited solubility of chitosan, restricted to sufficiently acid aqueous solutions, can be extended to an ample range of pH by reacting it with chloroacetic acid to result in carboxymethylchitosan. A set of such chitosan derivatives were prepared by varying the reaction time in the range 3h-10h and by employing different ratios chitosan/sodium hydroxide/chloroacetic acid. The ^{1}H and ^{13}C NMR spectra clearly showed the structural modifications resulting of the carboxymethylation of chitosan and they also revealed that the reaction took place at the hydroxyl and amine groups of chitosan, generating O,N-carboxymethylchitosan derivatives. The average degrees of substitution of the carboxymethylchitosan samples were determined by infrared spectroscopy and titrimetry and they varied in the ranged 0.5-1.5 depending on the reaction conditions. Unlikely chitosan, its carboxymethylated derivatives were soluble at pH>7.0 but they were insoluble in the range 3.0<pH. The X ray diffraction revealed that the carboxymethylation of chitosan dramatically decreased the intensity of the signals due to the presence of ordered domains. Also, regardless of their average degrees of substitution the carboxymethylchitosan samples were able to adsorb 2-3 times more water than the parent chitosan. The TG analysis in synthetic air atmosphere showed that the carboxymethylchitosan samples were less thermally stable than the parent chitosan, the temperature corresponding to the maximum weight loss being 315°C for the latter and ranging from 290°C to 297°C for the formers. The kinetics of the thermal degradation of chitosan and carboxymethylchitosans were studied by dynamic and isothermal experiments showing that the apparent activation energy is approximately 148kJ/mol for the former and ranges from 170kJ/mol to 205kJ/mol for the carboxymethylated derivatives.