Effect of solvent and temperature in the preparation of zinc(II) complex of biopolymeric Schiff bases

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Introduction

Schiff base complexes may exhibit anti-tumor, anti-bacterial, anti-viral, anti-fungical activities [1]. The complexes has attracted once they allow the formation of bidentate ligants with donor species of electrons, nitrogen and oxygen atmos [2], usually in complexation with different metals.
The Schiff base complex chitosan is biodegradable, biocompatible, low toxicity, mucoadhesive and so attractive to be used in complexes for biological uses as antimicrobial activitive [3]. The compounds obtained were characterized by hydrogen nuclear magnetic resonance spectroscopy of (H^1NMR), thermogravimetry (TGA), differential termal analysis (DTA).

Results ans Discussion

Synthesis of the Schiff base was performed in ethanol at 55 ℃ for 18 hours. The structural formu la of the compound is ilustraded in Figure 1. The degree of substitution was 49,13% determined from ^{1}H NMR. The results obtained by thermogravimetric curves (TG) in air atmosphere for chitosan purified and substituted with salicylaldehyde are presented in the Table 1. It were observed three thermal events, the first one associated with dehydration and release of volatiles, followed by two steps of decomposition and subsequent generation of carbonized materials. The modified chitosan presented the degradation onset temperature near to 184 ℃, while the degradation of purified chitosan is 175 ℃.

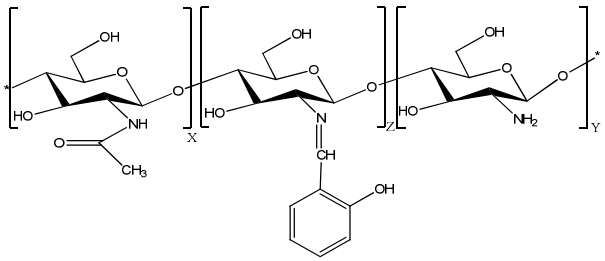

Figure 1. Structural formula of the biopolymeric Schiff base on chitosan.

The synthesis of Zn(II) complexes was performed in different conditions aiming to obtain the highest extension of complexation. The influence of reaction parameters, as well as the results are presented in the Table 2.

Table 1. Results of curves TG curves for purified chitosan (QP) and substituted (QS)

Polymer	Δm_1(%) ΔT(°C)	Δm_2(%) ΔT(°C)	Δm_3(%) ΔT(°C)	Δm_3(%) /Δm_2(%)
QP	7.9 22.3 – 175.3	46.5 175.3 – 392.4	45.6 392.4 – 629.6	0.98
QS	4.8 21.1 – 184.8	39.9 184.8 – 384.2	55.1 384.2 – 638.5	1.38

Table 2. Results of residue percentages obtained in diferent experimental conditions

Solvent	Temperature (°C)	Residue (%) at 1000 °C	Calculated residue (%)	Reaction time (h)	Molar ratio Base: Metal
Isopropanol	60	4.59	4.96	8	1:0.5
	40	5.54	6.12	8	1:0.5
Methanol	60	4.89	5.22	8	1:0.5
	40	5.01	5.41	8	1:0.5
Ethanol	60	2.80	2.92	4	1:0.5
	40	5.94	6.28	8	1:0.5
	40	7.50	8.13	8	1:1

Conclusion

The thermal stability of the polymer substituted with salicylaldehyde is higher than the purified chitosan. Thermal analysis data showed that the higher concentration of Zn^{2+} with chitosan biopolymeric Schiff base is achieved after 8 hours at 40 ºC, in ethanolic media, resulting in better yeld.

Acknowledgments

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