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STUDIES ON THE ULTRASOUND-ASSISTED DEACETYLATION OF CHITOSAN

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Chitin is a homopolysaccharide found in insects and invertebrates. The treatment of chitin with concentrated sodium hydroxyde results in chitosan, a polymer formed by 2-amino-2-deoxy-β-D-glucose and 2-acetamido-2-deoxy-β-D-glucose units which is soluble in moderately acidic medium [1]. The literature reports that the ultrasound irradiation reduces the polydispersity of chitosan, the reduction being greater the longer the irradiation duration [2]. In this study, the effects of the high intensity ultrasound irradiation on the deacetylation of chitosan are investigated by evaluating the characteristics of the resulting products.

Commercial chitosan (Galena Química Farmacêutica Ltda; Brazil) was used as the raw material for carrying out the ultrasound-assisted deacetylation (USAD process). It was dispersed in 40% sodium hydroxide and the resulting suspension (0.0442 g/mL) was submitted to ultrasound irradiation by using a Branson Sonifier 450S coupled to a ½ stepped probe. During the USAD process the reaction temperature was kept constant (60℃) and the irradiation amplitude was fixed to low amplitude (30%<Amax<50%). After 30min HCl was added to the reaction medium until pH 7.5-8.0, the solid was filtered, exhaustively washed with deonized water and then it was dried in a vacuum oven at 30℃. The parent chitosan (sample CCh) and the product of the USAD process (sample UCCh) were characterized by scanning electron microscopy (SEM), X-rays diffraction (XRD), thermogravimetric analysis (TGA), nuclear magnetic resonance (NMR) and infrared spectroscopy. The average degree of acetylation (DA) and viscometric average molecular weight (Mv) of the samples (Table 1) were determined by conductimetric titration and capillary viscometry, respectively.

Table 1: Characteristics of the chitosan samples.

Sample	*DA (%)*	*Mv (g/mol)*	*Tmax (ºC)*
CCh	26.1	60,451	297.0
UCCh	8.9	45,710	267.0

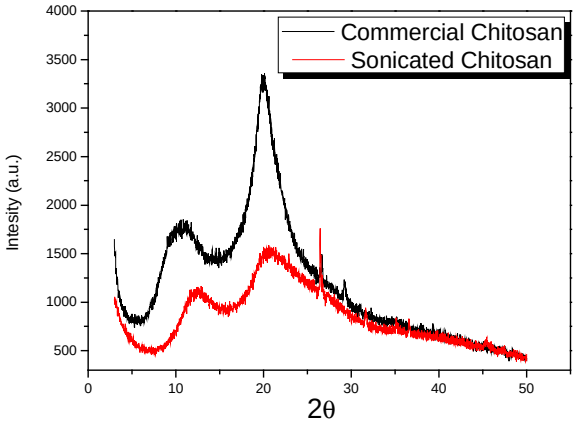

Fig. 1. X-ray diffraction of chitosans samples.

As shown in Table 1, the application of the USAD Process promoted the efficient deacetylation of the commercial chitosan (sample CCh) resulting in a product with DA=8.9% as compared to DA=26.1% for the parent chitosan. However, the deacetylation was accompanied by depolymerization which reduced the viscosity average molecular weight. The deacetylation of sample CCh affected the thermal stability of the polymer as sample UCCh, resulting from the application of the USAD Process, exhibited a lower temperature of maximum degradation as compared to the parent chitosan (Table 1). Comparing the XRD patterns of the samples CCH and UCCh (Fig. 1) reveals that the arrangement of the polymer chains in the solid state was also affected as the latter sample exhibits less intense and broader peaks.

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