V Simpósio Ibero-americano de Quitina
Sociedade Ibero-americana de Quitina
Marzo, 2010, Santiago, Chile

USAD PROCESS APLLIED TO BETA-CHITIN: INFLUENCE OF THE ULTRASOUND IRRADIATION ON THE RESULTING CHITOSAN.

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Chitosan, the main derivative of chitin, has many interesting properties such as its solubility in aqueous solutions of dilute acids, biodegradability, biocompatibility and low toxicity, among others. Thus, chitosan has been proposed for applications in many different fields, in industries, such as the cosmetic and food industry, as well as in medicine and dentistry as a biomaterial [1]. The reaction employed in most industries and research laboratories to convert chitin into chitosan, the alkaline hydrolysis leading to the deacetylation of chitin, is a heterogeneous process which results in chitosan whose characteristics depend on the accessibility to the reactive sites [2]. In this work, the isothermal and non-isothermal ultrasound-assisted deacetylation (USAD Process) is applied to beta-chitin and the effect of the process parameters on the characteristics of the resulting chitosan is discussed. Beta-chitin was extracted from the pens of *Loligo plei* and *L. sampaulensis*, according to the procedure described by Chaussard et al. [3]. The USAD Process was applied to beta-chitin suspended in aqueous 40% NaOH (w/w), submitted to high intensity ultrasound irradiation by using a Branson Sonifier 450S coupled to a ½` stepped probe (Fig. 1) and the process parameters varied as: i)irradiation amplitude: H=high (75%<Amax<90%), M=medium (55%<Amax<70%) and L=low(30%<Amax<50%); ii)irradiation duration: L=Long (50-60min), M=medium (35-40min) and S=short (10-30min) and iii) reaction temperature was adjusted to 50°, 60°, 70° or 80ºC (isothermal process) or the reaction temperature increased during the ultrasound irradiation, attaining 90°C after 5-10 min. (non-isothermal process). The USAD products were characterized by conductimetric titration and capillary viscometry to determine their average degree of acetylation (DA) and the viscosity average molecular weight (Mv), respectively. The thermal behavior, crystallinity and morphology of the samples were determined by thermogravimetric analysis (TGA), X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The samples were also characterized by size exclusion chromatography (SEC), which provided the data of weight average molecular weight (Mw) and polydispersity index (PI).

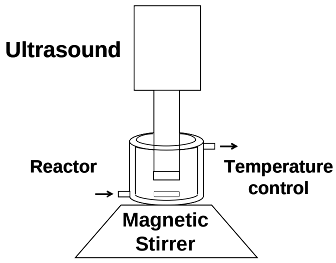

Fig. 1. Schematic representation of the reactor used for carrying out the USAD process.

When the non-isothermal USAD process was used, it is observed that increasing the irradiation amplitude and/or time resulted in more deacetylated chitosan (DA=7,5%) which were also more depolymerized (Mv=1,2 x10^5 g/mol).
From the data in Table 1 it is concluded that the isothermal USAD process is more efficient than any other process reported in the literature as it produces extensively deacetylated chitosan (DA<10%) after a short time reaction (30min) carried out at unusually low temperature (60 – 70ºC).

Table 1. Data for DA and Mv of chitosan samples obtained by the IUSAD process (low amplitude).

Sample	*DA (%)*	*Mv (g/mol)*
ILS50	25,4	452.000
ILS60	6,2	479.000
ILS70	9,4	683.000
ILS80	12,4	603.000

The characteristics of the chitosans resulting from the USAD process are strongly influenced by the processing parameters and its proper adjustment, mainly of irradiation amplitude and duration and the reaction temperature, allows the preparation of chitosan with well defined characteristics.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the funding agencies CAPES, CNPq and FAPESP for financial support.

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