

# Analysis of the Adsorption of Algae in Hydrogels Using Ftir and Drx



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## Abstract

In the research, hydrogels of chitosan with glutaraldehyde were synthesized as a cross-linking agent to carry out the adsorption of Spirulina algae and to establish bases for the development of a controlled release system via transdermal for patients with low levels of iron. The determination of functional groups and the adsorption was carried out by IR spectroscopy where decreases of these groups in the fingerprint zone were verified and the results were verified through XRD through the formation of two crystalline zones.

**Keywords:** Spirulina algae; Chitosan; Hydrogels; FTIR; DRX

## Introduction

Hydrogels are materials with three-dimensional structure that can increase their volume due to the thermodynamics of hydrophilic groups with polar groups of water or biological fluids [1,2]. Hydrogels are normally synthesized from natural materials such as gelatin, marine algae and animal collagen, which are called polysaccharides and are constituted by ortho-glycosidic repeating units, although they are also obtained from acrylate polymers. The excellent biocompatibility, biodegradation, smooth consistency as well as porosity of the structure allows the hydrogels to be used in medical applications such as tissue engineering, controlled release systems, biosensors and hemostasis sales [2-4]. A characteristic of hydrogels is the bioavailability where from the adsorption of enormous amounts of drugs the side effects are reduced by improving the release at the specific site [5].

Currently, hydrogels are synthesized by biodegradable and biocompatible natural polymers. Chitosan represents the most important natural polymer in the synthesis of hydrogels, which is obtained from the alkaline deacetylation of chitin and is the most widely used polysaccharide in medical areas attributed to the amino groups and the cationic charge of their structure. The adsorption of drugs for the development of controlled release systems both oral and subcutaneous in treatments against cancer and diabetes [6]. In addition, biocompatibility with biological systems allows to be used as scaffold for cell and tissue growth [5].

At present, natural polymer based nanostructures are being used with the aim of obtaining a controlled release system in iron adsorption because the low concentration of iron in the bloodstream represents one of the main nutritional problems to Worldwide, attributed to it is responsible for oxygenation, hormone synthesis, cell cycle control, as well as enzymatic reactions in the human body and a low iron diet, infections, intestinal diseases among others are factors that represent a low level of iron in the blood system and over time anemia occurs [7]. The Spirulina algae is composed of

- The central region containing the nucleic acid,
- The peripheral region, is constituted by cytoplasmic structures and is responsible for the process of photosynthesis and
- The cell wall formed by peptidoglycan [8].

Spirulina represents a source of natural iron source and can be found in green foods, meat and cereals [9]. In 2002, it was determined that it can be used in treatments against anemia, cancer, hepatotoxicity, cardiovascular problems, high cholesterol and triglycerides, hypoglycemia, chronic fatigue and ulcers [10]. This was attributed to the fact that Spirulina has 60% protein in addition to amino acids, vitamins (A, B1, B2, B3, B6, B9, C, D, E), minerals (calcium, copper, chromium, phosphorus, iron, magnesium, manganese, potassium, selenium, sodium, iodine and zinc) as well as 15-20% carbohydrates [11-12].

On the other hand, although there is no universal methodology, there are several techniques of physicochemical analysis for the study of adsorption of drugs like DSC, DTA and DTG, however, the main techniques of spectroscopic analysis (FTIR, XRD and NMR) allow an analysis Qualitative and quantitative in the development of release systems [13]. FTIR and RAMAN techniques have become increasingly important in pharmaceutical and biopharmaceutical applications from physicochemical characterization to kinetic analysis in drug adsorption and release to determine drug performance. FTIR allows the study of adsorption and release of semi-solid formulations, pellets, transdermal systems, as well as *in vivo* studies [14]. Thus, the objective of the investigation was to analyze the adsorption of Spirulina algae in hydrogels of chitosan using FTIR spectroscopy and DRX to establish the bases for a transdermal release system.

## Methodology

### Materials

Chitosan from shrimp shells degree of deacetylated  $\geq 75\%$ , Aldrich), Acetic acid (PM 60.05gr/gmol, J.T. Baker), Glutaraldehyde 25% (J.T. Baker), Methanol (anhydrous 99.8%, Aldrich).

### Methods

**Chitosan crosslinking:** The crosslinking of the chitosan was first carried out by preparing a 1 and 3%w/v solution of chitosan in 2%v/v acetic acid, then agitation was carried out for 5mins. For a subsequent rest of 24hrs. In petri dishes [15,16]. Cross-linking was carried out for 24h and various concentrations of glutaraldehyde were used from 3-10%, then methanol was added to remove unreacted reactive groups, then washed with distilled water and finally placed in an oven at 25°C for 24h [15]. The degree of swelling (SR) was calculated by measuring the weights before ( $W_0$ ) and after ( $W_s$ ) of the addition of water using the equation (1):  $SR = [(W_s - W_0) / W_0] * 100$  [17].

**Adsorption of Spirulina algae:** The dried gels were placed in a solution of water and Spirulina algae in different proportions for 24h under constant agitation and the percentages of swelling were subsequently determined.

**FTIR:** FTIR were recorded using a NICOLET iS10 spectrometer. The freeze-dried hydrogels were ground into powders, blended with dry spectroscopic grade KBr powders, and pressed into small disks for FTIR measurements. Samples were scanned a resolution of 2cm<sup>-1</sup> in the scan range of 400–4000cm<sup>-1</sup>.

**DRX:** X-ray diffraction patterns of the composite films were measured using an X-ray diffractometer (model D8 Advance; Bruker, Germany) with Ni-filtered Cu radiation generated at 30 kV and 30mA as the X-ray source. The diffraction patterns were determined over a range of diffraction angles  $2\theta = 5^\circ$  to  $70^\circ$  and a step of  $0.060^\circ$  ( $2\theta$ ).

## Results

### Spirulina algae

Figure 1 show the FTIR of pure Spirulina algae where the vibrations at 3560–3500cm<sup>-1</sup> were attributed to the OH stretching of carbohydrates and amino acids [18] in addition to the presence of alcohols and phenols [19]. In the range of 3500–3300cm<sup>-1</sup> the presence of the NH stretching vibration of the secondary amines corresponding to the protein and lipids was observed while from 3000 to 2850cm<sup>-1</sup> the aliquot CH stretching vibration of the alkenes. The frequency ranges 3300–2500cm<sup>-1</sup> represent the presence of the aliphatic OH stretching vibration of the carboxylic falls. With respect to the signals of 2260–2100cm<sup>-1</sup> was assigned to the triple bond C–C of the alkynes [20]. The vibration of the carbonyl group (C=O) was located between 1750–1735cm<sup>-1</sup>. From 1680–1640cm<sup>-1</sup> corresponded to the stretching vibration of the C=C bond whereas the NH flexion of the ketone appeared between 1650–1580cm<sup>-1</sup> and the flexion of the CH<sub>2</sub> group was observed between 1435–1405cm<sup>-1</sup>, respectively [18]. The range of 1550 to 1475cm<sup>-1</sup> was observed asymmetric stretching N–O corresponding to the nitrogenous compounds of the alga [19]. The frequencies attributed to the C–O stretching vibrations and the O–H flexions were appreciated between 1350–1260cm<sup>-1</sup>.

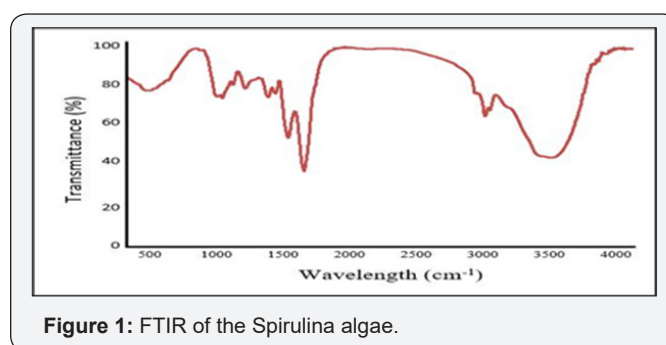


Figure 1: FTIR of the Spirulina algae.

The presence of the C–O and O–H asymmetric stretching of the COC group and the symmetrical stretching between 1120–1030cm<sup>-1</sup> of the esters, carboxylic acids, ethers and alcohols [18], the C–N bond characteristic of the aliphatic amines was located between 1250–1020cm<sup>-1</sup>; while the antioxidant enzymes present in the alga were located from 1050 to 1010cm<sup>-1</sup> attributed to symmetrical stretching of SO<sub>3</sub> as well as ionic sulfonates. From 700 to 620cm<sup>-1</sup> the SO vibration of the sulfonic compounds was appreciated and the N–H vibrations of the primary and secondary amines were found between 910–665cm<sup>-1</sup>. Finally, the C–Br bond due to the presence of the alkyl halide compounds were determined in the area between 690–515cm<sup>-1</sup> [19,20].

### Adsorption of spirulina algae in chitosan hydrogels

The percentages of swelling using the equation 1 were 25 and 26% respectively. Figure 2 shows that in the range of 4000–3400cm<sup>-1</sup> there is a wide overlap of the OH stretching vibrations of the water present in the hydrogel, as well as the NH stretching of the primaries amines. Stretching bands at 1800–17700cm<sup>-1</sup>

(asymmetrical and symmetrical) corresponded to carbonyl groups (C=O) of primary amides. The bands at 1550–1600 $\text{cm}^{-1}$  were attributed at the stretching vibrations of secondary amides (NH). Between 1330 and 1250 $\text{cm}^{-1}$  was assigned to free CO bound, meantime the stretching vibrations at 1100 $\text{cm}^{-1}$  represented to CC/CO relation of polysaccharides ethers. The range of 1200–1100 $\text{cm}^{-1}$  were observed the hydroxyl bands [21]. Stretching of the secondary amine appeared between 3250–3000 $\text{cm}^{-1}$ .

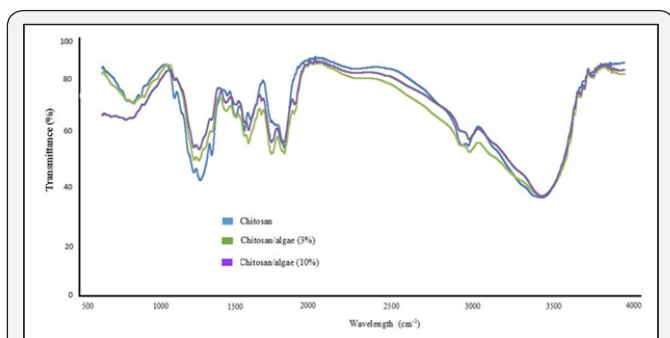


Figure 2: FTIR of the adsorption of the Spirulina algae in chitosan hydrogels.

The area between 2900–2800 $\text{cm}^{-1}$  shows a decrease in the CH (symmetrical and asymmetric) stretches of the hydrogels that are replaced by the amino acids of the Spirulina algae during adsorption at different concentrations. Halogenated compounds were located at 1400 $\text{cm}^{-1}$  was assigned to the C–F and 1000–750 $\text{cm}^{-1}$  stretching corresponding to the C–Br and C–I stretches. The fingerprint region of chitosan decreases with respect to the algae increase. This is attributed to the hydrogen bonds and van der Waals interactions of the hydroxyl and carboxylic groups of algae and the amino groups of chitosan.

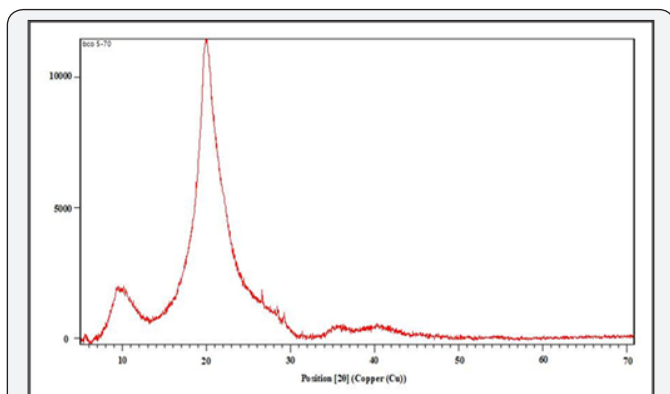


Figure 3: FTIR of the adsorption of the Spirulina algae (10%) in chitosan hydrogels.

Figure 3 shows two peaks at 9–11° and 20° on the 2θ scale which indicate that the adsorption of the algae is carried out on the surface of the chitosan because if there were a low compatibility the crystalline peaks of each material, in addition, we observed the formation of the amorphous phase indicating that there is a consistency with the FTIR of Figure 2 [22].

## Conclusion

Chitosan-based hydrogels can absorb different drugs to develop controlled release systems via transdermal. Greater swelling was observed in hydrogels with 3% chitosan and 10% glutaraldehyde due to an increase in the cross-linking that allows greater water adsorption. A percentage of swelling was obtained between 25–26%. FTIR spectroscopy allowed the observation that the adsorption of amine groups from the Spirulina algae in the fingerprint zone is also verified by the formation of an amorphous phase.

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