

**EXTRACTION AND CHARACTERIZATION OF MICRO
FIBRILLATED CELLULOSE FROM TEXTILE COTTON
WASTE**

Jayasinghe Mudalige Ravisrini Jayasinghe

159483 A

Degree of Master of Science

Department of Materials Science and Engineering

University of Moratuwa.

Sri Lanka.

July 2020

**EXTRACTION AND CHARACTERIZATION OF MICRO
FIBRILLATED CELLULOSE FROM TEXTILE COTTON
WASTE**

Jayasinghe Mudalige Ravisrini Jayasinghe

159483 A

**This Dissertation submitted in partial fulfillment of the requirements for the
Degree of Master of Science in Material Science.**

Department of Materials Science and Engineering

University of Moratuwa

Sri Lanka

July 2020

Declaration

I do herewith declare that the reported work in this entire thesis and the experiment was carried out by myself under the supervision of Mr. A. M. P. B. Samarasekara and Dr. D.A.S. Amarasinghe. This thesis describes the results of my own independent experimental work except where I have mentioned due references made in the text. There was no part in this thesis submitted elsewhere as a part of an any other or the same degree program.

.....

Date

.....

J.M.R Jayasinghe

I endorse the declaration by the candidate.

.....

Mr. A. M. P. B. Samarasekara.

Supervisor.

Date:

.....

Dr. D.A.S. Amarasinghe

Supervisor.

Date:

Abstract

Cotton is a natural staple fiber that almost consist with cellulose compared to wood. The major economic value of the cotton is in Textile Industry. In the past recent years, cotton consumption demand was increased than the production. In textile industry cotton are blending with various other synthetic fibers such as polyester, nylon and lycra to obtained desirable properties. Therefore, the fabric recycling methods are quite complicated although it is highly available as pre-consumer garment waste. In this work a method was developed to identify the amount of cotton present in the cotton/polyester blended fabric by using Fourier transformed infrared (FTIR) second-order derivative spectrum. Then the cotton waste composition was determined and used to extract cellulose. Then purify cellulose was subjected to extract Micro Fibrillated Cellulose (MFC) by using acid hydrolysis method. MFC has very high economic value compared to cotton fabric waste in various applications such as bio-composites, medicine, cosmetic, pharmaceutical, tissue engineering, bio-sensors, paints and coating, flexible electronics, air filters and high tech applications including aviation and automobile.

However, the major challenge of extracting MFC is the low amount of yield in acid hydrolysis, although it considered to be as most cost effective method of MFC extraction. Laboratory extracted small quantities are not sufficient in industrial applications such as in reinforcing composites. Three experimental factors including; acid concentration, hydrolysis time and temperature show the highest effect in yield and quality of MFC. Therefor this experiment was designed to optimize the three independent factors effect on two responses of yield (%) and Width (nm) of MFC. Response surface methodology was employed to design the experiment and ANOVA statistical test results were used to determine the significance of the parameter effect on acid hydrolysis. Further extracted MFCs physical and structural properties were discussed. Morphological features and size of the fibers were examined by scanning electron microscopy (SEM), structural features and chemical functionality was determined by Fourier transformed infrared (FTIR) spectroscopy, degree of crystallinity was obtained by X-ray diffraction (XRD) spectroscopy and thermal properties were determined by Thermo gravimetric analysis (TGA).

Keywords: Micro Fibrillated Cellulose, chemical purification, acid-hydrolysis, second-order derivative spectroscopic method.

Acknowledgement

My foremost gratitude is expressed to my research supervisors Mr. A. M. P. B. Samarasekara and Dr. D.A.S. Amarasinghe for their continuous valuable guidance and great support given to me throughout this research period.

I express my sincere gratitude for all staff members of the Materials Science & Engineering department of University of Moratuwa for their kind assistance and willingness to help for success the research.

Finally, I would like to thankful all of my friends and colleagues who are not been mentioned here personally in making this work success. And specially thanks to my loving parents and brother for their patience, assistance, care and love in all the time.

Table of Content

Declaration.....	i
Abstract.....	ii
Acknowledgement	iii
1. Introduction.....	1
2. Literature Review.....	5
2.1. Cellulose Structure.....	5
2.2. Cotton.....	7
2.3. Cotton fabric recycling.....	8
2.4. Ageing of textiles during laundering	8
2.5. Extraction and Classification of Micro and Nanoscale cellulose.....	9
2.6. Optimization of extraction of nanocellulose by acid hydrolysis process.....	11
3. Methodology	13
3.1. Cotton fabric characterization.....	13
3.2. Moisture content of the cotton fabric.....	13
3.3. Ash content of the cotton fabric.....	13
3.4. FTIR analysis of cotton fabric and cotton linters.....	13
3.5. XRD analysis of cotton fabric and cotton linters	13
3.6. TGA analysis of cotton fabric and cotton linters	14
3.7. Composition analysis of cotton-polyester blend.....	14
3.8. Mixture Ratio Analysis by Beer-Lambert law	15
3.9. Chemical pre-treatment of cotton	16
3.10. Preparation of MCC.....	16
3.11. Design of experiment to optimize the acid hydrolysis parameters	17
3.12. Preparation of Microfibrillated-cellulose.....	18
4. Results and discussion	22
4.1. Analysis of the polyester amount in cotton/polyester fibre blend.....	22
4.2. Waste cotton fabric characterization.....	30
4.2.1. Moisture content of the waste cotton fabric.....	30
4.2.2. Ash content of the waste cotton fabric.....	30
4.3. Waste cotton fabric and raw cotton linters – FTIR analysis	31
4.4. XRD analysis of waste cotton fabric and cotton linters	32
4.5. TGA analysis of waste cotton fabric and cotton linters	33
4.6. Experimental design to optimized the acid hydrolysis parameters	34
4.7. Development of the regression model	34
4.8. ANOVA analysis and lack-of-fit	35

4.9.	Process Variables Optimization	39
4.10.	Contour Plots and surface plot of width of MFC.....	40
4.11.	Contour plots and surface plot of Yield of MFC	43
4.12.	FTIR Characterization of the MFC.....	47
4.13.	Morphology of the MFC.....	50
4.14.	Crystallinity of MFC.....	55
4.15.	Thermal degradation behaviour of MFC	58
5.	Conclusion	59
6.	References.....	61

List of Figures.

Figure 1 : Classification of cellulose [67]	10
Figure 2: Preparation of MCC from cotton fabric	17
Figure 3: Preparation of MFC by using MCC as the starting material	20
Figure 4: Acid hydrolysis slurry of cellulose	20
Figure 5: Final freeze dried sample	21
Figure 6: Characteristic absorption spectrums of cotton/polyester blended samples	22
Figure 7: The FTIR spectrum of 100% cotton and the 100% polyester	25
Figure 8: The FTIR first-order derivative spectrum wavenumber range of 1573 - 1860 cm^{-1} .	26
Figure 9: The FTIR second order derivative spectrum wavenumber range of 1573 - 1860 cm^{-1}	26
Figure 10: Absorption versus amount of polyester present in fiber blend	29
Figure 11: Absorption versus amount of polyester present in fiber blend	29
Figure 12: FTIR spectra of celluloses present in the cotton linters and the cotton fabric	31
Figure 13: X-ray diffraction patterns of raw cotton linters and cotton fabric	32
Figure 14: TGA curve for cotton linters and the waste cotton fabric	33
Figure 15: Parato chart of standardized effect of concentration, time and temperature on width of MFCs	37
Figure 16: Pareto chart of standardized effect of concentration, time and temperature on yield of MFCs (response is yield (%), $\alpha = 0.05$)	39
Figure 17: Surface plot of mean width vs. time and concentration (hold temperature)	40
Figure 18: Contour plot of mean width vs. time and concentration (hold temperature)	40
Figure 19: Surface plot of mean width vs. temperature and concentration (Hold time)	42
Figure 20: Contour plot of mean width vs. temperature and concentration (hold time)	42
Figure 21: Surface plot of yield vs. time and concentration (hold temperature)	43
Figure 22: Contour plot of yield vs. time and concentration (hold temperature)	43
Figure 23: Surface plot of yield vs. time and concentration (hold time)	45
Figure 24: Contour plot of yield vs. time and concentration (hold time)	45
Figure 25: FTIR Spectrum of MFC samples	48
Figure 26: FTIR spectra of celluloses studied in the region between 1512 cm^{-1} to 800 cm^{-1}	49
Figure 27: Cellulose fibres of sample MFC 8	50
Figure 28: Cellulose fibers of sample MFC-09	51
Figure 29: Cellulose fibres of sample MFC 2	52
Figure 30: Cellulose fibers of sample MFC 14	53
Figure 31: XRD patterns of MFC	56
Figure 32: TGA curves of MFC	58

List of Tables

<i>Table 1: Cotton Polyester blend weights and identification.</i>	14
<i>Table 2: Parameter levels of experiment</i>	18
<i>Table 3: Experimental coded values and results of Mean width and Yield</i>	19
<i>Table 4: Identification of cotton polyester FTIR Wave number values [70]</i>	23
<i>Table 5: Summary of the FTIR spectroscopic data for polyester in each series.</i>	28
<i>Table 6: The moisture content of the waste cotton fabric</i>	30
<i>Table 7: The Ash content of the waste cotton fabric</i>	30
<i>Table 8: Independent variable used for the experiment and their coded values</i>	35
<i>Table 9: ANOVA statistical analysis and the lack-of-fit test results for width of the MFC</i>	36
<i>Table 10: Modal summary</i>	36
<i>Table 11: the ANOVA statistical analysis and the lack-of-fit test results percentage yield of the MFC.</i>	38
<i>Table 12: Model summary</i>	38
<i>Table 13: Microfibrilated cellulose samples that gives minimum values for the width.</i>	46
<i>Table 14: The crystal structure given band position in $2\theta^\circ$ and their d-spacing values.</i>	57