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X-RAY DIFFRACTION STUDIES OF THE CHITOSAN NATURAL POLYMERS

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ABSTRACT

Chitin, a β-(1→4)-linked polymer of 2-acetamido-2-deoxy-D-glucose (*N*-acetyl-D-glucosamine), is mainly derived from the exoskeletons of insects, crustaceans and the cell walls of fungi and some algae. A new method to determine the degree of deacetylation (DD) of α-chitin and chitosan in the range of 17–94% DD using X-ray powder diffraction (XRD) is proposed.

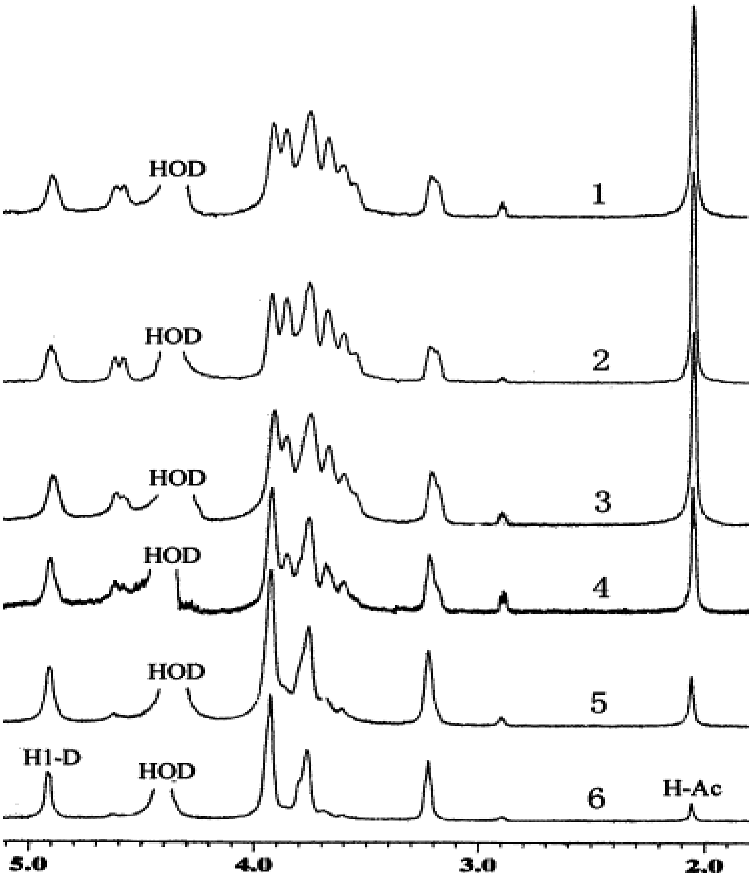
KEYWORDS: Chitin, insects, deacetylation, X-ray powder diffraction.

INTRODUCTION

There are three polymorphs in chitin, α-, β- and γ-chitin, which differ in regard to the arrangement of the chains within the crystalline regions[1]. The most abundant and stable form is α-chitin, which has been studied with X-ray diffraction and infrared spectroscopy.[3] It has a two-chain unit cell with $P2_12_12_1$symmetry, indicating an antiparallel arrangement of the chitin chain with strong intermolecular hydrogen bonding.[4] Chitosan, its N-deacetylated derivative formed by the action of concentrated alkali and high temperature treatment, has received much attention because of its numerous and wide variety of applications ranging from biomedical uses to waste-water treatment and to uses in the fibre industry[2-3]. The chemical behaviour of chitin and chitosan depends considerably on the degree of deacetylation (DD), a parameter defined as the mole fraction of deacetylated units in the polymorph chain. Therefore, the determination of DD has been one of the routine analyses performed for quality control on chitin and chitosan preparations. Processing adjustment based on the DD of chitin material is frequently required to facilitate the quick and feasible preparation of chitosan. Thus, the search for quick, user-friendly, low cost and accurate methods to determine the DD has been one of the major concerns over many decades[4]. Determination by X-ray powder diffraction (XRD), if feasible, could provide these advantages. In the present work, we set out to find the relationship(s) between the crystalline state and DD by way of X-ray powder diffractograms.

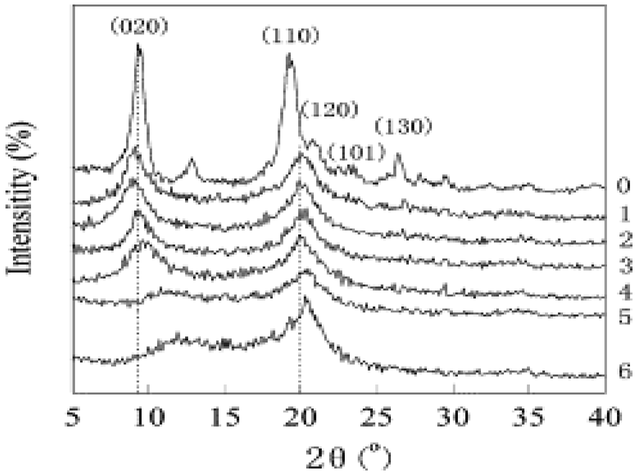
Determination of DD by1H NMR and FTIR spectroscopy and by potentiometric titration.

Chitin was mixed with highly concentrated NaOH in vacuum in order to pump out the air from chitin particles for accelerating the mass diffusion of NaOH to the inside of the chitin. This procedure provided a homogeneous deacetylation at high temperature and resulted in homogeneity of DD in a chitosan sample. shows the results of these seven samples. Nuclear magnetic resonance (NMR) spectroscopy is one of the most powerful absolute techniques, allowing a direct determination of DD, where the solvent (HOD) peak does not interfere with H-1D and HAc, as shown in Figure 1. In this experiment, NMR spectroscopy was used to calibrate the other techniques including FTIR spectroscopy, potentiometric titration and XRD[5].



X-ray diffraction analysis

Chitin and chitosan are polymorphic forms occurring as the α-form in shrimp and crab shell wastes. Figure 2 shows the XRD patterns of chitin and chitosans with different DDs. Five crystalline reflections were observed in the 2θ range of 5–40°. They were indexed as 020, 110, 120, 101 and 130 from the lower angle for chitin[6]. It is noted that the maximum peak of intensity at 020 reflection decreased with the increase of DD, and moved to a higher angle, and the second maximum peak of intensity at 110 reflection also decreased with the increase of DD, but the peak appeared at about the same reflection except that of chitin. Therefore, we proposed a crystalline index (CrI; %) expressed as $\mathrm{CrI}_{020} = (I_{020} - I_{\mathrm{am}}) \times 100/I_{020}$; another equation using I_{110} was expressed as $\mathrm{CrI}_{110} = (I_{110} - I_{\mathrm{am}}) \times 100/I_{110}$[7]. By peak fitting of the diffraction profiles in, the *d*-spacing and relative intensity were calculated as shown in. It was found that the *d*-spacing change of the (020) plane was the most obvious at around 50% DD, with more expansions of the crystal lattices than that of lower or higher DD, and thus moved to a wider diffraction angle with the increase of DD. This result provided another piece of evidence for the half-deacetylated chitosan with less intermolecular forces in relation to moisture absorption, which may interfere with FTIR determinations[8].



CONCLUSIONS

X-ray diffractograms on powder samples were obtained using a Bruker AXS D8 Advance X-ray diffractometer under the following operating conditions: 40 kV and 40 mA with Cu $K\alpha_1$ radiation at λ 1.54184 Å and acceptance slot at 0.1 mm. About 20 mg of the sample was spread on a sample stage, and the relative intensity was recorded in the scattering range (2θ) of 5–40° in steps of 0.1°.

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