ISETC2025

UNIVERSITI TEKNOLOGI MARA

ISETC2025

2 ND INTERNATIONAL SCIENCE, ENGINEERING AND TECHNOLOGY COLLOQUIUM



THE POWER OF SCIENTIFIC INNOVATION

INTELLIGENT SOLUTIONS FOR A SUSTAINABLE WORLD

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EDITORS

Dr. Emi Norzehan Mohamad Mahbob
Dr. Saiyidah Nafisah Hashim
Ms. Lili Widarti Zainuddin
Ms. Nurul Nazwa Mohammad
Ms. Rozaina Saleh

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PREFACE

Welcome to the 2nd International Science, Engineering and Technology Colloquium 2025 (ISETC 2025)!

It is with great pleasure that we present this year's colloquium, a distinguished gathering of scholars, researchers, and professionals dedicated to the advancement of applied sciences. Held virtually on 9th July 2025, ISETC 2025 serves as a platform to highlight cutting-edge research, foster meaningful networking, encourage collaborations, and explore innovative solutions to global challenges.

This year's theme, "The Power of Scientific Innovation: Intelligent Solutions for a Sustainable World" highlights our shared goal to solve global challenges through applied science. It celebrates innovation as a key to building a more sustainable and resilient future. We're proud to feature keynote speakers and presenters who bring fresh insights, bold ideas, and breakthroughs shaping tomorrow's world.

We would like to extend our heartfelt appreciation to the Faculty of Applied Sciences, Universiti Teknologi MARA, Perak Branch, Tapah Campus, for their outstanding support and assistance throughout the planning and execution of the event. Their dedication has been instrumental to the success of this colloquium. We would like to sincerely thank all participants and researchers for their valuable contributions. We also appreciate the reviewers for their careful evaluations and helpful feedback. Your hard work and support mean a lot to us.

As editors, we are proud to present this collection of proceedings. It is an honour to bring together the interesting and meaningful work of our speakers and authors. We hope the ideas shared will lead to new discoveries, encourage more research, and help solve global challenges through science and innovation.

Warm regards,

Dr. Emi Norzehan Mohamad Mahbob
Dr. Saiyidah Nafisah Hashim
Ms. Lili Widarti Zainuddin
Ms. Nurul Nazwa Mohammad
Ms. Rozaina Saleh

ISETC 2025 Proceedings Editors

SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF MODIFIED CHITOSAN VIA ACETYLATION AND BENZOYLATION

Aliya Husna Mohd Hafiz[1], Hussein Hanibah[2*]

[1]*School of Chemistry and Environment, Faculty of Applied Sciences, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*
[2]*Centre of Foundation Studies, Universiti Teknologi MARA, Cawangan Selangor, Kampus Dengkil, 43800 Dengkil, Selangor, Malaysia*

**Corresponding author: drhussein@uitm.edu.my*

Abstract: Chitosan is a derivative of chitin composed of glucosamine and N-acetyl glucosamine. It consists of functional groups including hydroxyl (OH) and amino (NH_2) groups that allow for chemical modifications. However, its application is limited by poor solubility in most organic solvents. This study aimed to synthesize chitosan derivatives that might improve the solubility in organic solvents. The acetylation and benzoylation are applied for the structure modification. In this study, the modified chitosan was synthesized by chemical modification and the resulting physicochemical changes were characterized. Chitosan powder was modified through acetylation by reacting with acetic acid and acetic anhydride at 50-60ºC for 6-8 hours. The mixture was neutralized with NaOH, precipitated with cold 75% ethanol, filtered, and dried. Characterization was performed using FTIR. Benzoylation is the second step, involving the addition of benzoyl chloride after the initial acetylation. Sodium bicarbonate is added to neutralize hydrochloric acid byproduct, and maintain the pH. Solubility tests using distilled water, ethanol, chloroform, diethyl ether and toluene were done for the synthesized product. From the FTIR spectrum of unmodified chitosan, the amide band (1200-1700 cm^{-1}) was not sharply seen. Post-acetylation, FTIR confirmed successful chemical modification and the introduction of acetyl groups onto the amino sites. New absorption bands appeared around 1500-1600 cm^{-1}, confirming the formation of secondary amide. Meanwhile, in benzoylated chitosan, a peak around 1600-1650 cm^{-1} associated with C-O stretching of ester groups and C=C stretching of aromatic ring. This confirmed substitution of benzoyl group in chitosan backbone. The study successfully demonstrated the synthesis of acetylated and benzoylated chitosan derivatives, confirmed by FTIR analysis and solubility test.

Keywords: *Chitosan, acetylation, benzoylation, diethyl ether, FTIR*

INTRODUCTION

Chitosan is a substance derived from chitin with its chemical name (1-2)-2-amino-2-deoxyl-β-D-dextran and is a copolymer composed of glucosamine and N-acetyl glucosamine (Pellis et al., 2022). Chitosan is made by partially or completely deacetylating chitin, which removes acetyl groups from the chemical chain to create amino groups (Ardean et al., 2021). Chitosan's functional groups include 2 hydroxyl groups at 3 and 6 carbon position, a 2-position amino group and a glycosidic linkage. The -NH_2 and -OH groups are chemically reactive, allowing for the synthesis of many chitosan derivatives through various chemical modifications (Chen et al., 2022).

Chitosan's applicability is severely limited due to its poor solubility in most organic solvents. The solution proposed in this study is chemical modification through acetylation and benzoylation, which introduces hydrophobic groups to the polymer backbone. Acetylation is done by reacting chitosan with acetic anhydride while benzoylation is done by further reacting it with benzoyl chloride. The objectives of this study are to synthesize chitosan derivatives that might have better solubility in organic solvents via acetylation and benzoylation and to characterize the physicochemical changes of chitosan after the modification. Spectroscopic analysis by using FTIR was done to confirm the additional functional groups while solubility test was done to observe the solubility behaviours of unmodified and modified chitosan in various organic solvents. From both analysis, it can be confirmed that amide and benzoyl group had been introduced in the chitosan backbone and increased the organic solubility.

MATERIALS AND METHODS

The materials used in this study are chitosan powder, acetic acid (CH_3COOH) from ASTRAL, acetic anhydride $(CH_3CO)_2O$ from EMSURE, ethanol from Chemiz, sodium hydroxide (NaOH) from MERCK, sodium bicarbonate ($NaHCO_3$) and benzoyl chloride (C_7H_5ClO) from R&M.

The acetylation and benzoylation steps have been carry out for the structure modification of chitosan. For the first part of the synthesis process acetylation is done by reacting chitosan powder with CH_3COOH and $(CH_3CO)_2O$ which will modify 2-position amino group to amide (Pillai et al., 2009). The first step is reacting chitosan with CH_3COOH and $(CH_3CO)_2O$. CH_3COOH is used to dissolve the chitosan in water as chitosan is not readily soluble in water (Santoso et al., 2020). Then, $(CH_3CO)_2O$ is added and the mixture is heated to 50ºC-60ºC for about 6-8 hours for acetylation purpose. After that, the mixture was neutralized with NaOH solution to deactivate remaining CH_3COOH or excess $(CH_3CO)_2O$ which can hydrolyze to CH_3COOH (Sánchez et al., 2021). The neutralization was confirmed by checking the pH of the mixture with pH paper. Then, cold 75% ethanol is added to the reaction mixture to facilitate the modified chitosan-amide product. Finally, to obtain the solid, it was filtered with vacuum pump and dried. A total of five chitosan-amide samples were prepared by varying the concentrations of CH_3COOH and $(CH_3CO)_2O$ during the reaction process as shown in Table 1. This is done to investigate the effect of reagent concentration on the synthesis.

Table 1. Variation of concentration of reagents in the mixture

Sample No	Acetic Acid (%)	Acetic Anhydride (%)
1	1.00	10
2	2.00	20
3	2.00	12
4	1.25	10
5	2.50	18

The second step is benzoylation on the chitosan-amide mixture. The first step was repeated until the chitosan-amide mixture is synthesized completely. After it has been precipitated with cold ethanol, benzoyl chloride is added to introduce the benzene group to the chitosan's backbone. Sodium bicarbonate ($NaHCO_3$) is added during benzoylation to neutralize the hydrochloric acid (HCl) by-product, maintain a suitable pH, and enhance reaction efficiency and product stability. After cold ethanol is added to the mixture, it will be filtered with vacuum pump and dried in the oven at 50ºC-60ºC overnight to obtain the final product.

Characterization process is done by FTIR to confirm the formation of new functional groups (Claverie et al., 2023). Another analysis was done which is the solubility test. In this procedure, around 0.5g of unmodified and modified chitosan were weighed and transferred into beakers. First, distilled water was added to each beaker. Then the samples were left to stir on the magnetic stirrer for 1 hour to observe the solubility behaviour. The procedure was then repeated using other solvents with different polarity index as shown in Table 2.

Table 2. Polarity index of solvents used in solubility test

Solvent	**Polarity index**
Distilled Water	10.2
Ethanol	5.2
Chloroform	4.1
Diethyl Ether	2.8
Toluene	2.4

RESULTS AND DISCUSSION

Figure 1 shows the FTIR spectra of unmodified chitosan and modified chitosan while Figure 2 shows the structure of the final product. In the structure, the additional of new ester bond and benzene group can be seen. The benzene group from benzoyl chloride has more affinity toward hydroxyl groups of alcohol than amide group in the previous compound leading to its attachment to the oxygen atom. FTIR spectra of unmodified, acetylated, and benzoylated samples reveal distinct structural differences due to chemical modifications. Both unmodified and modified chitosan samples show absorption band around 1020 cm^{-1} represent C-N stretching of amine group which is one of the important groups in chitosan backbone. The spectrum of unmodified chitosan showed a broad absorption band around 3200cm^{-1} - 3400 cm^{-1}, which is attributed to O–H and N–H stretching vibrations, indicating the presence of -OH and -NH_2 groups on the chitosan backbone. The acetylated spectrum exhibits strong absorption bands at 1406 cm^{-1} and 1541 cm^{-1}, corresponding to C=O stretching and N–H bending of a secondary amide group, confirming successful acetylation. In contrast, the benzoylated sample shows stronger absorption peak around 1027 cm^{-1} indicating the overlapping C-N stretching of amine and new ester bond formed. A significant peak at 1632 cm^{-1} indicate the overlapping of ester C=O stretching and aromatic ring C=C stretching. Thus, upon modification, new absorption bands can be noticeably seen around 1350 cm^{-1} to 1650 cm^{-1} that indicate C-N and C=O stretching of secondary amide and C=C of aromatic ring which were not present in unmodified chitosan. These spectral differences confirm the structural changes introduced through acetylation and benzoylation. Acetylated compounds containing secondary amide functionalities and benzoylated compounds additionally contain benzene rings and ester linkages.

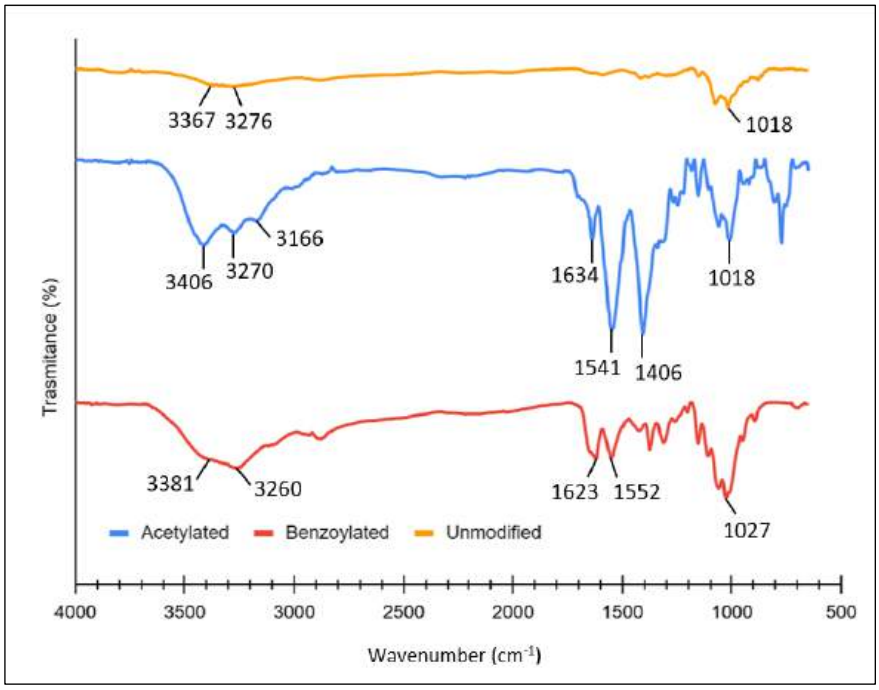


Figure 1. FTIR spectra of unmodified and modified chitosan

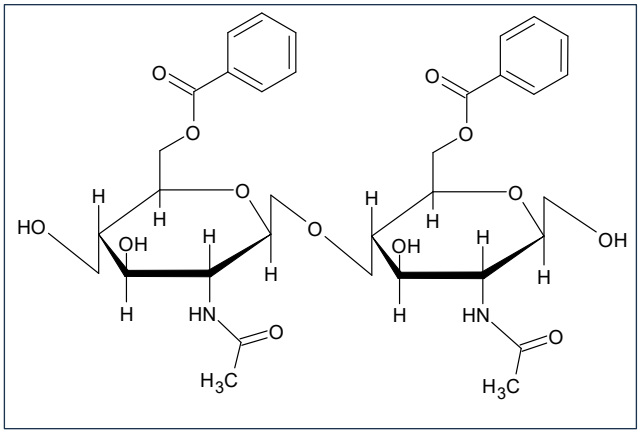


Figure 2. Structure of final product, benzoylated chitosan

The solubility of both unmodified and modified chitosan samples evaluated using various solvents which are distilled water, ethanol, chloroform, diethyl ether and toluene. Unmodified chitosan showed slightly solubility in distilled water and ethanol while it is completely insoluble in chloroform, diethyl ether and toluene. In contrast, acetylated chitosan showed partial solubility in ethanol, diethyl ether and toluene. This suggests that the introduction of acetyl groups by reacting with acetic anhydride slightly increased the hydrophobicity. Benzoylated chitosan showed the most substantial change in solubility behaviour. It is soluble in the majority of organic solvents tested. The added bulky benzoyl groups from benzoyl chloride disturbed existing hydrogen bonds while also introducing aromatic moieties, improving compatibility with non-polar and moderately polar organic solvents.

CONCLUSIONS

The study successfully demonstrated the synthesis of chitosan derivatives via acetylation and benzoylation using acetic anhydride and benzoyl chloride to enhance its solubility in most organic solvents. The FTIR spectroscopic analysis confirmed the introduction of acetyl and benzoyl groups, indicated by characteristic new peaks and shifts in the spectra of the modified chitosan derivatives. The solubility tests clearly showed a significant improvement in the organic solubility of the final product, benzoylated chitosan compared to unmodified chitosan. This directly supports the hypothesis that introducing hydrophobic groups disrupts the extensive hydrogen bonding and crystalline structure of chitosan, thereby increasing its compatibility with organic solvents. These findings underscore the potential of chemically modified chitosan for a broader range of industrial and biomedical applications where organic solvent compatibility is crucial.

Recommendations suggested for this study is to apply other characterization techniques such as ^{1}H and C-12 NMR and mass spectroscopic analysis to support the identification of the new functional groups. Besides, it is also recommended to study different catalysts or reaction media that can be used to improve reaction efficiency and better solubility profiles. Furthermore, exploring other hydrophobic substituents for chitosan modification may result in improved organic solubility and the ability to modify specific characteristics for a variety of applications.

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