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Title: Synthesis of biopolymer from chitosan/xanthan

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Editorial label ECORFAN: 607-8324
BCIE Control Number: 2016-01
BCIE Classification (2016): 221116-0101

Pages: 14

RNA: 03-2010-032610115700-14

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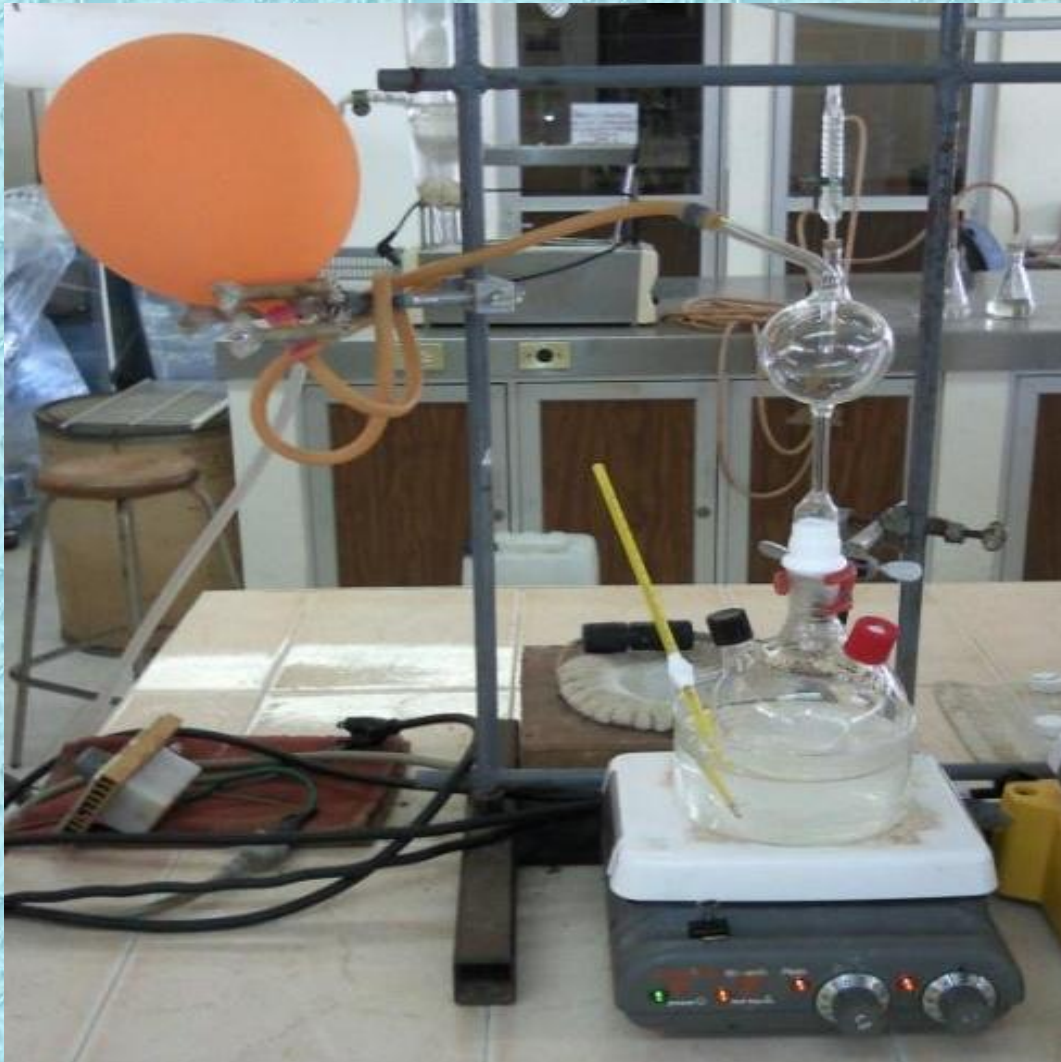
INTRODUCTION

Water pollution by heavy metals is a serious and hard problem to the ecology and health. Biosorption is a process used for metal removed, and several materials have been studied as adsorbents, particularly chitosan (Lee and col.,2001), because it has been found that hydrogels of this material improve the adsorption capacity to metal ions. The present work presented the synthesis of Chitosan (Q) with Xanthan (X) for obtained gels, using a cross-linking agent, in this case glutaraldehyde (GA), to concentrations of 0.5, 1 y 1.5% w/w of the mass total. Also, were studied three Q/X ratios 75/25, 50/50 and 75/25, and were analyzed by FTIR Spectroscopy to determine functional groups in xerogels, besides the determination of the water content in the materials.

EXPERIMENTAL

1500 mg total sample was used to prepare biopolymers Q/X. For the synthesis of hydrogels, solutions of each polymer with a final concentration of 0.65% by weight, and solvent for the chitosan powder was a solution of acetic acid [0.4 M] were used, while for xanthan, water was used deionized. It was stirred at 200 rpm, at a temperature of 35 ° C for 2 h until completely dissolved. The solution was injected xanthan drop wise in chitosan, with the help of an insulin syringe (in order to keep the size of the formed sphere constant) all with slow stirring for 2 h. Both polymers were allowed to interact, for 2 h after complete addition of the xanthan to chitosan solution, to complete formation of the physical hydrogel. To make the crosslinking reaction, the temperature was raised to 70 ° C and stable once rose, glutaraldehyde solutions (GA) and catalyst HCl (0.5, 1.0 and 5% by weight) were added, and allowed to react for a period 3 h, while maintaining an inert atmosphere and constant stirring. After this time, we proceeded to filtering hydrogels and washed with deionized water until a neutral pH. The spheres were placed in a petri plastic box, which took a stove at 40 ° C for 48 h, for drying.

EXPERIMENTAL



Design of Experiments

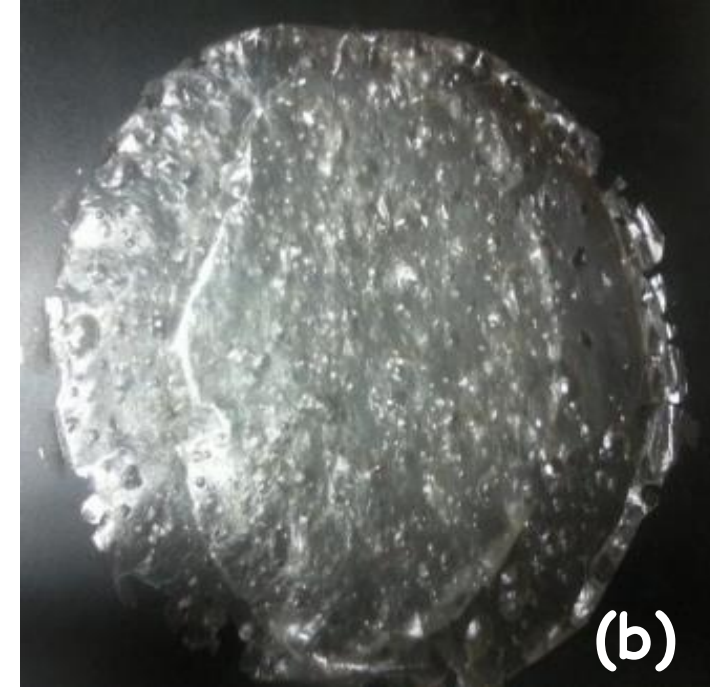
<i>Q / X Ratio</i>	<i>Catalyst (HCl) [1x10⁻² M]</i>		
	<i>Crosslinker (GA) [2.5x10⁻³ M]</i>		
	<i>[0.5 %]</i>	<i>[1.0 %]</i>	<i>[5 %]</i>
<i>50/50</i>	QX ₁₁	QX ₁₂	QX ₁₃
<i>67/33*</i>	QX ₂₁	QX ₂₂	QX ₂₃
<i>75/25</i>	QX ₃₁	QX ₃₂	QX ₃₃

* Ratio Q/X according to literature

RESULTS

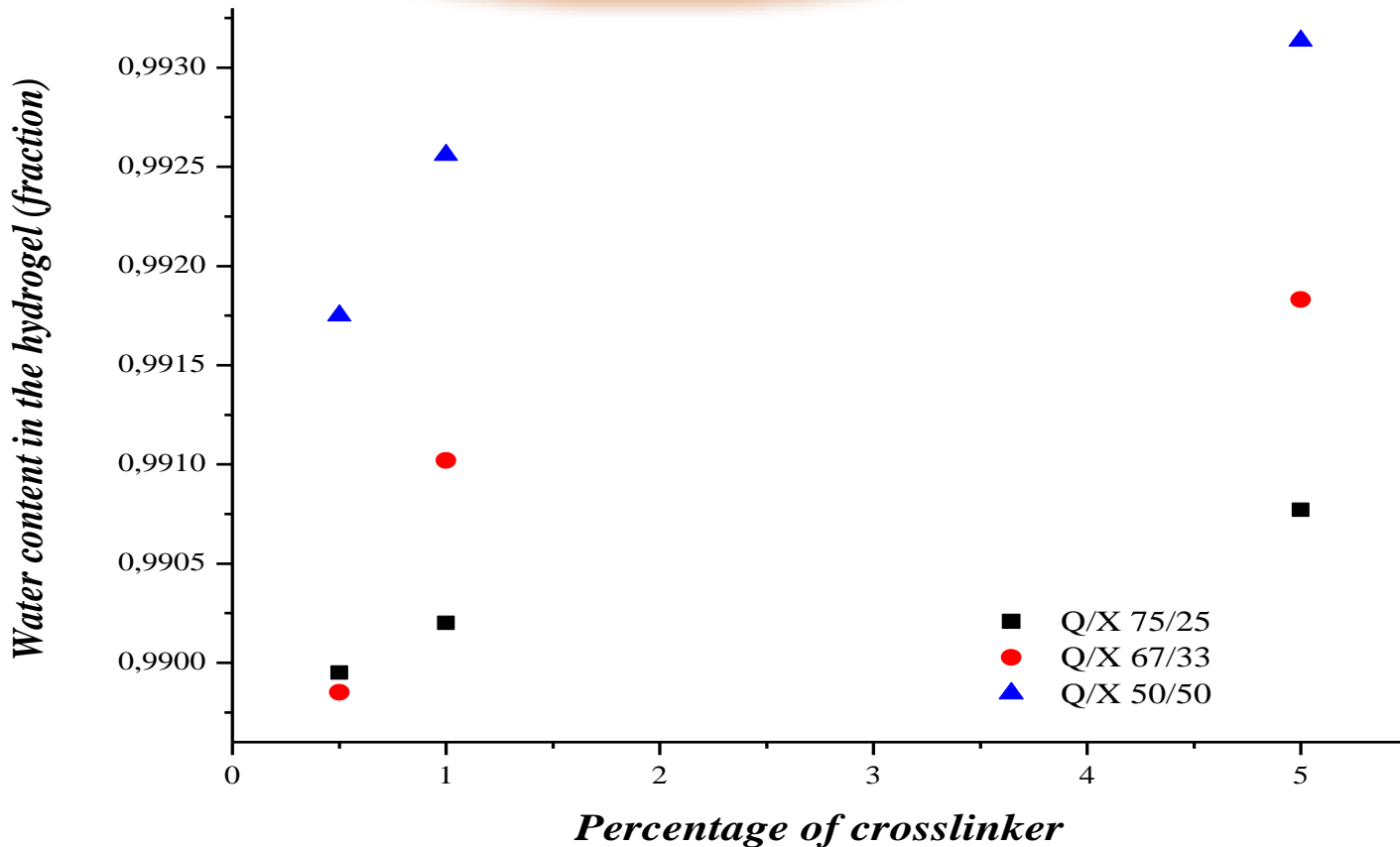
Hydrogel showed uniform appearance and was soft to the touch; physical appearance can be seen in Figure (a), which shows the hydrogel with 50/50 ratio and a concentration of 5% GA.

Once in xerogel form, after drying for 48 h at 40 °C, the surface showed a nonuniform formed, thin and somewhat brittle, rough appearance due to air bubbles formed during drying. Its physical appearance is presented in Figure (b).



*Biopolymer Q/X, 50/50 and 5% crosslinker
(a) hydrogel and (b) xerogel*

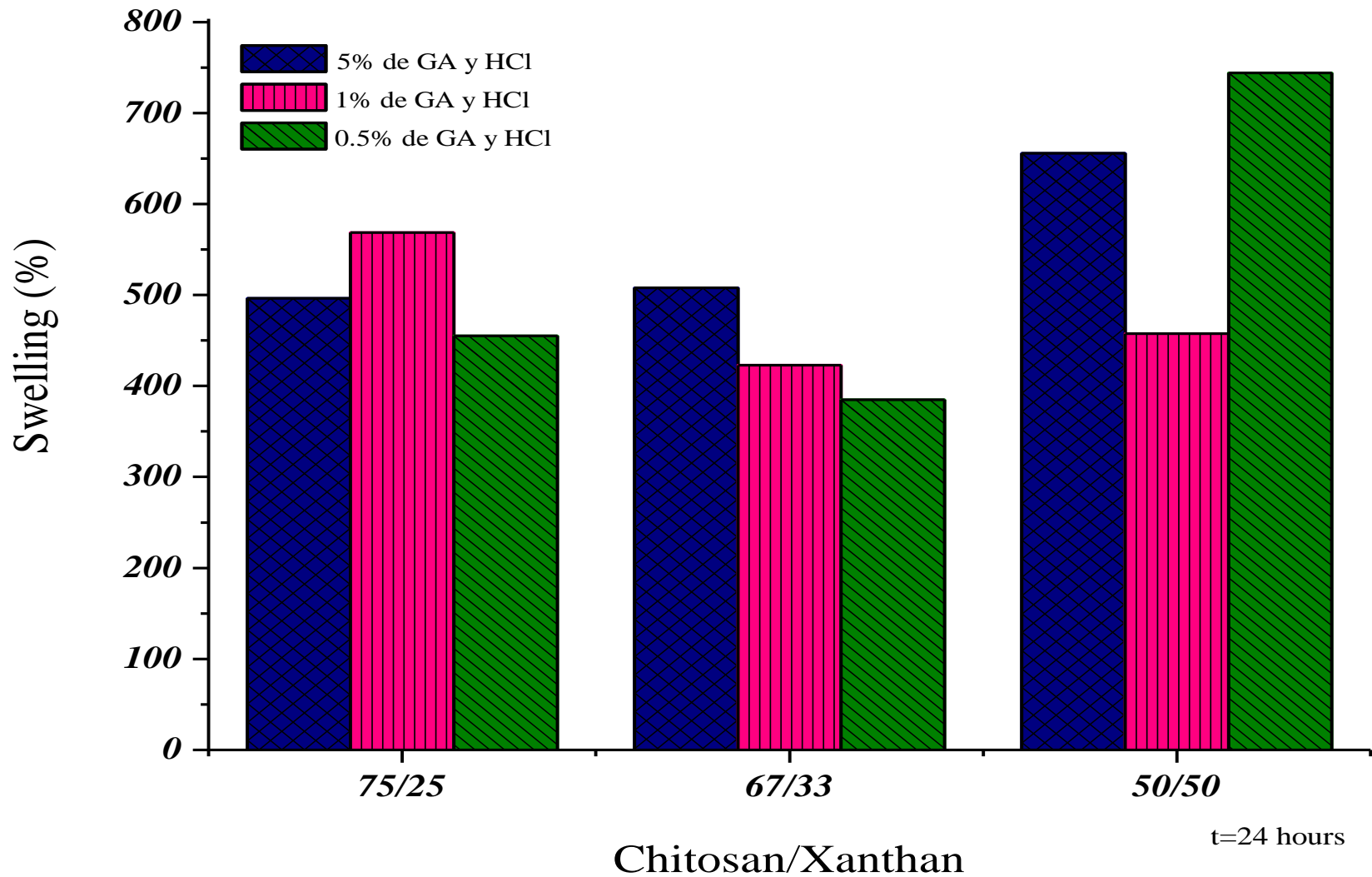
RESULTS



Effect of crosslinker on the values of the water fraction by weight of Q/X hydrogels, the three ratio polymers studied: 75/25, 67/33 and 50/50

This phenomenon can be explained considering the helical structure of xanthan, assuming that during the crosslinking reaction and the effect of high temperature, this structure is destroyed and therefore a part of the hydroxyl groups which stabilize this helical structure by hydrogen bonding take part in the reaction and another part was available to interact with the water during the swelling process. Thus a network that the initial, less compact is created with spaces in the larger network, which allow increased water absorption. The values of the determinations of water fractions are high and range between 98 and 99% in water content, having some significant changes, these percentages between the studied relations of both polymers in the network.

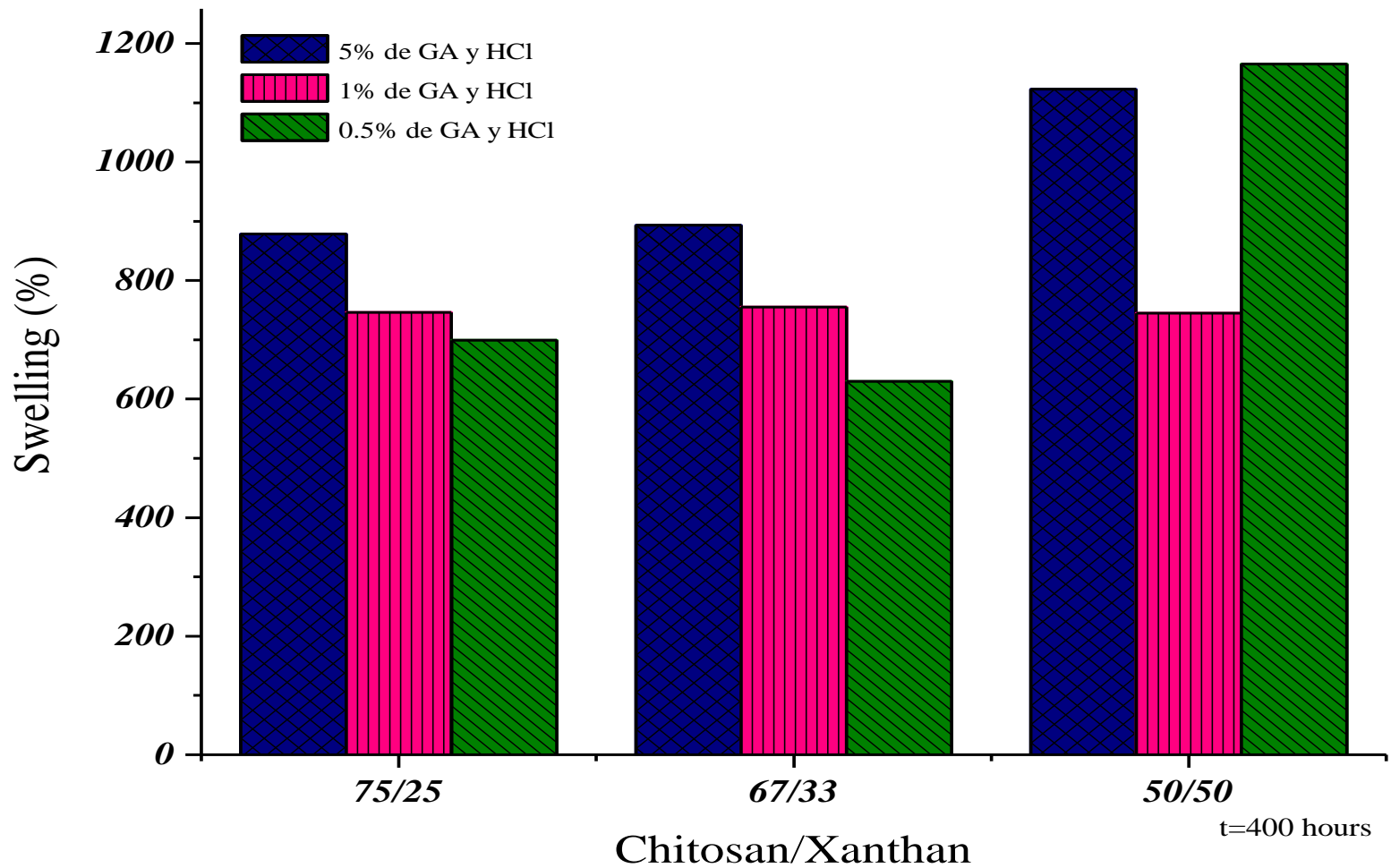
RESULTS



Swelling of the hydrogel is carried out the diffusion of water into the polymer matrix, this process involves the migration of water with preexisting spaces formed between the polymer chains.

Graphic shows the percentages of swelling at 24 h of hydrogels three ratio biopolymer Chitosan/Xanthan and three concentrations of glutaraldehyde.

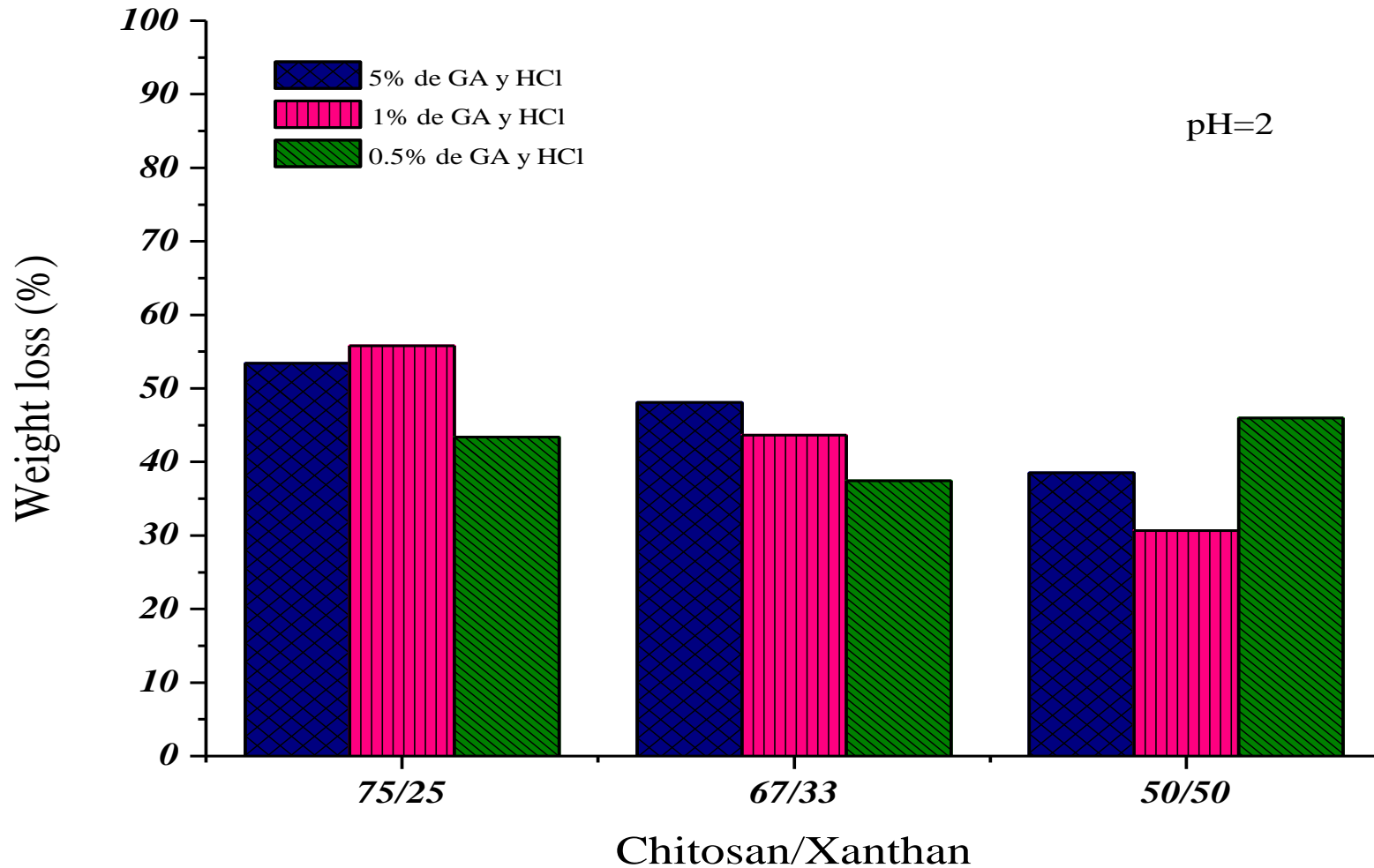
RESULTS



Graphic shows percentages of swelling of hydrogels in time of 400 h for each ratio Q/X, at concentrations of GA studied. For the 67/33 ratio remains the trend observed in the previous graph, the largest percentage of swelling hydrogel for 5% to 893% of fluid retention, followed by crosslinked materials with 1 and 0.5%.

That is, it is possible that the large number of ionizable groups present in xanthan, suffer repulsions, for example between COO- groups, increasing the spaces in the hydrogel network, leading to increased swelling.

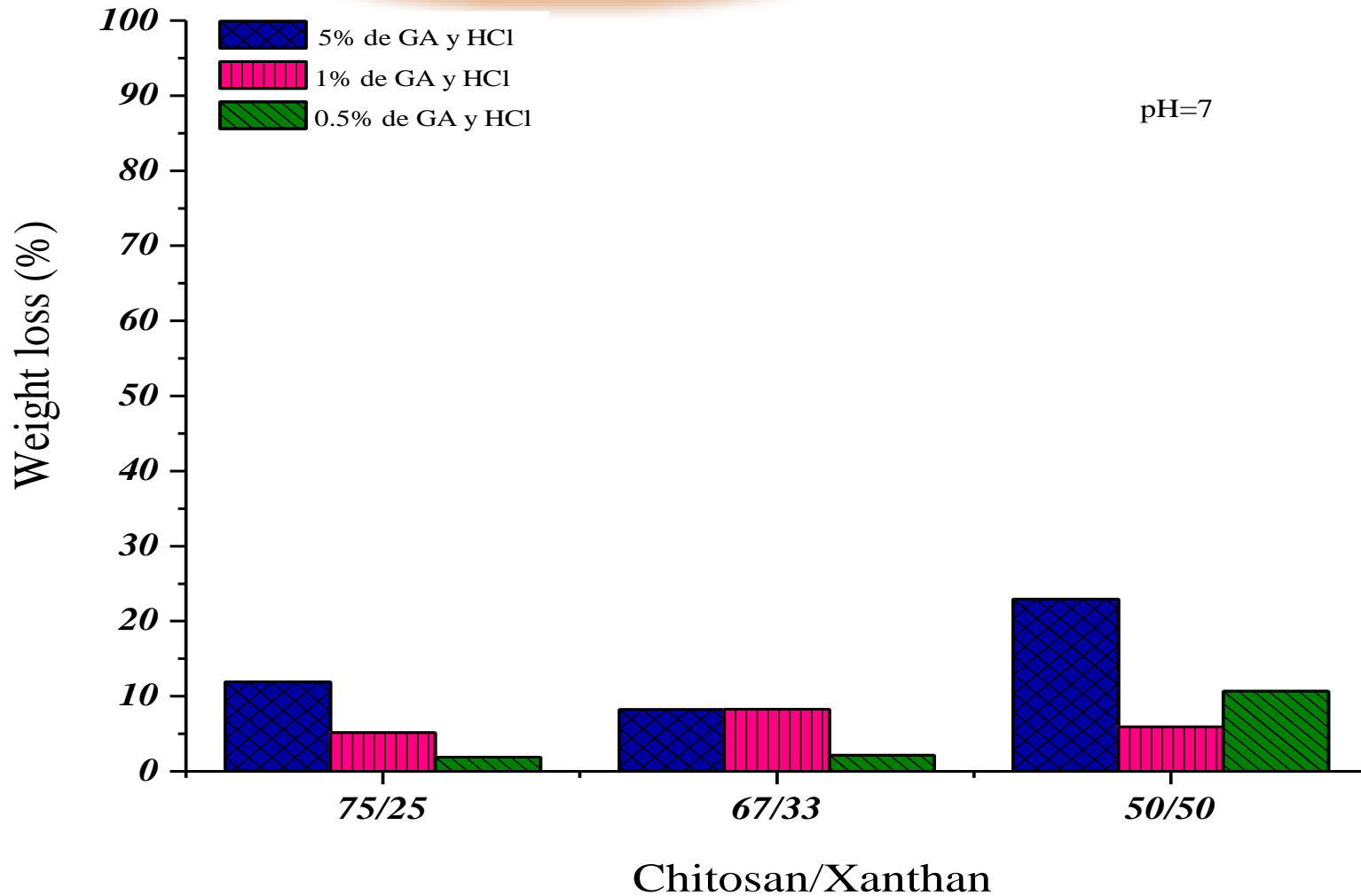
RESULTS



Solubility test. Xerogels obtained was subjected to tests solubility in different media: basic and neutral acid, to evaluate their behavior and physical endurance in different pH ranges.

Graphic 4 shows the percentage weight loss hydrogels, after 24 h of being in contact with the solution of pH = 2, showing that high values of weight loss in the material is obtained, having higher values of chitosan content higher, reaching up to 55% loss ratio 75/25.

RESULTS

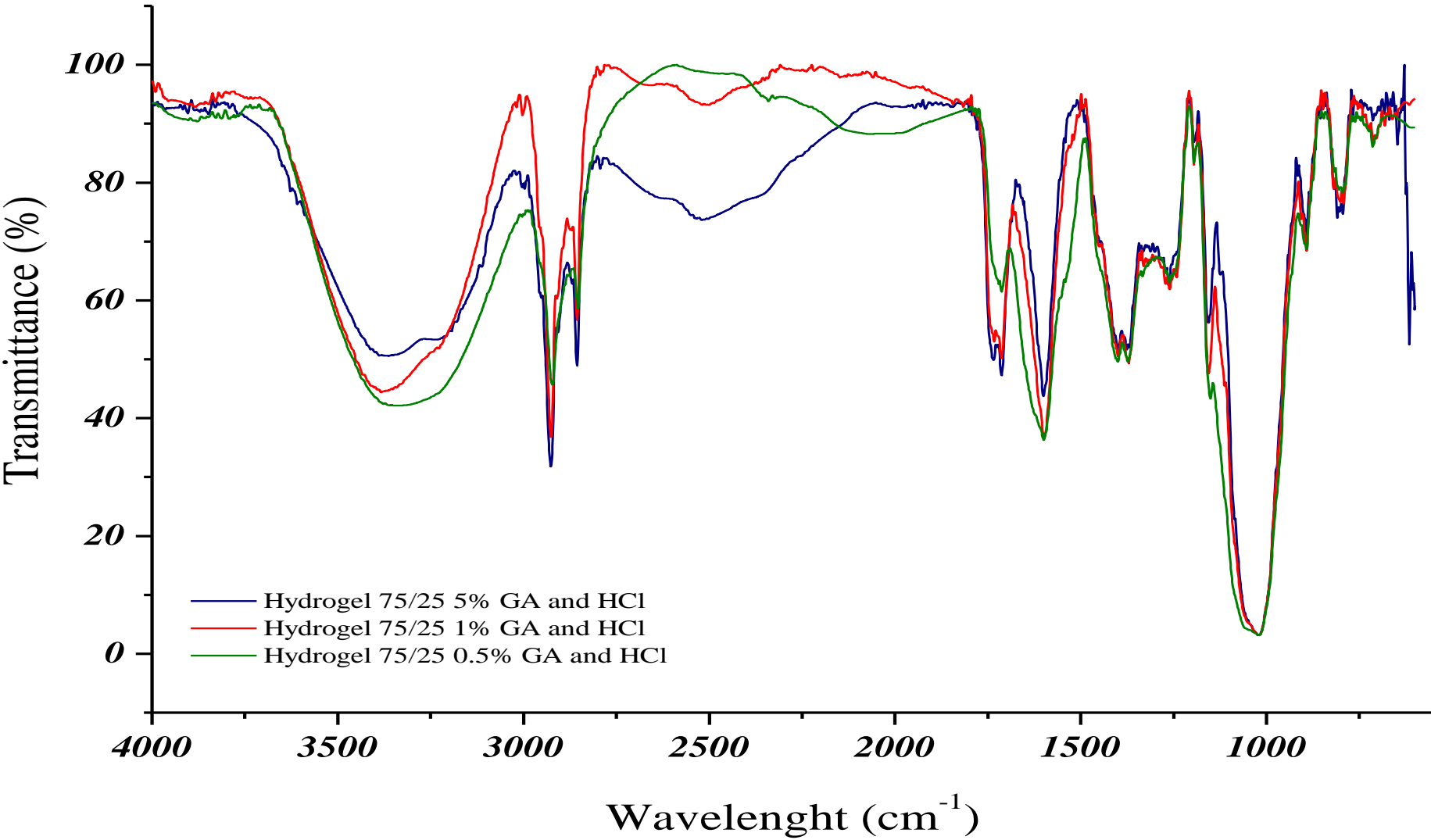


Graphic presents the percentages of weight loss in the hydrogels after 24 h of being in contact with the solution of pH 7. It can be seen as the percentages are much lower compared to thrown to pH 2. The hydrogel which present the highest value was the highest content of Xanthan i.e. 50/50, with 22% loss of material.

A basic pH was not possible to record the weights for the material after 24 hours in contact with the solution pH 10, as they lost their properties and resistance, mostly solubilized. This can be attributed to that between, basic those corresponding to xanthan, carboxylic groups pass carboxylate groups, and because of repulsion between them and the environment, the hydrogel network relaxes completely, allowing more liquid inlet until solubilize completely.

RESULTS

Infrared Spectroscopy Fourier Transform (FTIR)



FTIR spectrum for the hydrogel Q/X ratio 75/25 at three concentrations of HCl and crosslinker agent (0.5, 1 and 5%) is presented. In the three spectra a broad band between 3600-300 cm⁻¹, corresponding to stretching vibration characteristics of the O-H and N-H groups present in both polysaccharides are presented.

RESULTS

The peaks present at 2918 and 2855 cm^{-1} are due to vibrations of symmetric and asymmetric stretching of C-H bonds, while the signals present at 2500 cm^{-1} , may correspond to stretching vibrations of bonds N-H. The peaks located at 1370 cm^{-1} and 1259 cm^{-1} , attributable to deformation vibrations of the OH groups and the peak at 1406 cm^{-1} located deformation vibration of C-H bonds. The weaker signals present at 793 cm^{-1} and 713 cm^{-1} , due to vibrations shake N-H bonds and C-H respectively.

The signal located at 1630 cm^{-1} may be attributed to stretching vibration of C=N, corresponding to the formation of the imino groups during the crosslinking reaction. Baroni in 2008 reports this signal around 1655 cm^{-1} in its work on chitosan membranes crosslinked with glutaraldehyde, used for adsorption of chromium ions. The signal located between 1100 cm^{-1} and 1000 cm^{-1} corresponds to vibrations of symmetrical and asymmetrical stretching of the bonds C-O-C.

CONCLUSIONS

- ❑ *Biopolymer synthesis chitosan-xanthan was performed, in which thin, flexible, transparent films were obtained, regardless of the ratio of Q/X used. Determining the water content in the material, as a fraction of water, high values between 0.98 and 0.99 was obtained, which classifies it as a hydrogel high hydration, wherein the maximum value more content ratio of xanthan (50/50) and the highest percentage of glutaraldehyde (5%).*
- ❑ *Swelling tests showed the highest percentages for the 50/50 ratio with 5% and 0.5% glutaraldehyde, reaching values of 1123 and 1165% respectively, which may favor when used on adsorption of metal ions facilitating the diffusion process in the hydrogel.*

CONCLUSIONS

□ The solubility tests performed show that all materials have good properties, when their physical resistance to pH 2 and the neutral, since no solubilized, not so for the pH 10, as loss of material was. A suitable range for the use of materials in serious adsorption tests 4 to 8, still more favorably pH 7, using the relative 75/25 hydrogel with 5% crosslinker because it presented the least loss of material, one testing in a neutral context, with a value of 1.88%.

□ By FTIR, the better groups present on both polymers were determined in the network, such as absorptions present of OH, C=O, NH, N=H, CH₂ and CO groups in which a greater intensity in the corresponding peaks in the region of the carbonyl group and CH bonds, as the concentration of crosslinking agent is increased, which can be attributed to the aldehyde group and the signal around 1630 cm⁻¹, may correspond to a imino bond, reaction product of chitosan with glutaraldehyde. Spectra is equally materials containing functional groups still present N atoms, which means sites still available to take place a good adsorption process with metal ions.

REFERENCES

- Bailey, S. E., Olin, T. J., Bricka, R. M., & Adrian, D.D. (1999). A review of potentially low-cost sorbents for heavy metals. *Water Research*, 33, (11), 2469-2479.
- Baroni, P., Vieira, R.S., Meneguetti, E., Da Silva, M.G.C., & Beppu, M.M. (2008). Evaluation of batch adsorption of chromium ion on natural and crosslinked chitosan membranes. *Journal of hazardous materials*, 152, 1157.
- Crini, Grégorio. (2005). Recent developments in polysaccharide-based materials used as adsorbents in wastewater treatment. *Progress in Polymer Science*, 30, 38-70.
- Katime Amashta, I., Katime Trabanca, O., & Katime Trabanca, D. (2003). Los materiales inteligentes de este milenio: Los hidrogeles macromoleculares. Síntesis, propiedades y aplicaciones. *Servicio Editorial de la Universidad del País Vasco (UPV/EHU)*. España.
- Li, N., & Bai, R. (2005). Copper adsorption on chitosan-cellulose hydrogel beads: behaviors and mechanisms. *Separation and Purification Technology*, 42, (3), 237-247.
- Martínez-Rubalcaba, A., Chornet, E., & Rodríguez, D. (2007). Viscoelastic properties of dispersed chitosan/xanthan hydrogels. *Carbohydrate Polymers*, 67, (4), 586-595.
- Park, J. S., Park, J. W., & Ruckenstein, E. (2000). Thermal and dynamic mechanical analysis of PVA/MC blends hydrogels. *Polymer*, 42, 4271-4273.
- Rubio Cruz, Ernesto F. (2009). Estudio comparativo de materiales biocompatibles sintetizados a partir de gomas vegetales, goma biosintética e hidroxietilcelulosa. *Tesis de Doctorado en Ciencias en Ingeniería Química*, Instituto Tecnológico de Ciudad Madero. México.



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