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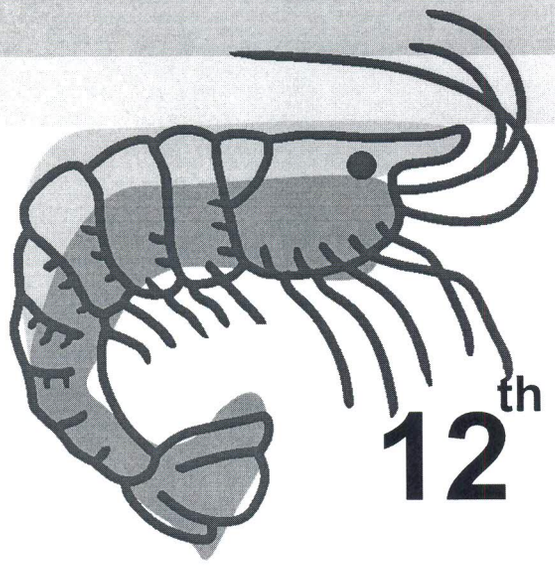

6th Iberoamerican Chitin Symposium

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12th International Conference on Chitin and Chitosan

VI SIAQ / XII ICCC
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Fortaleza / Brazil

ABSTRACTS

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CHARACTERIZATION OF N,N,N-TRIMETHYL CHITOSAN/TPP NANOPARTICLES LOADED WITH VITAMIN

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N,N,N-trimethyl chitosan (TMC) is a cationic polyelectrolyte obtained by the huge methylation of chitosan parent polymer [1]. The resultant water soluble derivative has largely used for several applications mainly as nanoparticles [2]. Typical features obtained via ionic gelation process with sodium tripolyphosphate (TPP), are nanoparticles size in the range of 110-180 nm with drug entrapment efficiency up to 90%, for evaluation of bovine serum albumin as a model drug. Several others important applications have since emerged for TMC nanoparticles obtained by ionic gelation with TPP, such as: nasal and oral vaccine delivery system; protein carrier, insulin controlled release, and food industry. Considering this, the possibility of producing TMC nanoparticles with using TPP are being investigated in this work, aiming their food application as intelligent packaging. This make sense, once the development of new and natural products to prolong the shell-life of foods has been stimulated by a growing consumer demand for healthy, residue-free fresh products and also concern on ecological packaging.

The TMC was synthesized by methylation of chitosan with dimethylsulfate at 70°C [1-3]. The nanoparticles were obtained according the method related by por Moura et al. [4].

The nanoparticules were characterized by AFM, SEM and Zeta Potencial, FTIR and ^{13}C NMR Spectroscopy CP-MAS in solid state.

Combinations of concentrations of TMC and TPP resulted in nanoparticles with varying sizes for which the capability for loading with vitamins was investigated. The tested vitamins were: B12, B9 and C. Zeta-potential measurement demonstrated that the size of the nanoparticles was optimized (196 ± 8 nm) when the lowest TMC and TPP amounts were used, *i.e.*, 0.86 mg mL^{-1} and 0.114 mg mL^{-1} respectively (Table 1). As the TMC and/or the TPP concentrations increase, the resulting size of the particles increases. Such nanoparticle formation is governed by the neutralization ratio between positively charged TMC and negatively charged TPP. After the TMC-TPP nanoparticle formation, a shift was observed from 3416 to 3438 cm^{-1} associate NH_2 and OH bands turning into a shaper band.

Table 1. Influence of TMC and TPP concentration on the nanoparticles size and zeta potential.

TMC (mg mL^{-1})	TPP (mg mL^{-1})	Particle size (nm)	Zeta potential (mV)
0.86	0.114	196 ± 8	31.6 ± 0.6
1.14	0.114	302 ± 15	24.7 ± 1.0
1.14	0.214	606 ± 25	12.7 ± 0.4

After interaction with TPP in nanoparticle formation, changes were observed on the TMC ^{13}C NMR spectrum. TPP does not have carbon atoms detectable by ^{13}C NMR spectroscopy, but its bonding with TMC charged sites decreases the intensity of some C signals (Figure 1). The most sensible change was found for carbon 1 substituted and carbon of the trimethyl signals which, in effect, are the signals directly related with the charged amino group.

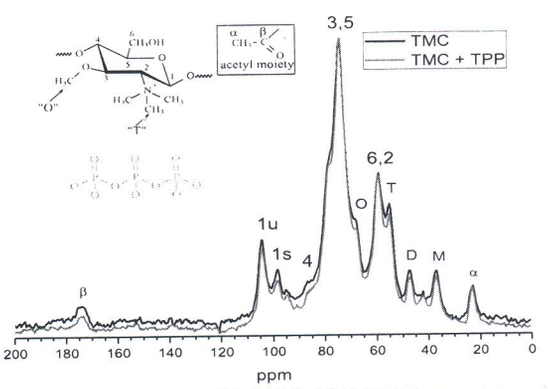

Fig. 2. - Solid state CP-MAS 13C NMR spectrum of pure TMC (———) and in nanoparticles combination with TPP (.....).

The solid state ^{13}C NMR spectroscopy was also very important to verify the encapsulation of vitamins molecules by the TMC-TPP nanoparticles. The spectrum of TMC-TPP-VitB9 nanoparticles clearly confirms the incorporation of a vitamin molecule within the nanoparticle.

By AFM, the cast nanoparticles film displays a regular distribution over the polar glass surface. Surface analysis conducted by SEM also revealed a regular distribution of the nanoparticles over the glass surface

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