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PREPARATION AND CHARACTERIZATION OF ULTRAFINE BIOPOLYMER PARTICLES

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ABSTRACT

A new method was developed to prepare chitosan ultrafine particles through biopolymer interaction with sodium bis (2-ethyl hexyl) sulfosuccinate, an anionic branched chained surfactant (AOT). Chitosan the second abundant biopolymer after cellulose is a natural, linear and cationic polymer, with the chemical structure: 2-amino-2-deoxy-(1→4)-β-D-glucopyranan. The micro- and nanoparticles are formed instantaneously when the drops of cationic biopolymeric solutions come into contact with anionic surfactant solution.

In order to obtain ultrafine particles an experimental apparatus equipped with a high pressure cell was used. The biopolymer solution in contact with high pressure CO_2 was sprayed into surfactant solution bath, through a stainless steel capillary nozzle. A maturation step of 12-14 hours is required before separation by centrifugation of the obtained particles from the liquid phase containing surfactant. The chitosan/AOT particles are washed 3-4 times with Millipore water and ethanol to remove the traces of surfactant. The washing process is followed by centrifugation and the supernatant containing nanoparticles was decanted and lyophilized.

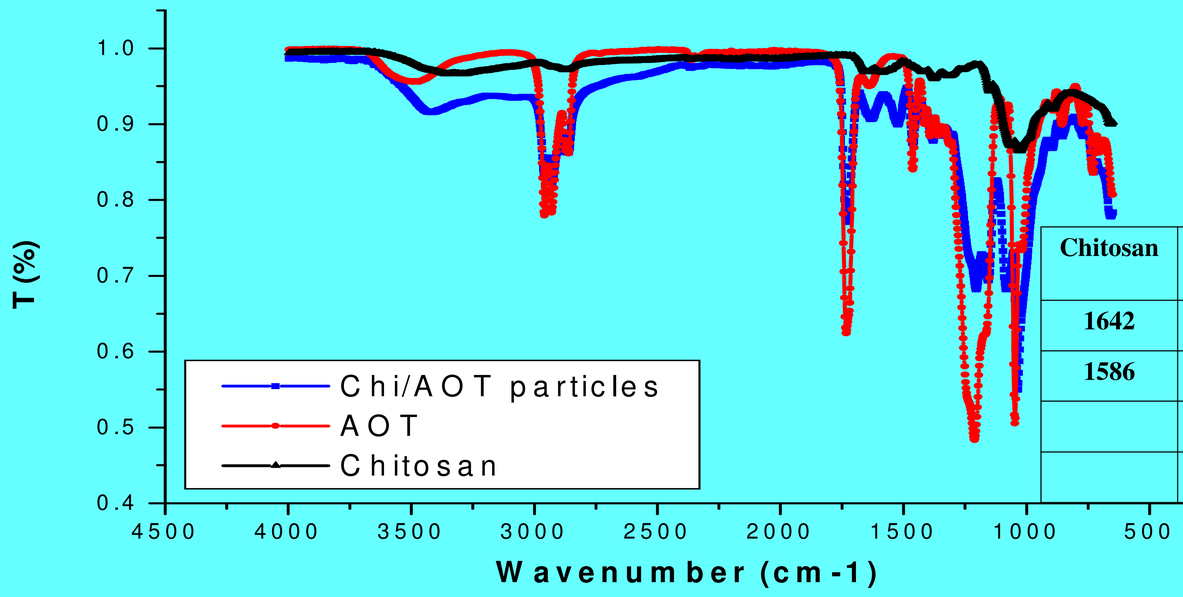
The effect of experimental parameters including the spraying pressure and the distance between the nozzle and the surface of surfactant solution on size and shape of ultrafine particles was studied. The obtained particles were characterized by Fourier Transform Infrared Spectroscopy (FTIR), dynamic light scattering measurements (DLS), scanning electron microscopy (SEM) and optical microscopy.

The micro-sized lyophilized particles can be used to remove organic pollutants from wastewaters.

Experimental apparatus

FTIR

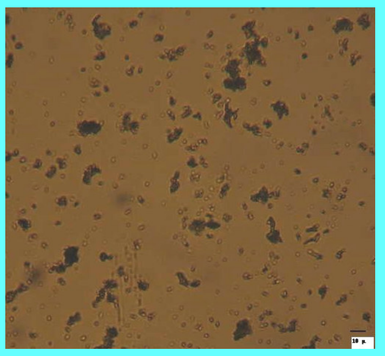
Biopolymer-surfactant complex formation was investigated by Fourier Transform Infrared Spectroscopy



Chitosan	AOT	Chi/AOT	Assignment
1642		1634	ν_{NH}, δ_{NH2}
1586		1522	δ_{NH2}
	1212	1204	ν_{SO}
	1093	1082	ν_{SO}

Optical microscopy

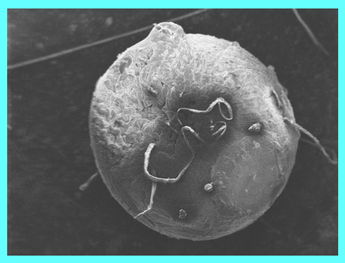
Microparticles of Chi/AOT



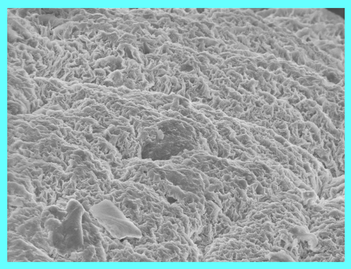
Wires of Chi/AOT

Morphology - SEM Images

Chi-AOT microparticle

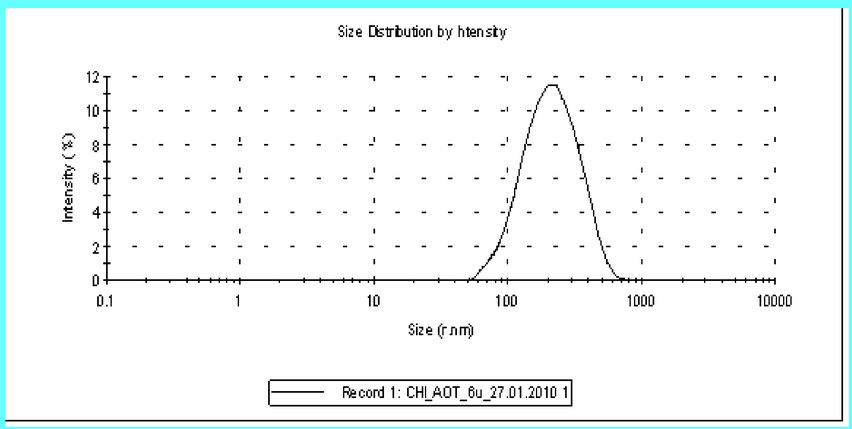


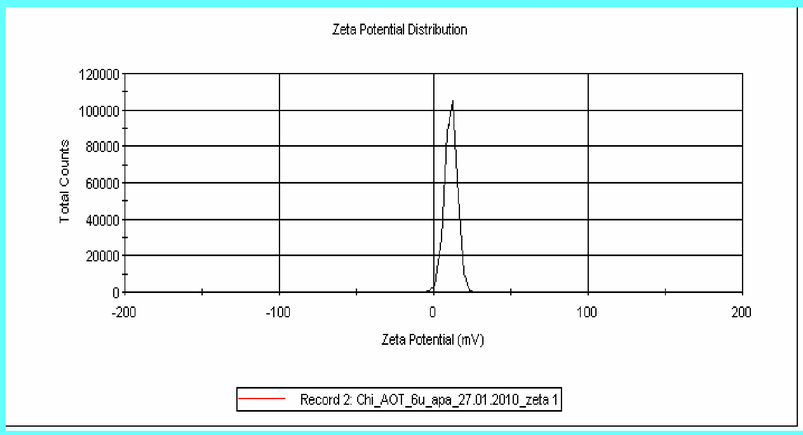
Surface of microparticle



DLS

Lyophilized particles were resuspended in water, subjected to ultrasonication for Dynamic Light Scattering measurements

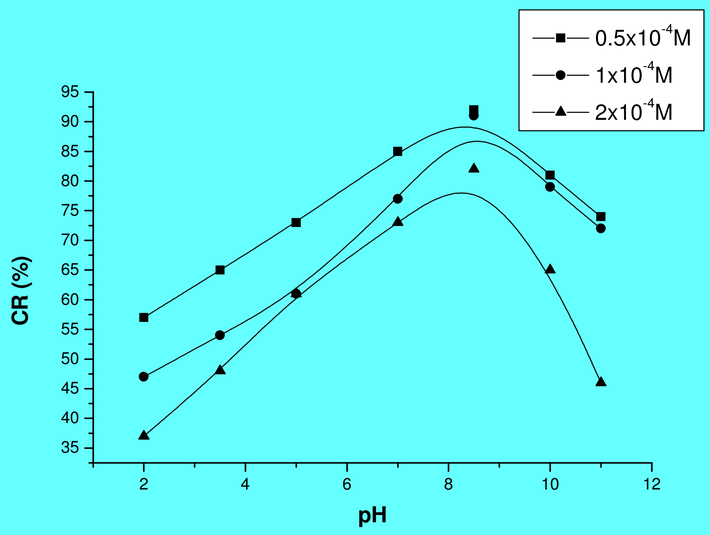




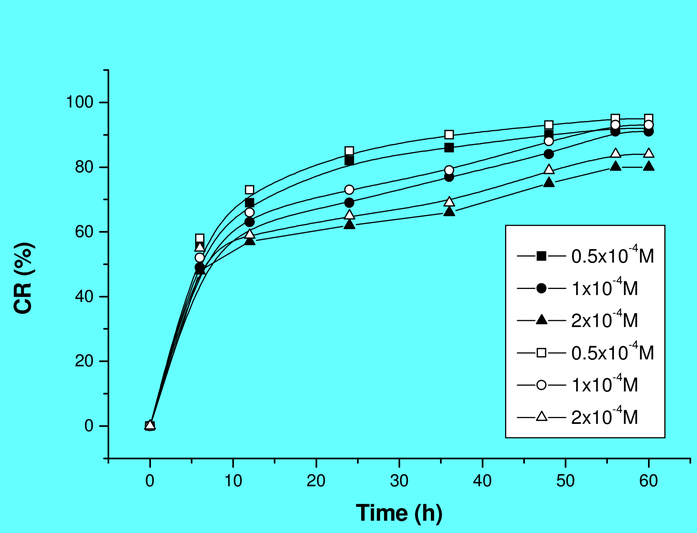
Effect of spraying pressure on particle size

Sample	Pressure (bar)	$\bar{D}p$ (nm)	Zeta potential (mV)
4u	20	234.0	+34.6
6u	30	194.8	+11.2
7u	40	112.8	+6.97

Effect of pH and initial concentration on Contaminant Removal (CR) for Phenol



Contaminant removal kinetics for Phenol at different initial concentration; amount of sorbent: 5g/L (black symbols), 10g/L (empty symbols), pH=8.5



CONCLUSIONS

- **It was developed a new method of producing particles by using an experimental apparatus equipped with a high pressure for spraying the polymer into surfactant solution.**
- **As a result of the interaction between a cationic chitosan biopolymer and anionic sodium bis (2-ethyl hexyl) sulfosuccinate branched chained surfactant (AOT), micro- and nanoparticles were obtained.**
- **By varying the experimental parameters such as spraying pressure and the distance between the nozzle and the surface of surfactant solution, particles of different sizes are obtained.**
- **SEM measurements show that microparticles treated by lyophilization process presents a rough surface with many pores.**
- **The FTIR measurements confirm the interaction between amino group of chitosan and sulfonate group of AOT.**
- **The obtained nanoparticles have a narrow size range (from DLS measurements) and presents positive charges which gives them some stability.**
- **Lyophilised microparticles have been successfully used in advanced treatment of wastewaters for the retention of phenol.**
- **The adsorption efficiency of phenol is optimal in the range of pH 8-9, increases with the amount of used particles and decreases with increasing of initial pollutant concentration.**

REFERENCES

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