

Abstract

This work is focused on studying the influence of temperature, concentration, shearing time and storage time on the rheological properties, *i.e.*, the dynamic viscosity and shear stress as a function of shear rate of chitosan solubilized in the three selected solvents. Results showed that within the temperature range we studied (20°C to 50°C), a pronounced shear thinning was observed. It was also found that all the solutions obeyed the Arrhenius equation. When the effect of concentration was studied, it was observed that the shear thinning behavior was more remarkable at the highest concentration (10 g L⁻¹) for all solutions. The effect of shearing time on the dynamic viscosity of chitosan solutions did not show any significant differences at all shearing times studied in this study. Furthermore, more shear thinning behavior for chitosan in formic acid solutions, but less shear thinning for chitosan in acetic and propionic acids solutions were obtained with extending the period of storage to four months. A general decrease in viscosity with time for chitosan in formic acid solutions was observed while an increase in viscosity as a function of time was recorded for chitosan in acetic and propionic acids solutions at a constant shear rate.

Chitosan (CS) films dissolved in three selected solvents were also investigated in this work, by employing thermogravimetric analyses (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), atomic force microscopy (AFM), FTIR and mechanical properties. Results showed that the film made with acetic acid had the best thermal stability and lowest denaturation energy according to TGA and DSC measurements respectively. After a second run, the DSC curves showed small endothermic peaks at 186.20°C, 179.41°C and 171.43°C for chitosan films with formic, acetic and propionic acid solvents respectively. In addition, SEM measurements showed relatively smooth and homogenous surfaces for all films, which was confirmed by

AFM. The FTIR spectra of all chitosan films showed similar spectra with a little shift of some bands towards lower or higher wave numbers. Furthermore, chitosan film cast from acetic acid recorded the highest elongation at break, but the lowest tensile strength among the three types of film.

Abstrak

Penyelidikan ini tertumpu kepada kajian tentang pengaruh suhu, kepekatan, masa ricihan dan masa storan ke atas sifat-sifat reologi, iaitu kelikatan dinamik dan stres ricihan sebagai satu fungsi kadar ricih untuk kitosan yang dilarut dalam tiga pelarut pilihan. Keputusan menunjukkan bahawa dalam julat suhu yang dikaji (20°C ke 50°C), penipisan ricih yang ketara diperhatikan. Adalah juga ditemui bahawa kesemua larutan mematuhi persamaan Arrhenius. Bila kesan kepekatan dikaji, adalah diperhatikan bahawa kelakuan penipisan ricih adalah lebih ketara pada kepekatan yang tertinggi (10 g L^{-1}) untuk semua larutan. Kesan masa ricihan ke atas kelikatan dinamik larutan kitosan tidak menunjukkan sebarang perbezaan yang signifikan untuk kesemua masa ricihan yang dikaji dalam kajian ini. Tambahan, kelakuan penipisan ricih yang bertambah untuk kitosan dalam larutan asid formik tetapi penipisan ricih yang berkurang dalam larutan asid asetik dan propionik diperolehi dengan memanjangkan tempoh storan kepada empat bulan. Penurunan η_{sp}/c dalam kelikatan dengan masa untuk kitosan dalam larutan asid formik diperhatikan manakala suatu peningkatan dalam kelikatan sebagai satu fungsi masa telah direkodkan untuk kitosan dalam larutan asid asetik dan propionik pada kadar ricih yang tetap.

Filem kitosan yang diperolehi daripada tiga pelarut pilihan telah juga diselidik dalam kerja ini dengan menggunakan analisis termogravimetri (TGA), kalorimetri imbasan berbeza (DSC), imbasan mikroskop elektron (SEM), mikroskop daya atom (AFM), FTIR dan sifat-sifat mekanikal. Keputusan yang diperolehi menunjukkan bahawa filem yang melibatkan dengan pelarut asid asetik mempunyai kestabilan terma yang terbaik dan tenaga denaturasi yang terendah masing-masing mengikut pengukuran TGA dan DSC. Selepas larian kedua, keluk DSC menunjukkan puncak endoterma yang kecil pada 186.20°C , 179.41°C dan 171.43°C masing-masing untuk pelarut asid formik,

asetik dan propionik. Sebagai tambahan, pengukuran SEM menunjukkan permukaan yang licin dan homogen untuk semua filem secara bandingan dan di sahkan oleh AFM. Spektrum FTIR untuk semua filem kitosan menunjukkan spektrum yang serupa dengan anjakan yang kecil bagi beberapa jalur kepada nombor gelombang rendah atau tinggi. Seterusnya, filem kitosan yang disediakan daripada asid asetik mencatatkan kekuatan regangan yang terendah di antara ketiga jenis filem.

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LIST OF ABBREVIATIONS

AC	Acetic acid
AFM	Atomic force microscopy
cP	Centipoise
CS	Chitosan
D	Dalton
DD	Degree of deacetylation
DSC	Differential scanning calorimetry
E_a	Activation energy
FA	Formic acid
GPC	Gel permeation chromatography
PA	Propionic acid
Ra	Surface roughness
SD	Standard deviation
SEM	Scanning electron microscopy
TG	Thermogravimetric
TS	Tensile strength