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ON THE ULTRASOUND-INDUCED DEACETYLATION OF CHITIN

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The preparation of extensively deacetylated chitosan calls for the execution of consecutive deacetylation reactions in concentrated sodium hydroxide at high temperatures, favoring the occurrence of depolymerization. The depolymerization is more pronounced the harsher the reaction conditions, the use of inert atmospheres and the addition of reducing agents being proposed to avoid severe depolymerization. In this work the effect of the high intensity ultrasound irradiation on the kinetics of the deacetylation of chitin is evaluated. The polymorphs alpha-chitin and beta-chitin were extracted from cephalothoraxes of *Macrobrachium rosenbergii*, and squid pens of *Loligo sp.*, respectively. The procedures described by Acosta et al. [1] and Chaussard et al. [2] were applied for the extraction of alpha-chitin and beta-chitin, respectively. To carry out the ultrasound-induced deacetylation, a Branson Sonifier 450 (v=20kHz) and a stepped ½' probe were employed to irradiate the suspension of chitin in 40% aqueous NaOH observing a ratio volume of 40% NaOH/dry weight of chitin of 17.5/1. Thus, the glass reactor containing the suspension of chitin was disposed over a magnetic stirrer into a sound-proof box, the ultrasound probe was inserted and the suspension was continuously stirred while it was irradiated with high intensity ultrasound. The ultrasound equipment was adjusted for the intermittent irradiation mode and the irradiation amplitude (**A**) was tuned to high ($A=0.9\mathbf{A}_{max}$). The chitin suspension was submitted to ultrasound irradiation during 30 min and following it the glass reactor was closed, immersed in an oil bath at the desired temperature and maintained at this condition for a given time with constant stirring. The reaction was interrupted by immersing the glass reactor in a bath at -15^0C, the reaction medium was neutralized by adding 4 M aqueous HCl, the solid was extensively washed with 80% ethanol and then it was dried at room temperature. The same procedure was adopted for the study on the kinetics of the ultrasound-induced deacetylation of beta-chitin.

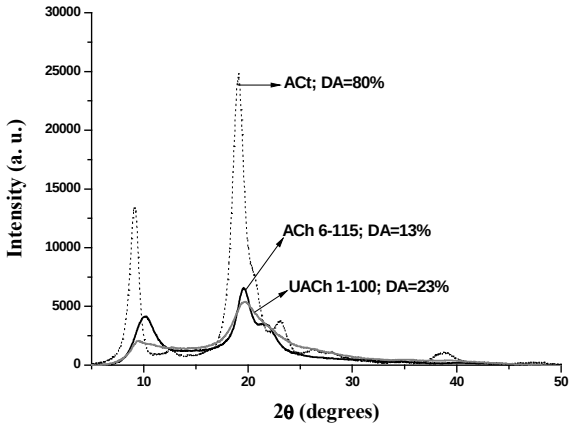

Fig. 1 X-rays diffraction of alpha-chitin (Act) and chitosan samples ACh 6-115 and UACh 1-100.

The ultrasound irradiation of the chitin suspension strongly favored the deacetylation reaction which was faster and more efficient as compared to the reaction of untreated chitin. Indeed, the application of the ultrasound treatment to alpha-chitin (sample Act) resulted in chitosan presenting DA=23% as compared to the chitosan produced from the same polymorph and by applying similar experimental conditions except for the use of ultrasound which presented DA=46.0%. The symbols ACh 6-115 and UACh 1-100 (Fig. 1) stand for chitosan prepared from alpha-chitin by carrying out the heterogeneous deacetylation for 6h at 115^0C and chitosan produced from ultrasound-treated alpha-chitin which was submitted to deacetylation for 1h at 100^0C, respectively. From the XRD patterns shown in Figure 1 it is concluded that the chitosan sample UACh 1-100 adopts a less ordered arrangement as compared to sample ACh 6-115. The study on the kinetics of the ultrasound-induced deacetylation of beta-chitin shown that a plateau corresponding to DA<15% is attained after 40min regardless of the reaction temperature in the range 100 – 120^0C, confirming that ultrasound-treated chitin is more efficient deacetylated than untreated chitin.

ACKNOWLEDGEMENTS

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