การคัดแปรผ้าฝ้ายด้วยสารประกอบแคต ใอออนิกรีแอคทีฟในการฟอก เพื่อเพิ่มการรับสีย้อมรีแอคทีฟ

นางสาวพูลสิริ รัตนนิยมกุล



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต สาขาวิชาวิทยาศาสตร์พอลิเมอร์ประยุกต์และเทคโนโลยีสิ่งทอ ภาควิชาวัสคุศาสตร์ คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2544

ISBN 974-03-0384-6

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

MODIFICATION OF COTTON FABRIC WITH CATIONIC REACTIVE COMPOUNDS IN BLEACHING TO IMPROVE REACTIVE DYEABILITY

Miss Pulsiri Rattananiyomkul

A Thesis Submitted in Partial Fulfillment of the Requirements

for the Degree of Master of Science in Applied Polymer Science and Textile Technology

Department of Materials Science

Faculty of Science

Chulalongkorn University

Academic Year 2001

ISBN 974-03-0384-6

Thesis Title	Modification of Cotton Fabric with Cationic Reactive	
	Compounds in Bleaching to Improve Reactive Dyeabilis	ty
Ву	Miss Pulsiri Rattananiyomkul	
Field of Study	Applied Polymer Science and Textile Technology	
Thesis Advisor	Assistant Professor Kawee Srikulkit, Ph.D.	
Accep	ted by the Faculty of Science, Chulalongkorn University in Partial	
Fulfillment of the Requ	irements for the Master's Degree	
*eacl	Dean of Faculty of Science	
	(Associate Professor Wanchai Phothiphichitr, Ph.D.)	
THESIS COMMITTEE		
	S. Chuaypulgit Chairman	
	(Associate Professor Saowaroj Chuayjuljit)	
	K Switzultit Thesis Advisor	
	(Assistant Professor Kawee Srikulkit, Ph.D.)	
	Payson Santel Member	
	(Associate Professor Paiparn Santisuk)	
	lls S. Member	
	(Usa Sangwatanaroj, Ph.D.)	
	Sirest Chauchide Member	

(Sireerat Charuchinda, Ph.D.)

นางสาว พูลสิริ รัตนนิยมกุล: การคัดแปรผ้าฝ้ายคัวยสารประกอบแคตใจออนิกรีแอกทีฟ ในการฟอกเพื่อเพิ่มการรับสีย้อมรีแอกทีฟ (MODIFICATION OF COTTON FABRIC WITH CATIONIC REACTIVE COMPOUNDS IN BLEACHING TO IMPROVE REACTIVE DYEABILITY) อ. ที่ปรึกษา: ผศ.ดร. กาวี ศรีกูลกิจ, 92 หน้า. ISBN 974-03-0384-6.

สารคัดแปรสมบัติการคูคติคสีรีแอคที่ฟของผ้าฝ้าย 3-amino-2-hydroxyl-propyltri methylammonium(3,5)-dichlorotriazine ได้ถูกสังเคราะห์และนำมาวิเคราะห์โครงสร้างโดยใช้ เทคนิคทางสเปกโทรสโกปี หลังจากนั้นนำสารคัดแปรไปผนึกลงบนผ้าฝ้ายในกระบวนการฟอก ขาวผ้า ผ้าที่ผ่านการคัดแปรแล้วได้นำมาวิเคราะห์หาเปอร์เซ็นต์ธาตุในโตรเจนและทดสอบความ ขาวของผ้า เปอร์เซ็นต์ธาตุในโตรเจนในผ้าฝ้ายคัดแปรจะเพิ่มขึ้นตามปริมาณความเข้มข้นของสาร คัดแปรที่ใช้ ในขณะที่ความขาวของผ้าจะลดลงเล็กน้อยแสดงว่าสามารถผนึกสารคัดแปรไปพร้อม กับการทำฟอกขาวผ้าได้และประสิทธิภาพการฟอกขาวของสารไฮโครเจนเปอร์ออกไซด์ในขณะที่มี สารคัดแปรอยู่ด้วยลดลงเล็กน้อย

ผลการข้อมผ้าฝ้ายคัดแปรด้วยสีรีแอกทีฟในภาวะไร้เกลือ ทำให้ผ้าฝ้ายมีการคิดสีสูงขึ้นตาม ปริมาณของสารคัดแปรที่ใช้ และสูงกว่าผ้าฝ้ายที่ไม่ได้ผ่านการคัดแปร ทั้งนี้เนื่องจากประจุบวกที่อยู่ บนสารคัดแปรซึ่งจะคึงคูดประจุลบของสีรีแอกทีฟได้ดี และจากผลการย้อมทำให้สรุปได้ว่าสมบัติ การย้อมผ้าฝ้ายที่ผ่านการคัดแปรจะขึ้นกับประสิทธิภาพในการยึดติดของสารคัดแปรบนเส้นใยเซลลู โลสในขั้นตอนการคัดแปร

ภาควิชาวัสคุศาสตร์ สาขาวิชาวิทยาศาสตร์พอถิ:มอร์ประยุกต์ฯ ปีการศึกษา2544 ลายมือชื่อนิสิต <u>พุงส์งั้งสนุนิยม กุล</u> ลายมือชื่ออาจารย์ที่ปรึกษา <u>กษั</u> <u>Nthana</u> ลายมือชื่ออาจารย์ที่ปรึกษาร่วม _____ ## 4272357223: MAJOR APPLIED POLYMER SCIENCE AND TEXTIE TECHNOLOGY KEYWORD: COTTON FABRIC / MODIFYING AGENT / BLEACHING / SINGLE-BATH

PULSIRI RATTANANIYOMKUL: MODIFICATION OF COTTON FABRIC WITH CATIONIC REACTIVE COMPOUNDS IN BLEACHING TO IMPROVE REACTIVE DYEABILITY. THESIS ADVISOR: ASSIST.PROF.KAWEE SRIKULKIT, Ph.D., 92 PAGES, ISBN 974-03-0384-6.

The reactive dyeability modifying agent of cotton fabric, 3-amino-2-hydroxyl-propyltrimethylammonium(3,5)-dichlorotriazine, was prepared. Spectroscopic technique (¹H NMR) was employed to characterize the chemical structure of the modifying agent. After that, the modifying agent was applied onto scoured cotton during bleaching process, aiming at modifying dyeability of the fiber using single-bath. The nitrogen content of modified cotton fabric was analysed by an elemental analyzer and whiteness was also evaluated. The results showed that nitrogen content in modified cotton fabric increased with an increase in the amount of the modifying agent concentration whereas whiteness index slightly decreased. Based on the finding results, it was possible that the dyeability modification and bleaching could be carried out in single bath process with a slight decrease in the performance of hydrogen peroxide.

The dye uptake and color strength of dyed modified fabric were markedly increased with an increase in the concentration of the modifying agent. In addition, those properties obtained from modified cotton fabric were higher than that obtained from the dyeing from unmodified cotton fabric. This was attributed to the presence of the cationic groups of the modifying agent that played a crucial role in attracting the anionic dyes from the dye-bath. The finding results tended to suggest that the dyeing properties of modified fabric were exactly dependent on the efficiency of the modifying agent fixation on cellulose during concurrent modifying and bleaching of cotton.

Department Materials Science

Field of study Applied Polymer Science and Textile Technology

Academic year 2001

Student's signature Pulsiri Rattananiyomkul.

Advisor's signature... K. Shtulky

Co-advisor's signature.....



I wish to express my deep gratitude to Asst. Prof. Dr. Kawee Srikulkit, my advisor, for his valuable guidance, advice and encouragement throughout this research work.

I am grateful to Assoc. Prof Saowaroj Chauyjuljit, Assoc. Prof. Paiparn Santisuk, Dr.Usa Sangwatanaroj and Dr. Sireerat Charuchinda for their comments and assistance for my study and the Department of Materials Science, Chulalongkorn University for the graduate course.

I wish to thank my lecturers and friends at the Department of Materials Science, especially to Miss Patcharee Larpsuriyakul for her kind assistance, and Mr. Nirun Thaipreecha for his helpful encouragement.

My thanks is also extended to the administration staff of the Materials Science Department for their assistance.

Finally, I would like to express my deepest appreciation to my family for their love, encouragement, and worthy moral and financial support throughout my study at Chulalongkorn University.

CONTENTS

		Page
Abstract ((Thai)	iv
Abstract (English)		V
Acknowledgement		vi
List of Tables		x
List of Figures		xii
List of Schemes.		xiv
Chapter		
I	Introduction	1
П	Literature Survey	4
	Cellulosic Fiber	4
	Bleaching	15
	Choosing Dyes of Suitable Substantivity	17
	Reactive Dyes for Cellulose	18
	Concept of the Modification and Bleaching of Cotton Fabric	
	In Single-bath Process	37
III	Experimental Procedures	40
	Materials	40
	Equipment	41
	Synthesis of the Modifying Agent, 3-amino-2-hydroxyl-	
	propyltrimethyl ammonium(3,5)-dichlorotriazine	42
	Method of the Modification and Bleaching of Cotton Fabric in	
	Single-bath Process	45
	General Dyeing Procedure	46

Contents (continued) Pa		Page	
		NMR Analysis	49
		Total Nitrogen Content Determination	49
		Evaluation of Whiteness Property of Modified Bleached	
		Cotton Fabric	50
		Measurement of Dye Exhaustion	50
		Measurement of Dye Fixation	51
		Image Microscopic Analysis (Fiber Cross Sectional	
		Examination)	52
		Measurement of Colorfastness	53
	IV	Results and Discussion.	55
		Synthesis of Modifying Agent: 3-amino-2-hydroxyl-propyltri	
		Methyl ammonium(3,5)-dichlorotriazine	55
		NMR Study of the Intermediate I, Intermediate II and	
		Modifying Agent	58
		Effect of the Modifying Agent Concentration on Whiteness	
		Property	64
		Total Nitrogen Content Determination	66
		Effect of Increasing Concentration of Modifying Agent on	
		Dye Uptake and Dye Fixation	67
		Effect of Different Dye Types on Dye Uptake and Color Yield	69
		Effect of Increasing Dye Concentration on Dye Uptake and	
		Color Strength	70
		Evaluation of Color Fastness to Light	73
		Microscopic Analysis of Cross-section of Dyed Yarn	74
		Dyeing of Unmodified Cotton Fabric	76
	V	Conclusions	79
	VI	Recommendations	81

References	82
Appendix	85
Biography	92

List of Tables

Tables		Page
2.1	Typical values for thr composition of a mature dry cotton fiber	10
2.2	Properties of cotton	11
2.3	Impurities present in cellulosic fibers (% by weight)	14
2.4	World consumption of dyes for cellulosic fibers by dye class	19
2.5	Classification of reactive dyes by substantivity	21
3.1	List of chemicals used in this project	40
3.2	Dyes for blue wool references 1 to 8	53
4.1	Chemical shifts of (3-chloro-2-hydroxypropyl)trimethylammonium	
	chloride and intermediate I	60
4.2	Chemical shifts of glycidyltrimethyl ammonium chloride and	
	intermediate II	62
4.3	Chemical shifts of modifying agent: 3-amino-2-hydroxyl-propyltri	
	methylammonium-dichlorotriazine	64
4.4	Whiteness index of modified bleached cotton fabric at various	
	modifying concentrations	65
4.5	Total nitrogen content of modifying agent on cotton fabric after	
	H ₂ O ₂ /NaOH	66
4.6	Color yield, % dye exhaustion and % fixation of modified fabrics at	
	various modifying concentrations	68
4.7	Color yield, % dye exhaustion and % fixation of modified fabrics at	
	different dye types	69
4.8	Effect of increasing dye concentration on dyeing properties of	
	modified fabric	71
4.9	Effect of increasing dye concentration on degree of dye fixation (%F)	
	and total dye fixation (%T) of modified fabric	71

Tables (continued)		Page
4.10	Light fastness of dyed cotton fabrics modified with various	
	concentrations of modifying agent	73
4.11	Color yield, and % dye exhaustion of unmodified fabrics dyed with	
	various concentrations of electrolyte	76
4.12	The degree of dye fixation (%F) and total dye fixation (%T) of	
	unmodified fabrics dyed with various concentrations of	77
	electrolyte	
4.13	Light fastness of unmodified cotton fabrics dyed with various	
	concentrations of dyes and electrolyte	77

List of Figures

Figur	ures	
2.1	Structure of cellulose	5
2.2	Cellulose fully extended conformational formula (flat ribbon)	6
2.3	Bilateral structure of mature cotton	8
2.4	Scanning electron micrographs of raw cotton fibers	8
2.5	Optical micrographs of raw cotton fibers x 184	8
2.6	Idealised diagram of cotton morphology	9
2.7	Relative reactivities of reactive dyes for cellulose fibers	18
2.8	Characteristic structural features of a reactive dye	22
2.9	Important representatives of these reactive groups	25
2.10	Figure 2.10	26
2.11	The specific base catalyzed addition of the nucleophilic functional	
	group HY of the textile fiber	26
2.12	Figure 2.12	33
2.13	Figure 2.13	38
3.1	Ahiba Polymat [®] laboratory dyeing machine	45
3.2	Macbeth spectrophotometer	50
3.3	UV/VIS spectrophotometer	51
3.4	Optical microscope.	53
3.5	Xenon Weather Meter	54
4.1	The modifying agent, 3-amino-2-hydroxyl-propyltrimethyl	
	ammonium(3,5)-dichlorotriazine.	55
4.2	Titration curve of intermediate I and standard hydrochloric acid	
	solution	56
4.3	Titration curve of intermediate II and standard hydrochloric acid	
	solution	57

Figu	Figures (continued)	
4.4	¹ H NMR spectrum of (3-chloro-2-hydroxypropyl)trimethyl	
	ammonium chloride using D ₂ O as solvent	59
4.5	¹ H NMR spectrum of intermediate I using D ₂ O as solvent	59
4.6	¹ H NMR spectrum of glycidyltrimethyl ammonium chloride using	
	D ₂ O as solvent	61
4.7	¹ H NMR spectrum of intermediate II using D ₂ O as solvent	62
4.8	¹ H NMR spectrum of modifying agent: 3-amino-2-hydroxyl-propyl	
	trimethylammonium-dichlorotriazine using D_2O as solvent	63
4.9	Whiteness index chart of modified bleached cotton fabric	65
4.10	Chart of the exhaust dyeing of modified cotton	68
4.11	Chart of the exhaust dyeing of modified cotton at 2% owf dye	
	concentration at different dye types	70
4.12	Chart of the exhaust dyeing of 30 g/l modifying agent at various dye	
	concentration: Procion Crimson CX-B	72
4.13	Optical micrograph of modifying agent treated yarn dyed with 2%	
	owf: Procion Crimson CX-B	75
4.14	Optical micrograph of modifying agent treated yarn dyed with 2%	
	Owf: Modercion yellow HE4R	75

List of Schemes

Sc	hemes	Page
	Scheme 2.1	5
	Scheme 2.2.	6
	Scheme 2.3	15
	Scheme 2.4.	16
	Scheme 2.5	16
	Scheme 2.6.	16
	Scheme 2.7.	24
	Scheme 2.8.	25
	Scheme 2.9.	26
	Scheme 2.10	28
	Scheme 2.11	29
	Scheme 2.12	29
	Scheme 2.13	30
	Scheme 2.14	31
	Scheme 2.15	32
	Scheme 2.16	32
	Scheme 2.17	34
	Scheme 2.18.	35
	Scheme 2.19	36
	Scheme 2.20	36
	Scheme 2.21.	38
	Scheme 2.22.	39
	Scheme 3.1.	43
	Scheme 3.2	44

List of Diagrams

Diagrams		Page
3.1	Concurrent bleaching and modifying cotton in single bath	46
3.2	The dyeing profile of modified fabric using exhaustion method	47
3.3	Exhaust dyeing of unmodified fabric	48