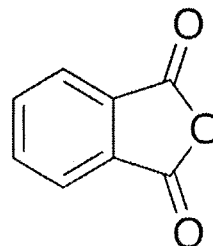


APPENDIX

Phthalic anhydride [91]Formula: $C_8H_4O_3$

Molecular structure:



Molecular Weight: 148.12 g/mol

Synonyms: Phthalic acid anhydride
1,3-Isobenzofurandione
Isobenzofuran-1,3-dione
1,2-Benzenedicarboxylic acid anhydride
1,2-Benzenedicarboxylic anhydride
1,3-Dihydro-1,3-dioxoisobenzofurane
1,3-Dioxophthalane
1,3-Phthalandione
Phthalandione

Physical state: White flakes or needles

Melting point: 131.6 °C

Boiling point: 284.5 °C

Density: 1.527 g/cm³

Vapor density: 6.6 (vs air)

Flash point: 152 °C

Solubility: 0.62 g/100 mL water at 20 °C

Very slightly soluble in cold water

Soluble in alcohol

Soluble in ether

Toxicity: Oral rat LD₅₀: 4020 mg/kgInhalation rat LC₅₀: > 210 mg/m³/1 hSkin rabbit LD₅₀: > 10 gm/k

Example: The Evaluation of degree of substitution (DS)

FTIR method

The DS of N-PhCS prepared at 25°C under stirring for 4 h, CS:PA at 1:5 mole ratio and neutralization pH at pH 5 by calculation from the ratio of absorbance at 1645 cm⁻¹ (amide I band) and the hydroxyl band at 3450 cm⁻¹ of FTIR spectra of N-PhCS (Figure 34a) and chitosan (Figure 34b). The degree of substitution was calculated using the following Equation (1):

$$\%DS \text{ of N - PhCS} = \left(\frac{A_{1645}}{A_{3450}} \right)_{N-PhCS} - \left(\frac{A_{1645}}{A_{3450}} \right)_{CS} \times 100 \quad \dots\dots\dots(1)$$

$$\%DS \text{ of N - PhCS} = \left(\frac{0.090}{0.311} \right)_{N-PhCS} - \left(\frac{0.025}{0.200} \right)_{CS} \times 100$$

$$\%DS \text{ of N - PhCS} = 16.43$$

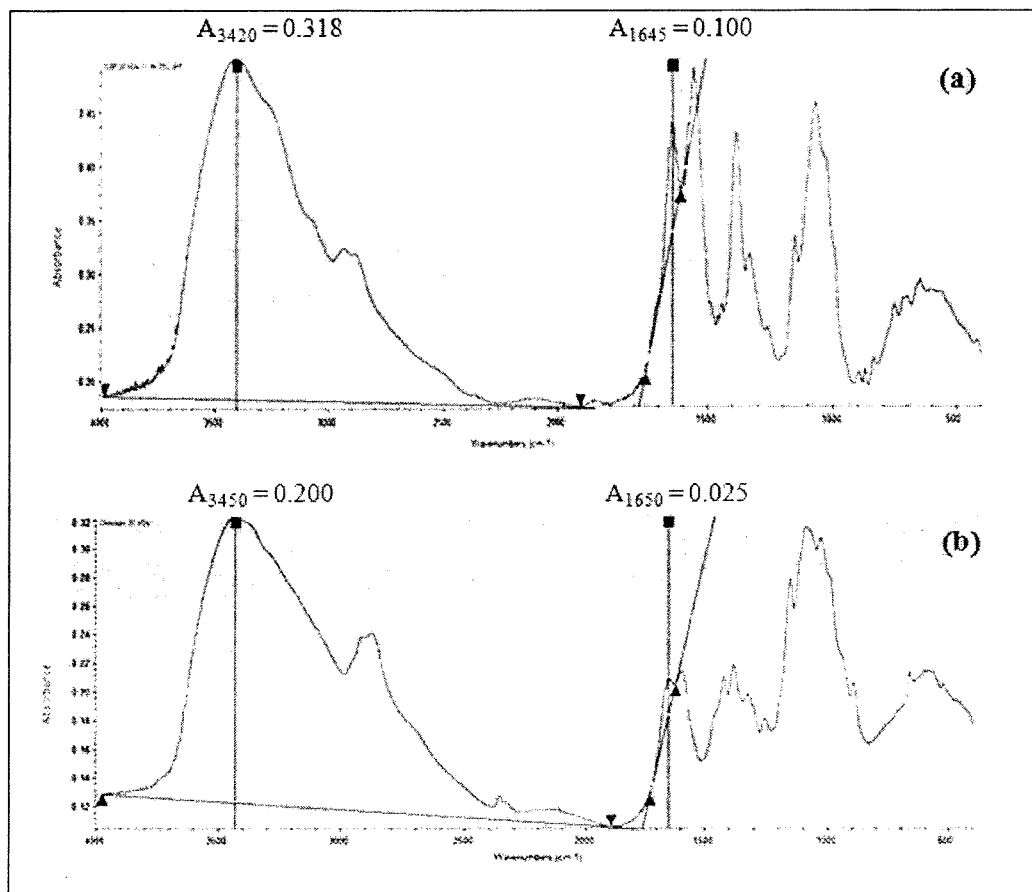


Figure 34 The FTIR spectra of (a) N-PhCS and (b) chitosan showing the baselines for calculating the amide I band absorbance for the ratio A_{1645}/A_{3420} .

¹H-NMR method

The degree of substitution of N-PhCS prepared at 25°C under stirring for 4 h, CS:PA at 1:5 mole ratio and neutralization pH at pH 5 by calculation from the ratio of area between the aromatic protons of phthaloyl group at 7.4-7.9 ppm and methyl proton of monosaccharide residue (H₃, H₄, H₅ and H₆) at 3.5-3.9 ppm (Figure 35). The degree of substitution was calculated using the following Equation (2):

$$\%DS \text{ of N - PhCS} = \left[\frac{\left(\frac{I_{\text{aromatic proton}}}{\text{number of proton}} \right)}{\left(\frac{I_{H_3+H_4+H_5+H_6}}{\text{number of proton}} \right)} \right] \times 100 \quad \dots\dots\dots(2)$$

$$\%DS \text{ of N - PhCS} = \left[\left(\frac{4.0}{4} \right) / \left(\frac{3.38 + 3.45}{5} \right) \right] \times 100$$

$$\%DS \text{ of N - PhCS} = 73.2\%$$

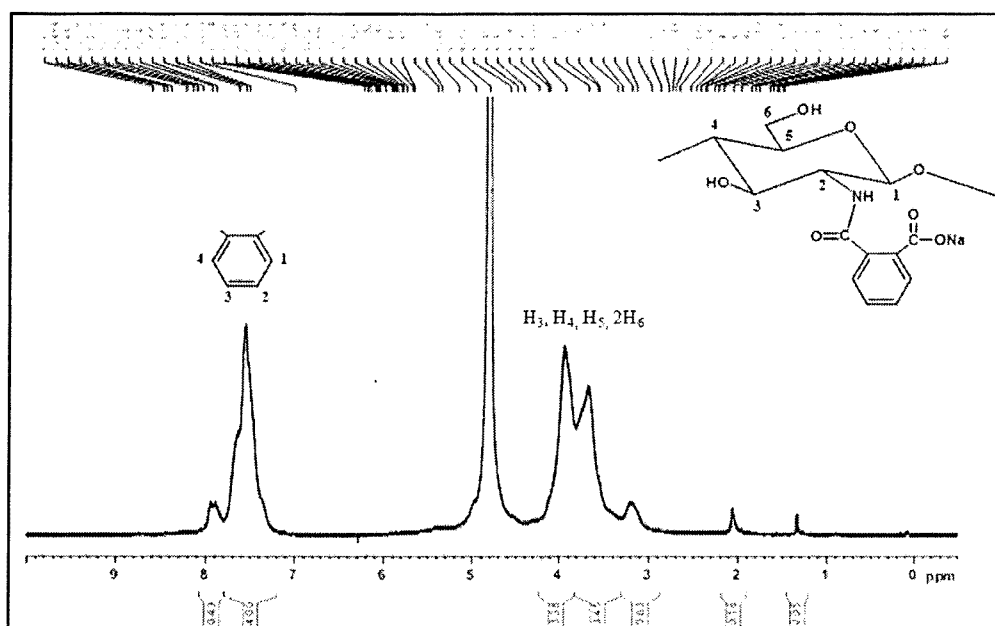


Figure 35 ¹H NMR spectra of N-PhCS prepared at 25°C under stirring for 4 h, CS:PA at 1:5 mole ratio and neutralization pH at pH 5.

Table 11 List of abbreviations.

Symbol	Definition
%	percent
>	more than
<	less than
°C	degree celsius
µg	microgram
µL	microliter
¹ H NMR	proton nuclear magnetic resonance
%v/v	percent volume by volume
%w/v	percent weight by volume
%w/w	percent weight by weight
A	absorbance
cm ⁻¹	wavenumbers
cm ²	square centimeter
CS	chitosan
CS-20	chitosan 20 kDa
CS-200	chitosan 200 kDa
N-PhCS	N-phthaloyl chitosan
DD	degree of deacetylation
D ₂ O	deuterium oxide
DS	degree of substitution
DSC	differential scanning calorimetry
e.g.	exemplī grātiā (Latin); for example
et al.	and others
etc.	et cetera (Latin); and other things/ and so forth
FTIR	fourier transform infrared spectroscopy
g	gram
HCl	Hydrochloric acid
h	hours

Symbol	Definition
<i>I</i>	integral (area under curve)
i.e.	id est (Latin); that is
kDa	kilodalton
kg	kilogram(s)
Lot. No.	lot number
mg	milligram
mL	milliliter
MW	molecular weight
N	normality
NaOH	Sodium hydroxide
nm	nanometer
PA	phthalic anhydride
<i>p</i>	p-value
pK_a	the negative logarithm of the dissociation constants
ppm	parts per million
PXRD	powder X-ray diffraction
SD	standard deviation
SGF	simulated gastric fluid
SIF	simulated intestinal fluid
TGA	thermogravimetric analysis
Tris	Tris (hydroxymethyl) aminomethane
W	weight
UV-VIS	ultraviolet -visible



