# SYNTHESIS AND CHARACTERIZATION OF WASTE NEWSPAPER CELLULOSE BASED FLOCCULANT FOR WATER TREATMENT

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A thesis submitted in fulfillment of the
requirements for the award of the degree of
Master of Engineering (Chemical)

Faculty of Chemical and Energy Engineering
Universiti Teknologi Malaysia

FEBRUARY 2016

To my beloved parents, family, and friends

## ACKNOWLEDGEMENTS

Alhamdulillah, all praises to the Almighty Allah S.W.T, finally my project thesis had successfully developed with my best efforts. I would never have been able to finish my project without the guidance of the following people. So here, I wish to acknowledge the excellent support and valued contributions from a number of persons that I have received in the completion of this thesis. I would like to express my deepest gratitude to my supervisor, Associate Professor Dr. Hanapi Mat, for his excellent guidance, patience, and providing me with an excellent atmosphere for doing research. His enthusiasm and encouragement has been extremely helpful. Grateful acknowledge to the Ministry of Higher Education and Universiti Malaysia Perlis (UNIMAP) for the financial support as well as Universiti Teknologi Malaysia (UTM) for providing amenities for the project accomplishment. Special thanks to my lab mates (AMPEN), for their valuable contributions, kindness, and moral support during my study. Thanks for the friendship and memories. My research would not have been possible without their helps. I would also like to express thousand appreciations to my family members for their endless love, prayers and encouragement. To those who indirectly contributed to this research, your kindness means a lot to me. Thank you very much.

## ABSTRACT

The quaternized cellulose derivatives (QCs) were synthesized through etherification reaction of cellulose extracted from waste newspaper (WNP) for water treatment. The cellulose was extracted from WNP by chemical treatment. The characterization results by using Fourier transform infrared (FTIR) spectrophotometer, x-ray diffractometer, thermogravimetric analysis, and scanning electron microscope show that the WNP properties changed after chemical treatment and the extracted product was confirmed cellulose. The QCs were homogeneously synthesized by reacting extracted cellulose with 3-chloro-2-hydroxypropyl trimethylammonium chloride (CHPTAC) using sodium hydroxide/urea aqueous solution as a reaction medium. The structure and properties of QCs were characterized using FTIR spectrophotometer, carbon nuclear magnetic resonance, hydrogen nuclear magnetic resonance, gel permeation chromatography and elemental analysis. The results indicated that the QCs having various nitrogen content could be obtained by changing the molar ratio of cellulose unit to CHPTAC. The flocculation performance of the QCs was evaluated by using a synthetic kaolin suspension carried out using the standard jar test method at different coagulant/flocculant dosages, kaolin concentrations, pH values, and settling times. It was found that the QC-15 exhibited a highly effective flocculation capability as compared to other synthesized QCs for over a wide pH values. The rate constant (k) of the coagulation/flocculation kinetics increased with cationic content. The sludge specific resistance decreased with increasing coagulant/flocculant dosage. The coagulation/flocculation of the surface water shows that the QC-15 was effective for the removal of turbidity and total suspended solid as compared to biochemical oxygen demand, chemical oxygen demand and pH. These results demonstrated that the WNP cellulose can be used for the development of effective and eco-friendly coagulant/flocculants which have good potential applications in water treatment.

# ABSTRAK

Terbitan selulosa berkuaternari (QCs) telah disintesis melalui tindak balas pengeteran selulosa daripada sisa akhbar (WNP) untuk rawatan air. Selulosa telah diekstrak daripada WNP dengan menjalankan rawatan kimia. Keputusan pencirian dengan menggunakan spektrometer inframerah transformasi Fourier (FTIR), difraktometer sinar-x, analisis termogravimetrik dan mikroskop imbasan elektron menunjukkan bahawa sifat-sifat WNP berubah selepas rawatan kimia dan produk yang telah diekstrak disahkan sebagai selulosa. Komposisi kimia WNP telah ditentukan sebelum dan selepas rawatan. QCs telah disintesis secara homogen oleh tindak balas ekstrak selulosa dengan 3-kloro-2-hidroksipropil trimetil ammonium klorida (CHPTAC) dengan menggunakan larutan natrium hidroksida/urea sebagai medium tindak balas. Struktur dan sifat-sifat QCs telah dicirikan menggunakan FTIR, karbon resonans magnetik nukleus, hidrogen resonans magnetik nukleus, kromatografi penelapan gel dan analisis unsur. Keputusan menunjukkan bahawa QCs mempunyai pelbagai kandungan nitrogen yang boleh diperoleh dengan menukar nisbah unit selulosa kepada CHPTAC. Prestasi pengelompokan daripada QCs dinilai dengan menggunakan ampaian kaolin sintetik yang dijalankan dengan kaedah ujian balang pada dos pengental/pengelompok berbeza, kepekatan kaolin, nilai pH, dan masa pemendapan. Keputusan menunjukkan bahawa QC-15 mempamerkan keupayaan pengelompokan yang amat berkesan dalam julat pH yang luas berbanding QCs lain. Pemalar kadar (k) kinetik pengentalan/pengelompokan meningkat dengan peningkatan kandungan kationik. Rintangan spesifik enapcemar menurun dengan peningkatan dos pengental/pengelompok. Pengentalan/pengelompokan permukaan air menunjukkan bahawa QC-15 berkesan untuk penyingkiran kekeruhan dan jumlah pepejal terampai berbanding dengan permintaan oksigen biokimia, permintaan oksigen kimia, dan pH. Keputusan ini menunjukkan bahawa selulosa WNP boleh digunakan untuk menghasilkan pengental/pengelompok berkesan dan mesra alam yang berpotensi baik dalam rawatan air.

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| | | |
|---|---|---|
| A | - | Filter area ($m^2$) |
| b | - | Slope of filtrate discharge curve (t/V versus V) ($s/m^6$) |
| $C_g$ | - | Mass of dried cake per unit volume of filtrate (kg/m3) |
| $C_k$ | - | Kaolin concentration |
| ºC | - | Degree Celsius |
| $D_f$ | - | Flocculant dosage |
| $E_f$ | - | Flocculation efficiency |
| g/mL | - | Gram per milliliter |
| I | - | Intensity (counts) |
| k | - | Rate constant |
| $M_w$ | - | Molecular weight |
| mg/g | - | Milligram per gram |
| mg/L | - | Milligram per Liter |
| $N_o$ | - | Initial number concentration of kaolin particles |
| $N_t$ | - | Number concentration of kaolin particles at time t |
| P | - | Filtration pressure ($N/m^2$) |
| T | - | Temperature |
| $T_s$ | - | Sedimentation time |
| $T_i$ | - | Initial turbidity |
| $T_f$ | - | Final turbidity |
| V | - | Volume of filtrate ($m^3$) |
| wt. % | - | Weight percent |
| $X_c$ | - | Crystallinity index |
| % | - | Percentage |
| % w/v | - | Weight per volume percent |
| µ | - | Viscosity of the filtrate (N s/m2) |
| υ | - | Wavenumber ($cm^{-1}$ |

## LIST OF ABBREVATIONS

| | | |
|---|---|---|
| AGU | - | Anhydroglucose unit |
| ATR | - | Attenuated Total Relectance |
| AAS | - | Atomic Absorption spectrophotometer |
| APHA | - | American Public Health Association |
| BHC | - | Benzene hexachloride |
| BMIMCl | - | 1-Butyl-3-methylimidazolium Chloride |
| $BOD_5$ | - | Biochemical oxygen demand |
| COD | - | Chemical oxygen demand |
| CHPTAC | - | 3-chloro-2-hydroxypropyl trimethylammonium chloride |
| CMCNa | - | Carboxymethylcellulose |
| $^{13}$CNMR | - | Carbon-13 Nuclear Magnetic Resonance |
| $CS_2$ | - | Carbon disulfide |
| CWSs | - | Community water systems |
| $C_6H_5CH_3$ | - | Toluene |
| $CH_3COOH$ | - | Acetic acid |
| $CH_3COCH_3$ | - | Acetone |
| DBP | - | Disinfection by product |
| DDT | - | Dichloro-diphenyl-trichloroethane |
| DOC | - | Dissolved organic carbon |
| DOM | - | Dissolved organic matter |
| DP | - | Degree of polymerization |
| DS | - | Degree of substitution |
| DWDs | - | Drinking water distribution systems |
| DTG | - | Derivative thermogravimetry |
| EA | - | Elemental analysis |
| EPTAC | - | 2,3-epoxypropyl trimethyl ammonium chloride |

| | | |
|---|---|---|
| FTIR | - | Fourier transform infrared |
| GTAC | - | Glycidyltrimethyl ammonium chloride |
| GPC | - | Gel permeation chromatography |
| $H_2O_2$ | - | Hydrogen peroxide |
| $H_2SO_4$ | - | Sulphuric acid |
| HDPE | - | High-density polyethylene |
| $^1$HNMR | - | Hydrogen Nuclear Magnetic Resonance |
| HPC | - | Heterotrophic plate count |
| ILs | - | Ionic liquids |
| KBr | - | Potassium bromide |
| MCL | - | Maximum contaminant level |
| $NaClO_2$ | - | Sodium chlorite |
| NaOH | - | Sodium hydroxide |
| NMMO | - | N-methyl morpholine Oxide |
| NOM | - | Natural organic matter |
| WNP | - | Waste newspaper |
| NTU | - | Nephelometric Turbidity Unit |
| $UV_{254}$ | - | Ultraviolet absorbance at 254 |
| PAHs | - | Polycyclic aromatic hydrocarbons |
| PBDE | - | Polybrominated diphenyl ether |
| PCBs | - | Polychlorinated biphenyls |
| RBF | - | Round bottom flask |
| SEM | - | Scanning electron microscopy |
| TAPPI | - | Technical Association of Pulp and Paper Industry |
| 2,3,7,8-TCDD | - | 2,3,7,8- Tetrachlorodibenzo-p-dioxin |
| TGA | - | Thermogravimetric analysis |
| THMs | - | Trihalomethanes |
| TSS | - | Total suspended solid |
| XRD | - | X-ray diffraction |
| QC | - | Quaternized cellulose |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Drinking water is one of the important needs of the community. It can be found from two main sources: ground water, which is pumped from wells; and surface water like lakes, rivers and reservoirs (Kapoor and Viraraghavan, 1997; Das and Acharya, 2003). In Malaysia, 98 % of the nation's potable water is supplied by the river and mostly treated by using conventional water treatment process (Santhi *et al*., 2012). It is necessary to ensure the society can access clean water at an affordable price which meets the national standard qualifications. However, water pollution has becoming a serious problem resulting from urbanization and industrialization that threaten human being, plants and animals. Human activities like municipal, industrial and agricultural integrated with the climate change could significantly affect the water quality. Failure access of the clean water may risk the human body, health, and cause several diseases such as typhoid, cholera and diarrhea.

Drinking water consists of several pollutants include solid and dissolved particles such as inorganic micropollutants, organic micropollutants, pathogens, and dissolved organic matter. The existing these kinds of pollutants will chemically, physically and biologically affect the quality of water as well as reduce the water quality level. Furthermore, in drinking water treatment, there are some issues to be highlighted due to the formation of disinfectant by products (DBPs) as a consequence of chlorinated water as an impact of the climate change. Thus, regarding to these matters, it is important to implement an effective technology for the drinking water treatment with the best performance efficiency and economic value.

In recent years, these problems have raised concern and a lot of technologies have been developed in water treatment process. The technologies included physical, chemical and biological treatment methods. Moreover, the technologies used must comply with the requirement which can be operated efficiently, low cost, simple and environmentally friendly. Several treatment processes like conventional and advanced technology have been adopted in many years in order to remove pollutants from water. It included adsorption, flocculation, filtration, chlorination, ozonation, and also Fenton oxidation (Simeunovic *et al*., 2013). Advanced technologies like membrane filtration, ultraviolet light and ozonation usually applied in the water treatment to remove pathogens and control the disinfection byproducts (DBPs) in order to achieve requirement standard quality. Nevertheless, advanced technologies have raised some issues due to the high price and high biofouling tendency (Zhou and Smith, 2002). Therefore, more research is needed to improve the current technology in order to obtain better performance.

## 1.2 Problem Statements

Coagulation/flocculation is one of the primary steps applied to water treatment owing to low cost, easy to handle, simple and effective techniques in solid liquid separation (Song *et al*., 2010; Khiari *et al*., 2010). It has been used in a wide range of application which includes wastewater treatment and industrial processes. Coagulation is a process of neutralization between the coagulant agent and colloidal particles, while flocculation refers to the process of solid liquid separation by aggregation of colloidal particles and formation of large stable flocs (Tassinari *et al.*, 2013).

In coagulation/flocculation process, coagulant/flocculant is significantly used and plays an important role to remove suspended solid by accelerating the colloidal particles in the aggregation process. Generally, coagulant/flocculant can be categorized into two which are inorganic and polymeric. As reported by the previous work, inorganic coagulant/flocculant has good performance, which leads to production of large and strong aggregates in flocculation. However, this material raised some issues regarding their toxicity, non-biodegradable and generated numerous of sludge, which are difficult to dehydrate (Bratby, 2007). The use of the inorganic coagulant such as aluminium sulfate in this process nowadays have been doubtful due to their high potential contribution to Alzheimer's disease (Li *et al.*, 2013). In comparison, polymeric coagulant/flocculant has received great attention among the researchers because these coagulant/flocculants only required a small dosage and inertness towards pH change.

Apart from that, polymeric coagulant/flocculant can be further separated into natural and synthetic. With the increasing demand for environmentally and health technologies, natural coagulant/flocculant has been researched widely in order to replace inorganic and synthetic coagulant/flocculant. Natural coagulant/flocculant are safe for human health and good in biodegradability (Szyguła *et al.*, 2009). Several natural polysaccharides such as starch, chitin, guar gum and cellulose have been extensively explored and reported as efficient coagulant/flocculant for water and wastewater treatment.

Cellulose is well known as one of the most abundantly available renewable resource in the world. It has become an attractive material due to its ability to accept new functional group in order to broaden the application in many industrial fields. In this regard, there is an increasing number of studies focusing on modification and utilization of water soluble cellulose derivatives as coagulant/flocculant. For example, cellulose grafted with acrylamide (Das *et al*., 2013; Song *et al*., 2009; Song *et al*., 2011), cationized with ammonium groups (Zaman *et al*., 2012), and anionized by periodate oxidation and sulfonation (Liimatainen *et al*., 2012). Various techniques have been employed in modification of cellulose, which included esterification, etherification, grafting, deoxyhalogenation and oxidation. Cellulose can be modified to a coagulant/flocculant which increases its functionality and improves its performance efficiency of the water treatment.

Cellulose mainly obtained from wood and non woody plants. Recently more research has been carried out on utilization of cellulose from non woody plant such as oil palm biomass (OPB), coconut husks and corn stover. One of the possible sources of cellulose is waste newspaper (WNP). WNP is a particularly attractive source for cellulose since it is readily available. Malaysia itself generated 57 000 tons each month of the paper waste which include newspaper (Rahman *et al.*, 2009). WNP can be categorized as the complex materials and consist mainly of cellulose (Wang and Li, 2009). Reducing, recycling and reusing are the several common methods to reduce landfill waste. However, this abundant waste will still end up in landfills due to the constant large quantity supply. Among developing Asian, 70 to 90% of the municipal solid wastes are being disposed in landfill (Ismail and Manaf, 2013). Landfill wastes cause a big problem in terms of the economical and environmental impact. In fact, several acres of land have been utilized to decompose large amount of trash where this land could actually be used for setting up new residential area or industries. Therefore, WNP has been used as a source of cellulose to develop a new useful product as an alternative way to preserve the environment.

Currently, cellulose from WNP has been used in many application such as bioethanol production (Wang *et al*., 2013), modified as an adsorbent for heavy metal removal (Adhikari *et al*., 2008), dye removal (Zhang *et al*., 2013) and also derivation of cellulose derivatives (Filho *et al.,* 2008; Unlü 2013). So far there is no report regarding to the derivation of quaternized cellulose from WNP. This study was aimed to synthesize cellulose based coagulant/flocculant derived from WNP. The extracted cellulose was modified into quaternized cellulose coagulant/flocculant. The effectiveness of developed coagulant/flocculant was evaluated on the kaolin suspension and surface water. The effects of coagulant/flocculant dosage, pH, kaolin concentration and sedimentation time were studied by measuring the residual turbidity of the settled suspension. The objectives of the present study are to develop a new water soluble coagulant/flocculant with high flocculation capacity and dewatering capability. The flocculation mechanism was also discussed based on the flocculation kinetics.

## 1.3 Objectives of the Study

i. To extract and characterize cellulose from waste newspaper (WNP).

ii. To synthesize and characterize quaternized cellulose based coagulant/flocculant derived from waste newspaper (WNP).

iii. To evaluate the performance of waste newspaper (WNP) based coagulant/flocculant for water treatment.

## 1.4 Scopes of the Study

Cellulose was extracted using alkali and bleaching treatment. Sodium Chlorite was used as a bleaching agent. The characteristics of cellulose was characterized using Fourier Transform Infrared (FTIR) spectroscopy to analyze the functional group, X-Ray Diffraction (XRD) for crystallinity property, Thermogravimetric Analysis (TGA) for thermal stability study and Scanning Electron Microscope (SEM) to investigate the surface morphology. The chemical composition of the WNP before and after treatment has also been investigated.

Quaternization of the extracted cellulose was carried out in NaOH/urea aqueous solution as a reaction medium. 3-chloro-2-hydroxypropyltrimethylammonium chloride (CHPTAC) has been used as a cationic moiety which reacted with cellulose under alkaline conditions. Six quaternized cellulose (QCs) derivatives were prepared in order to study the effect of molar ratio of cellulose to CHPTAC. The structure and properties of the QCs were studied using Fourier Transform Infrared (FTIR) spectroscopy in order to study the functional group of the QCs, Nuclear Magnetic Resonance (NMR) spectrophotometer to verify carbon-hydrogen structure of the developed coagulant/flocculant, Gel Permeation Chromatography (GPC) to determine the molecular weight and the Elemental Analyzer (EA) to determine the nitrogen content of the QC.

The performance of WNP based coagulant/flocculant was evaluated using a standard jar test. Several parameters were studied such as effect of molar ratio of cellulose to CHPTAC, coagulant/flocculant dosage, pH, kaolin concentration, and settling time. In jar test condition, the mixing speed was fixed at 250 rpm (3 minutes), 50 rpm for slow mixing (30 minutes) and was allowed to settle down within 20 minutes. The effectiveness of the coagulant/flocculant was tested on kaolin suspension. A coagulant/flocculant with the best performance was selected to further study on surface water. Performance indicator of the treated kaolin suspension is turbidity while for the treated surface water is turbidity, total suspended solid (TSS), chemical oxygen demand (COD) and biochemical oxygen demand (BOD). Dewatering capability of kaolin suspension in the presence and absence of coagulant/flocculant has been studied. The flocculation kinetics was also investigated in order to discuss in detail the mechanism of the flocculation.

## 1.5 Thesis Outline

Basically, this thesis consists 5 chapters. Chapter 1 presents a brief description of research background, problem statement, and objectives and scopes of the study. Chapter 2 presents the critical reviews on drinking water treatment, coagulation/flocculation process in drinking water treatment, and cellulose derivatives from WNP as natural coagulant/flocculant. All the materials and methods used throughout the study were described details in Chapter 3. In this chapter, the experimental procedure for the cellulose extraction, synthesis of quaternized cellulose, and the application of the developed coagulant/flocculant are described. The results obtained were discussed in Chapter 4. Finally, Chapter 5 summarizes overall research findings which were discussed in Chapter 4. Several recommendations for future research were also included in this chapter.

At the optimal dosage, the specific resistance decreased from 2.08E+12 m/kg to 1.19E+12 m/kg. The coagulation/flocculation of Skudai River water shows that high removal performance towards turbidity and total suspended solid (TSS) as compared to $BOD_5$, COD and pH was observed. These experimental results show that the quaternized celluloses such as QC-15 were potential flocculants for water treatment.

## 5.2 Recommendations for Future Work

The extraction of cellulose from WNP was successfully carried out by chemical treatment, which can be further improved by employing other chemicals or techniques in order to obtain higher cellulose yield by efficiently removing lignin and other impurities. The extraction of cellulose from various types of waste papers such as office paper, magazine and cardboard is also recommended for future study.

As demonstrated from the present study, cellulose was successfully quaternized in the NaOH/urea aqueous solution. It is suggested to use ionic liquids as a medium for the reaction since they are eco-friendly solvents. Other cellulose derivatives such as quaternized cellulose grafted polyacrylamide and anionic cellulose should be synthesized and tested for coagulation/flocculation properties used in various applications.

The synthesized coagulant/flocculants was only evaluated for the kaolin suspension and surface water. The research can be expanded to investigate the performance of the synthesized coagulant/flocculants to different sources of wastewaters such as dye, sewage water, paper industry wastewater and oily wastewater.

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