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### The effect of Chitin Alkaline Deacetylation at Different Condition on Particle Properties

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#### Abstract

The abundant biopolymer chitin, found mainly in crustaceous exoskeleton, such as crab, shrimp and lobster, can be deacetylated to yield chitosan. This slightly different biopolymer is more reactive than chitin, being more effective for many applications in fields as environmental remediation, biomedical sciences, catalysis and so on. The main process for chitin deacetylation used sodium hydroxide solutions at high temperatures for long times to obtain chitosan with high deacetylation degree (DD). The present study has evaluated the effect from room temperature (RT), 363 and 393 K, hydroxide concentration (2.0 or 10.0 mol dm<sup>3</sup>) and time (3 and 24 h) on shrimp chitin deacetylation. Similar amounts of chitin and sodium hydroxide solutions were stirred jointly and the resultant solids were filtered and washed until pH 7, than dried at environmental conditions. The obtained samples were characterized by several techniques, such as elemental analysis, X-rays diffraction (XRD), laser scattering and absorption spectroscopy at infrared region with Fourier transform (FTIR), which was used for DD calculation. The results showed that all chitin-chitosan samples did not reach DD > 90 %, as observed for some good commercial chitosans. The highest DD was obtained by the sample prepared at more drastic conditions, as expected, however the higher sodium hydroxide concentration leads to decrease of molecular mass when associated with high temperatures. The crystallinity was influenced mostly by reaction time, which change the positions and intensities as indicated by XRD main peaks, located at 9.3 and 19.4 ° 20. Particle sizes were strongly diminished by treatment at 393 K, what imply also some increase at the pressure, favoring chain dissociation reactions. This work mapped several properties for chitin-chitosan samples achieved by the described conditions.

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#### 1. Introduction

Chitin and chitosan are natural and abundant polymers with immense structural possibilities for chemical and physical modifications to engender novel properties, functions and applications, especially in environment and biomedical areas.<sup>1</sup> Despite its enormous availability, the use of chitin has been restricted by its intractability and insolubility. Thus, for many applications chitosan present better performance,<sup>2-4</sup> due higher basicity at amino groups,<sup>3-6</sup> which imply a correlation with the deacetylation degree (DD).<sup>7</sup> Chitosan and chitin are linear copolymers composed by  $\beta(1 \rightarrow 4)$ -[2-acetamido-2-deoxy- $\beta$ -D-glucopyranose/2-amino-2-deoxy- $\beta$ -D-glucopyranose] units<sup>8</sup> as shown at Figure 1. Consequently, the amino/acetamido group ratio will define if the polymers are either as chitosan or chitin forms.<sup>9</sup> Despite chitosan occurs as a component of the cell wall of some fungi, it is generally produced by carrying out the deacetylation of chitin, which is found in the shells of crustaceans, particularly lobsters, crabs and shrimps.<sup>10</sup> Due to its particular physical, chemical and biological properties chitosan has widespread applications in the food industry,<sup>11</sup> pharmacy and medicine,<sup>12,13</sup> agriculture,<sup>14</sup> and wastewater treatment.<sup>6,15,16</sup> Also many further modifications can be performed to improve and find out new uses in similar ways as observed for cellulose.<sup>17-19</sup> Thus, this investigation consisted to determine the effect of chitin alkaline deacetylation at different conditions, regarding the DD achieved, morphology, particle charge and crystallinity. Since deacetylation process is already mastered by the industry, no study about these intermediate conditions were reported yet.



Fig. 1. Chitin/chitosan schematic basic units, 2-acetamido-2-deoxy-β-D-glucopyranose (a) and 2-amino-2-deoxy-β-D-glucopyranose (b).

#### 2. Experimental Part

#### 2.1. Sample preparation

Commercial sodium hydroxide, chitin and chitosan were purchased from Aldrich. The present study has evaluated the effect of temperature, hydroxide concentration and time on chitin from shrimp in the deacetylation process. Similar amounts of chitin and sodium hydroxide solutions were stirred jointly at room temperature or 363 K or even aged under static conditions at 393 K into a stainless steel autoclave, after previous mixing at room temperature. NaOH concentrations of 2.0 and 10.0 mol dm<sup>3</sup> were employed while the reaction times were 3 and 24 h. The resultant solids were filtered and washed until pH 7, than dried at environmental conditions. The experimental conditions for each sample are summarized in Table 1 that also included some other results.

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