



THE REPUBLIC OF INDONESIA DEFENSE UNIVERSITY

**COMBINATION OF SONICATION AND DEEP EUTECTIC
SOLVENT IN THE BLEACHING FOR α -CELLULOSE
ISOLATION FROM FLAX FIBER**



FANIRAHMAWATI SID. 320200303008

**Undergraduate Thesis Written to Fulfill Part of the Requirements in
Obtaining the Bachelor Degree**

**FACULTY OF MILITARY MATHEMATICS AND NATURAL SCIENCES
DEPARTMENT OF CHEMISTRY
BOGOR 2024**

APPROVAL SHEET OF UNDERGRADUATE THESIS

Name : Fani Rahmawati
SID : 320200303008
Department : Chemistry
Faculty : Military Mathematics and Natural Sciences
Title of Undergraduate Thesis : Combination of Sonication and Deep Eutectic Solvent in the Bleaching for α -Cellulose Isolation from Flax Fiber

Supervisor I,



Dr. Rahmat Basuki, M.Sc.
NIP 199005132022031001
Date: 26 JUL 2024

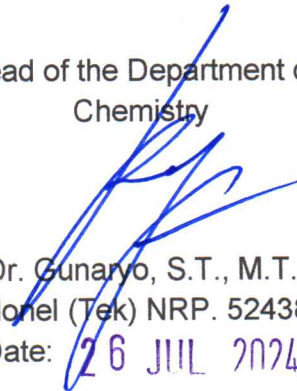
Supervisor II,



Elva Stiawan, S.Pd., M.Si.
NIP 199008242022031001
Date: 26 JUL 2024

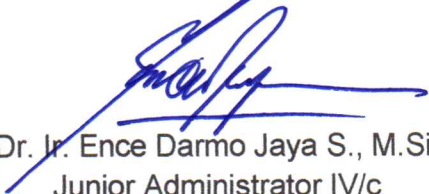
Acknowledge,

Head of the Department of
Chemistry



Dr. Gunaryo, S.T., M.T.
Colonel (Tek) NRP. 524384
Date: 26 JUL 2024

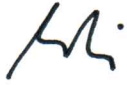
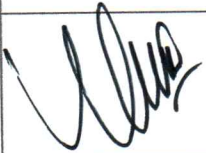
Dean of Faculty of Military
Mathematics and Natural Sciences



Dr. Ir. Ence Darmo Jaya S., M.Si.
Junior Administrator IV/c
Date: 26 JUL 2024

VALIDATION SHEET OF UNDERGRADUATE THESIS

Name : Fani Rahmawati
SID : 320200303008
Department : Chemistry
Faculty : Military Mathematics and Natural Sciences
Title of Undergraduate Thesis : Combination of Sonication and Deep Eutectic Solvent in the Bleaching for α -Cellulose Isolation from Flax Fiber

No	Name	Signature	Date
1	Supervisor I: Dr. Rahmat Basuki, M.Sc. NIP 199005132022031001		26 JUL 2024
2	Supervisor II: Elva Stiawan, S.Pd., M.Si. NIP 199008242022031001		26 JUL 2024
3	Examiner I: Anggi Khairina Hanum Hasibuan, S.Si., M.Si. NIP 199111032022032002		26 JUL 2024
4	Examiner II: Dr. Ir. Mirad Fahri, M.Sc. NIDN 4702126401		26 JUL 2024

ORIGINALITY STATEMENT

I hereby declare that this undergraduate thesis contains original work and has not been submitted for a degree at any university or at any level. To the best of my knowledge, it does not contain any phrases, sentences, paragraphs, or chapters of work that have been written or published.

If plagiarism in this thesis is proven in the future, I am willing to accept appropriate sanctions according to relevant laws and regulations.

Bogor, July 2024



Fani Rahmawati

**STATEMENT OF APPROVAL FOR PUBLICATION OF SCIENTIFIC
WORK FOR ACADEMIC PURPOSES**

I, the undersigned, as an academic member of the Defense University of the Republic of Indonesia:

Name : Fani Rahmawati
NIM : 320200303008
Departement : Chemistry
Faculty : Military Mathematics and Natural Sciences
Type of Work : Undergraduate Thesis

For the development of science, I agree to grant the Republic Indonesia Defense University a **non-exclusive, royalty-free right** to my scientific work entitled:

Combination of Sonication and Deep Eutectic Solvent in the Bleaching for α -Cellulose Isolation from Flax Fiber

along with existing devices (if needed). With this **Non-Exclusive Royalty-Free Right**, the Republic Indonesia Defense University has the right to store, transfer media/format, manage in the form of databases, maintain, and publish my final project as long as I keep my name as the author/creator and as the copyright owner.

Thus I make this statement truthfully.

Made in : Bogor
On date : 24th July 2024
Who declares



Fani Rahmawati
NIM. 320200303008

PREFACE

The formulation of the under graduated thesis, titled “Combination of Sonication and Deep Eutectic Solvent in Bleaching for α -Cellulose Isolation from Flax Fiber”, owes its realization to the benevolence and guidance of Allah SWT, consistently facilitating solutions to the challenges encountered. The development of this thesis represents the fulfillment of academic responsibilities as a student within the Military Chemistry study program at the Faculty of Mathematics and Natural Sciences of the Military Defense University, aimed at achieving a bachelor's degree.

Certainly the successful completion of this under graduated thesis is due to the contribution and support of various parties directly and indirectly. Therefore, on this occasion the cadet would like to express the heartfelt gratitude to:

1. Dr. Rahmat Basuki, M.Sc, served as my supervisor, consistently offering valuable insights and assisting in overcoming numerous challenges encountered in the completion of this thesis
2. Elva Stiawan, M.Si as my second supervisor who provided advice in the preparation of the thesis
3. Lieutenant General Jonni Mahroza, S.I.P., M.A., M.Sc.,CIQnR, Ph.D as Rector of Indonesia Defense University
4. Dr. Ir. Ence Darmo Jaya Supena, M.Si as the Dean of the Military FMIPA
5. Colonel (Eng) Dr. Gunaryo, S.T., M.T. as the head of the study program
6. My parents, for their selfless love, endless encouragement, and steadfast belief in my abilities
7. My best friend, whose friendship has been a constant source of strength and inspiration.

May the Almighty God reward the kindness of all those who have provided assistance.

I acknowledge that this under graduated thesis is not without imperfections. I welcome constructive criticism and suggestions to enhance its overall quality and completeness.

Bogor, 2024

Fani Rahmawati



ABSTRAK

KOMBINASI METODE SONIKASI DAN DEEP EUTECTIC SOLVENT DALAM PROSES BLEACHING UNTUK ISOLASI α - SELULOSA DARI SERAT RAMI

Penelitian mengenai bahan baku alternatif untuk isolasi selulosa telah banyak dilakukan, beberapa bahan baku alternatif yang dipertimbangkan adalah serat rami dan serat daun nanas. Proses isolasi selulosa dari kedua tanaman tersebut tergolong masih kurang efektif karena proses isolasi masih dilakukan dengan menggunakan agen *bleaching* hidrogen peroksida yang memiliki dampak cukup membahayakan bagi kesehatan lingkungan. *Deep eutectic solvent* (DES) merupakan salah satu agen *bleaching* alternatif yang dapat dipertimbangkan karena kemampuannya dalam mengisolasi senyawa selulosa serta dampaknya yang tidak berbahaya bagi kesehatan lingkungan. Kombinasi penggunaan DES dan metode sonikasi dalam proses isolasi selulosa, penelitian ini bertujuan untuk mengetahui pengaruh dari metode tersebut terhadap efektivitas proses *bleaching* dari serat rami dan serat daun nanas. Penelitian ini dilakukan melalui metode pengkombinasian silang antara hidrogen peroksida, *deep eutectic solvent* dan metode sonikasi untuk mendapatkan hasil yang paling optimal. Hasil yang didapatkan menunjukkan bahwa proses isolasi α -selulosa berjalan paling efektif pada serat rami menggunakan larutan *deep eutectic solvent* yang dikombinasikan dengan metode sonikasi.

Kata kunci: *Bleaching*, *Deep Eutectic Solvent*, Selulosa, Serat Rami, Sonikasi.

ABSTRACT

COMBINATION SONICATION AND DEEP EUTECTIC SOLVENT IN BLEACHING FOR α - CELLULOSE ISOLATION FROM FLAX FIBER

The investigations into alternative raw materials for cellulose isolation have been conducted, with flax fiber and pineapple leaf fiber emerging as some of the materials under consideration. The efficacy of the cellulose isolation process from the botanical sources remains comparatively inadequate. This is primarily attributed to the prevailing utilization of hydrogen peroxide bleaching agents in the isolation process, which poses considerable environmental health risks. One such alternative is the utilization of deep eutectic solvents, which exhibits the capacity to isolate cellulose compounds while concurrently mitigating adverse environmental health effects. The chemical composition of the deep eutectic solvent (DES) compound, comprising two constituents, namely a hydrogen bond donor and quaternary ammonium salt facilitate the disruption of the lignocellulosic bond between lignin and cellulose, thereby enabling the isolation of cellulose from undesirable interfering compounds. The combination of deep eutectic solvent and sonication methodology in the cellulose isolation process endeavors to assess the impact of these techniques on the efficacy of the bleaching process for flax and pineapple leaf fiber. The research was conducted with cross combination of hydrogen peroxide, deep eutectic solvent, and sonication method to produce the optimum results. The research showed that the combination of deep eutectic solvent and sonication method gave the most effective results in flax fiber.

Keywords: Cellulose, bleaching, Deep Eutectic Solvent, Flax fiber, and Sonication.

TABLE OF CONTENTS

APPROVAL SHEET OF UNDERGRADUATE THESIS.....	i
VALIDATION SHEET OF UNDERGRADUATE THESIS	ii
ORIGINALITY STATEMENT.....	iii
STATEMENT OF APPROVAL FOR PUBLICATION OF SCIENTIFIC WORK FOR ACADEMIC PURPOSES.....	iv
PREFACE.....	v
ABSTRAK.....	vii
ABSTRACT.....	viii
TABLE OF CONTENTS.....	ix
LIST OF FIGURES	xii
LIST OF TABLE.....	xiv
CHAPTER I.....	1
1.1 Background.....	1
1.2 Problem Statement.....	5
1.3 Research Objectives.....	5
1.4 Theoretical Benefits.....	6
1.5 Practical Benefits.....	6
CHAPTER II	9
2.1 Theoretical Foundation	9
2.1.1 Nitrocellulose	9
2.1.2 Nitrocellulose Manufacturing Process.....	9
2.1.3 Bleaching Agent.....	10
2.1.4 Deep Eutectic Solvent.....	11
2.1.5 Sonication	11

2.2	Previous Research Results.....	12
2.3	Framework of Thinking	15
2.4	Research Hypothesis.....	16
2.4.1	First Hypothesis	16
2.4.2	Second Hypothesis	16
2.4.3	Third Hypothesis	17
CHAPTER III		17
3.1	Research Method and Design	17
3.2	Place and Time of Research	17
3.3	Tools and Material	18
3.3.1	Tools.....	18
3.3.2	Materials	19
3.4	Research Variable.....	19
3.5	Inclusion and Exclusion Criteria.....	19
3.6	Population and Sample.....	19
3.7	Research Procedure.....	20
3.7.1	Sample Preparation	20
3.7.2	Sample Characteristics Test (Moisture Content Test SNI 03-1971-1990).....	20
3.7.3	Sample Characteristics Test (α -cellulose Content Test SNI 04444-2009).....	20
3.7.4	Sample Characteristics Test (Lignin Content Test SNI 0492-2008).....	21
3.7.5	Delignification.....	22
3.7.6	Deep Eutectic Solvent Preparation	22
3.7.7	Bleaching with Hydrogen Peroxide	22

3.7.8 Bleaching Using Hydrogen Peroxide in Combination with Sonication Method	23
3.7.9 Bleaching Using Deep Eutectic Solvent	23
3.7.10 Bleaching Using Deep Eutectic Solvent in Combination	
with Sonication Method	23
3.7.11 Bleaching Using Deep Eutectic Solvent in Combination with Hydrogen Peroxide	23
3.7.12 Qualitative Analysis of The Presence of Target Compounds	24
3.7.13 Qualitative Analysis of α –Cellulose Compound	
Morphology	24
CHAPTER IV	26
4.1 The Results of α -Cellulose Content Test and Lignin Content Test in Samples before and After the Isolation Process	26
4.2 The Mechanism Reaction of α -Cellulose Content Test	27
4.3 Mechanism Reaction of Lignin Content Test	31
4.4 Sample Moisture Content Test	35
4.5 Delignification	36
4.6 Bleaching	39
CHAPTER V	53
5.1 Conclusions	53
5.2 Suggestions	53
APPENDIX	54

LIST OF FIGURES

Figure 2.1	Cellulose Nitration Reaction Mechanism.....	9
Figure 4.2.1	Mechanism reaction of NaOH and lignocellulose.....	27
Figure 4.2.2	Mechanism reaction redox of lignin and potassium dichromate.....	29
Figure 4.2.3	Reaction between Cr^{6+} that does not react to lignin with Ferrous ammonium sulfate.....	30
Figure 4.2.4	Reaction between Cr^{6+} in blank solution with ferrous ammonium sulfate.....	30
Figure 4.2.5	Sample solution (a), sample adding potassium dichromate solution (b), sample adding potassium dichromate and sulfuric acid concentrated solution (c) and sample adding potassium dichromate and sulfuric acid solution after titrating (d).....	30
Figure 4.3.1	Mechanism reaction of ethanol and lignocellulose.....	31
Figure 4.3.2	The result of reaction between Ethanol and Lignocellulose in pineapple leaf fiber (a) and flax fiber (b).....	32
Figure 4.4.1	Sample flax fiber (a) and pineapple leaf fiber.....	34
Figure 4.6.1	The filtrate of delignification flax fiber (a) and pineapple leaf fiber (b).....	36
Figure 4.6.2	The results of delignification flax fiber (a) and pineapple leaf fiber (b).....	37
Figure 4.6.3	Mechanism reaction delignification of sample using NaOH.....	37
Figure 4.7.1	The comparison of the bleaching result.....	40
Figure 4.7.2	Bleaching results of flax fiber using des; flax fiber using des and sonication, flax fiber with hydrogen	

	peroxide and sonication; flax fiber using hydrogen peroxide; and flax fiber using des and hydrogen peroxide.....	41
Figure 4.7.3	Bleaching results of pineapple leaf fiber using hydrogen peroxide and sonication and pineapple leaf fiber using hydrogenperoxide.....	42
Figure 4.7.4	Comparison of the FTIR result bleaching using DES...	43
Figure 4.7.5	Mechanism reaction of lignocellulose and DES in two step: First step (a) and second step (b).....	44
Figure 4.7.6	Mechanism reaction of lignocellulose and DES in two step: First step (a) and second step (b).....	45
Figure 4.7.7	Mechanism reaction of lignocellulose and DES in two step: First step (a) and second step (b).....	46
Figure 4.7.8	Mechanism reaction of hydrogen peroxide and lignocellulose.....	47
Figure 4.7.9	The comparison of IR spectrum in bleaching using hydrogen peroxide.....	48
Figure 4.7.10	The comparison of IR spectrum in bleaching using DES and sonication and bleaching using hydrogen peroxide and sonication.....	49
Figure 4.7.11	The effect of sonication in IR spetrum of bleaching using hydrogenperoxide.....	51
Figure 4.7.12	The effect of sonication in IR spectrum of bleaching using DES.....	51
Figure 4.7.13	The different of SEM analyzed results of DES + Sonication and Hidrogen Peroxide Sonication.....	52

LIST OF TABLE

Table 3.1	Research Schedule.....	19
Table 4.1	α -cellulose content and lignin content of flax fiber before and after the isolation.....	26
Table 4.2	Lignin content and lignin content of pineapple leaf fiber before and after the isolation.....	26

