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THE POSSIBILITY OF USING EXPERIMENTAL EQUATIONS TO CALCULATE THE DEGREE OF DEACETYLATION OF CHITOSAN

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INTRODUCTION

Chitosan and its potential for wide industrial applications have been intensively studied in recent years. The degree of deacetylation (DD) is a chemical characteristic of importance for the physical and biological properties of chitosan, on which the performance crucial for its application largely depends. In this work, three samples of chitosan with different viscosity and a known degree of deacetylation (between 75 and 85%) were used in order to examine the possibility of applying the experimental equations reported in the literature to determine the degree of deacetylation of chitosan in laboratory conditions.

MATERIAL AND METHOD

Comercial chitosan with high viscosity CHV, medium viscosity CMV and low viscosity CLV with degree of deacetylation between 75 and 85% was used. Methods for testing degree od deacetylation were:

Acid-base titration: Chitosan was dissolved in 0.1 M HCl. Methyl orange was added as an indicator. 0.1 M NaOH was used to titrate the solution. The percentage of free amino groups in chitosan was calculated as follows $NH_2\%=[(C_1V_1-C_2V_2)\times0.0016]/[G\ (1-w)]\times100$. Free NH2%= NH2% /9.94% ×100%.Theoretical value of NH2% for chitosan = (16/161)×100% = 9.94%, where C1: HCl concentration (M)C2: concentration of NaOH (M)V1: volume of HCl added (ml)V2: volume of NaON used for titration (ml)G: mass of weighed sample (g)w: sample moisture content (%)0.0016: equivalent to the content of NH2 (g) in 1 ml of 1M HCl

Potentiometric titration

Chitosan was dissolved in 0.3N HCll and the solution was titrated with a standard solution of 0.1 N NaOH. A pH titration curve was generated in relation to the volume of NaOH used for the titration. Free amino groups percentage in chitosan was calculated: NH2% = 16.1×(y-x)/M, where:M: sample mass (g); y:first inflection point from the graph; x:second inflection point from the graph

Infrared spectrophotometry

Using FTIR Spectrophotometer, model "Nicolet iS10“ spectra of cast films was recorded, and apsorbances on selected wavelengths determined. For DD determination five reported experimental equasions were used: 1)DA= (A1655/A3450)×100/1.33; 2) DA=(A1655/A3450)×15 ; 3) A1320/A3450= 0.03146 + 0.00226× DA ; 4) A1320/A1420= 0.3822+ 0.03133× DA ; 5) A1560/A2875= 0.0125× DA + 0.2

DISCUSSION

The results obtained using acid-base titration and potentiometric titration to determine the degree of deacetylation of the tested samples are shown in Table 1. It can be seen that the results obtained for all three examined chitosan samples using acid-base titration are in the expected range of values for the degree of deacetylation, while potentiometric titration did not give expected results, Using FTIR method (Table 2), the results showed that using the experimental formula number 4 was appropriate, while for the other reported equasions the obtained values varied significantly both within the same sample and in comparison with other samples. In addition to the significant deviation of the values from the expected ones, the reproducibility of the values was also not achieved, therefore in this case the other experimental equations cannot be considered adequate for calculating the degree of deacetylation.

Sample	DD	
	Acid-base titration	Potentiometric titration
CLV	77.8%	46.2%
CMV	76.6%	/
CHV	81.1%	42.5%

Table 1. Deacetylation degree of tested samples using diferent titration methods

	DD				
Sample	1	2	3	4	5
CLV	80.87	70.74	-32.69	82.89	-356
CMV	19.37	-23.33	-714.98	83.84	-55.98
CHV	64.44	45.61	-280.72	86.82	-441.33

Table 2. Deacetylation degree of tested samples using different experimental equasions

CONCLUSION

Based on the results obtained in this paper, it was shown that acceptable methods for assessing the degree of deacetylation are the acid-base titration method and the infrared spectrophotometry method. What should be taken into account when choosing a method is that when it comes to the acid-base titration method, it is an instrumentally simple, fast and economical method that is also suitable for less equipped laboratories, while the infrared spectrophotometry method is relatively fast, but instrumentally more demanding,

Key words: *chitosan, deacetylation degree, experimental equations*

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