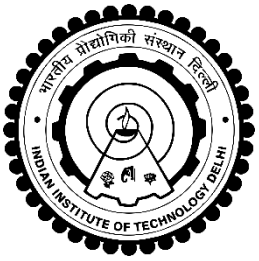
ECO FRIENDLY PROCESSING OF WOOL

SRIKRISHNA NATARAJAN



DEPARTMENT OF TEXTILE TECHNOLOGY

INDIAN INSTITUTE OF TECHNOLOGY DELHI

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ECO FRIENDLY PROCESSING OF WOOL

by

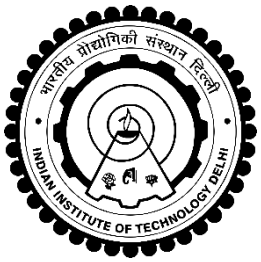
SRIKRISHNA NATARAJAN

Department of Textile Technology

Submitted

In fulfillment of the requirements for the award of the degree of Doctor of Philosophy

to the



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CERTIFICATE

This is to certify that the thesis entitled "**Eco friendly processing of wool**" submitted by **Mr. Srikrishna Natarajan** to the **Indian Institute of Technology Delhi** for the award of the degree of **Doctor of Philosophy**, in the Department of Textile Technology, is a record of bonafide research work carried out by him. Mr. Srikrishna Natarajan has worked under my guidance and supervision and has fulfilled the requirements for the submission of this thesis.

The results contained in this thesis are original and have not been submitted in partial or full, to any other university or institute for the award of any degree or diploma.

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(Srikrishna Natarajan)

ABSTRACT

In this thesis, industrial waste products of natural origin were used to impart value adding properties to wool fabric.

Wool was coloured by *in situ* polymerization of catechol by potato juice using one and two bath method. In the two bath process, maximum colour depth was obtained with 4% catechol w/v, pH 4.5, 95°C. For the one step method, a statistical design of experiments was employed to determine the optimum parameters for obtaining a range of colours on wool. Mechanism of colour formation was attributed to formation of melanin by oxidative polymerization of catechol by potato juice. Structure of colourant showed the presence of ether linkages. Strength, elongation, wetting and antioxidant property of wool was enhanced after colouration. Theoretical studies on kinetics of colour formation reaction showed catechol to be an excellent substrate for potato juice. Kinetic constants for the reaction V_{Max} and K_M were found to be 0.01814 $\pm$ 0.002 Abs/min and 0.4148 mM respectively.

Melanin produced for colouration of wool in the first study, was successfully used as a simultaneous reductant cum capping agent for synthesis of silver nano particles from silver nitrate. Process parameters were optimized (0.1% catechol, 25% potato juice, pH 6.5, 95^0C,45 min) to obtain melanin having maximum reduction potential. Monodispersed, spherical silver nanoparticles, with average particle size of 45 ± 5 nm were obtained with 100% yield. The nanoparticles were applied to wool fabric by exhaust method. Treated fabric showed high antimicrobial activity against *S.aureus* and *E.coli*.

A robust and reproducible test method was developed for testing shrinkage of wool fabric in the laboratory, to simulate the results obtained by ISO 6330 test method. The proposed test method involves Relaxation shrinkage test (1 g/l detergent, pH 6, 40˚C, 60 minutes) followed by Felting shrinkage test (0.3g/l detergent, pH 6, 40˚C, 60min, 1:20) on samples of size 12x12cm. The process can be tuned to simulate various laundering conditions.

Wool was made shrink proof by an eco friendly process based on functionalization of wool by UV exposure, followed by coating of scales by sericin. Treated wool fabric showed smooth appearance and enhanced dyeability by acid and reactive dyes.

सारांश

इस थीसिस में, प्राकृतिक मूल के औद्योगिक अपशिष्ट पदार्थों का प्रयोग ऊन कपड़े के गुणों को जोड़ना मूल्य प्रदान करने के लिए किया गया था।

एक और दो स्नान पद्धति का उपयोग करके आलू का रस द्वारा कैटेचॉल की स्वस्थानी बहुलकीकरण द्वारा ऊन रंगा हुआ था। दो स्नान प्रक्रिया में, अधिकतम रंग गहराई 4% कैटेकॉल वाई / वी, पीएच 4.5, 95 डिग्री सेल्सियस से प्राप्त की गई थी। एक कदम विधि के लिए, ऊन पर रंगों की एक श्रृंखला प्राप्त करने के लिए इष्टतम मापदंडों को निर्धारित करने के लिए प्रयोगों के एक सांख्यिकीय डिजाइन का इस्तेमाल किया गया था। आलू के रस द्वारा कैटेचोल के ऑक्सीडेटिव बहुलकीकरण द्वारा मेलेनिन के गठन के लिए रंग गठन के तंत्र को जिम्मेदार ठहराया गया। रंगीन की संरचना ने ईथर लिंक की उपस्थिति दिखायी। रंगापन के बाद ऊन की शक्ति, बढ़ाव, गीला और एंटीऑक्सिडेंट गुण बढ़ाया गया। रंग गठन की प्रतिक्रिया के कैनेटीक्स पर सैद्धांतिक अध्ययन ने आलू के रस के लिए एक उत्कृष्ट सब्सट्रेट साबित किया है। प्रतिक्रिया VMax और केएम के लिए काइनेटिक स्थिरांक क्रमशः 0.01814 + 0.002 एबीएस / मिनट और 0.4148 एमएम पाए गए।

मेलेनिन ने पहले अध्ययन में ऊन के रंगाई के लिए उत्पादित किया, जिसका उपयोग चांदी नाइट्रेट से चांदी नैनो कणों के संश्लेषण के लिए एक साथ रिडाकटेंट कम कैपिंग एजेंट के रूप में किया गया। अधिकतम मापन क्षमता वाले मेलेनिन प्राप्त करने के लिए प्रक्रिया मापदंडों का अनुकूलन किया गया (0.1% कैटेचॉल, 25% आलू का रस, पीएच 6.5, 950 सी, 45 मिनट)। मोनोडाइस्डर्ड, गोलाकार रजत नैनोकणों, जो कि औसत कण आकार 45 ± 5 एनएम के साथ 100% उपज के साथ प्राप्त किया गया था। निकास विधि द्वारा ऊन कपड़े के लिए नैनोकणों को लागू किया गया था। उपचारित कपड़े एस। एयरस और ईकोली के खिलाफ उच्च रोगाणुरोधी गतिविधि दिखाते हैं।

आईएसओ 6330 परीक्षा पद्धति द्वारा प्राप्त परिणामों को अनुकरण के लिए, प्रयोगशाला में ऊन कपड़े के सिकुड़ने के परीक्षण के लिए एक मजबूत और प्रतिलिपि प्रस्तुत करने योग्य परीक्षण विधि विकसित की गई थी। प्रस्तावित परीक्षण पद्धति में आराम से संकोचन परीक्षण (1 ग्राम / एल डिटर्जेंट, पीएच 6, 40 ° सी, 60 मिनट) शामिल है, जिसके बाद छाछ के परीक्षण का परीक्षण किया जाता है (0.3 ग्राम / डी डिटर्जेंट, पीएच 6, 40 ° सी, 60 मिनट, 1:20) आकार 12x12cm के नमूनों पर प्रक्रिया को विभिन्न शोधन वाली स्थितियों का अनुकरण करने के लिए ट्यून किया जा सकता है
ऊन को यूको अनावरण द्वारा ऊन के कार्यात्मककरण के आधार पर पारिस्थितिकी के अनुकूल प्रक्रिया द्वारा सिकोड़ित किया गया था, इसके बाद सीरिसिन द्वारा तराजू कोटिंग किया गया था। ट्रेडेड ऊन कपड़े में अम्ल और प्रतिक्रियाशील रंजियों द्वारा चिकनी दिखने और बढ़ाया डाइवियो दिखाया गया था।

CONTENT SUMMARY

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF SYMBOLS & ABBREVIATIONS

S.No	Symbol/Abbreviation	Full form
1	AOX	Absorbable organic halogen
2	18-MEA	18-methyl eicosanoic acid
3	CMC	Cell membrane complex
4	DFE	Directional frictional effect
5	L-DOPA	L-3, 4 dihydroxy phenyl alanine
6	HRP	Horse radish peroxidases
7	PPO	Polyphenol oxidases
8	DABSA	2,5, diamino benzene sulfonic acid
9	ABTS	2, 2 azino-bis-(3-ethylthiazoline-6 sulfonate
10	HBT	1- hydroxybenzotriazole
11	DMP	2, 6- Dimethoxyphenol
12	EDC	3-(3-dimethyl aminopropyl) carbodiiamide hydrochloride
13	BSA	Bovine serum Albumin
14	BR	Bradford reagent
15	EDTA	Ethylene di amine tetra acetic acid
16	E	Enzyme
17	ES	Enzyme substrate complex
18	V_0	Change in initial rate or velocity
19	[S]	Substrate
20	V_{Max}	Velocity maximum at the maximum substrate concentration
21	K_M	Michaelis constant
22	FC reagent	Folin-Ciocalteau (FC) reagent
23	GAE	Gallic acid equivalent
24	RFD	Ready for dyeing

S.No	Symbol/Abbreviation	Full form
25	HT-HP	High temperature high pressure
26	% v/v	% Volume/Volume
27	µl	Microliter
28	µg/ml	Microgram/milliliter
29	% w/vol	% Weight/Volume
30	mg/l	Milligram/litre
31	g/mole	Gram/mole
32	g/l	Gram per litre
33	eV	Electron volt
34	KV	Kilo volt
35	Min	Minutes
36	DOE	Design of experiments
37	RSM	Response surface methodology
38	PJ	Potato juice
39	DPPH	2.2- diphenyl-1-picryl-hydrazil
40	D.I water	Deionized water
41	ISO	International organization for standardization
42	AATCC	American Association of Textile Chemists and Colorists
43	ASTM	American Society for Testing and Materials
44	TLC	Thin layer chromatography
45	NMR	Nuclear magnetic resonance
46	IR	Infrared
47	CFU	Colony forming units
48	K/S	Colour value
49	L^*	Lightness or darkness
50	a^*	Redness or greenness

S.No	Symbol/Abbreviation	Full form
51	b^*	Blueness or yellowness
52	ANOVA	Analysis of variance
53	XRD	X-ray diffraction
54	FT-IR	Fourier transform infrared spectroscopy
55	*S. aureus*	*Staphylococcus aureus*
56	*E.coli*	*Escherichia coli*
57	*B.mori*	*Bombyx mori*
58	RSA	Radical scavenging activity
59	UV-Vis	Ultraviolet/visible
60	DLS	Dynamic light scattering
61	TEM	Transmission electron microscopy
62	ICP-MS	Inductively coupled plasma mass spectrometry
63	µm	Micrometer
64	Nm	Nanometer
65	Gf	Gram force
66	gm/m^2	Gram per metre square
67	kDa	Kilo Dalton
68	DNA	Deoxyribonucleic acid
69	ATP	Adenosine triphosphate
70	DMF	*N*-*N*-dimethyl formamide
71	PVP	Polyvinyl pyrrolidone
72	PMVE	Poly methyl vinyl ether
73	SDS	Sodium dodecyl sulphate
74	CTAB	Cetyl trimethyl ammonium bromide
75	SNSE	Sulfur nano-silver ethanol
76	SPR	Surface plasmon resonance
77	PCS	Photon correlation spectroscopy

S.No	Symbol/Abbreviation	Full form
78	QELS	Quasi elastic light scattering
79	SEM	Scanning electron microscopy
80	EDX	Energy-dispersive X-ray spectroscopy
81	TOF-SIMS	Time of flight secondary ion mass spectroscopy
82	LMIG	Liquid metal ion gun
83	UHV	Ultra high vacuum
84	YI	Yellowness Index
85	M	Mole
86	mM	Milli mole
87	ppm	Parts per million
88	Ag	Silver
89	TPI	Twist per inch
90	Epc	Ends per cm
91	PPc	Picks per cm
92	T.B	Tris hydroxymethyl aminomethane buffer
93	MCT/VS	Monochlorotriazine/Vinyl sulphone
94	S.D	Standard deviation
95	MLR	Material to liquor ratio
96	S.E	Standard error
97	C.V	Coefficient of variation
98	TGases	Transglutaminases
99	DMDHEU	Dimethyl dihydroxy ethylene urea
100	Rpm	Revolutions per minute
101	7A	Relaxation cycle
102	5A	Felting cycle
103	RH	Relative humidity