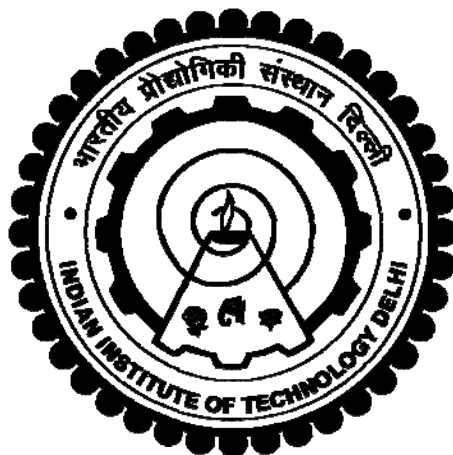


STUDIES ON VANILLIN–BASED DERIVATIVES AS
MULTI-FUNCTIONAL AGENTS FOR TEXTILE
SUBSTRATE

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DEPARTMENT OF TEXTILE & FIBRE ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY DELHI

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by

VEERENDER SHARMA

Department of Textile & Fibre Engineering

Submitted

in fulfilment of the requirements of the degree of Doctor of Philosophy

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

May 2023

DEDICATED
TO
MY GUIDE, FATHER & CHILDREN

CERTIFICATE

This is to certify that the thesis entitled ‘**STUDIES ON VANILLIN-BASED DERIVATIVES AS MULTI-FUNCTIONAL AGENTS FOR TEXTILE SUBSTARTE**’, being submitted by **Mr. Veerender Sharma**, to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of bonafide research work carried out by him. He has worked under my guidance and supervision and fulfilled the requirements for submission of the thesis which has attained the standard required for a Ph.D. degree of this Institute.

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



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ABSTRACT

The demand for functional textiles has been increased exponentially for various end applications. It has been reported that market for functional textile finishing agents is growing at the rate of 6.1% cumulative annual growth rate (CAGR) and the market for functional textile agents at US is expected to reach 162.5 million USD dollar in 2025. Among the functional textiles, antimicrobial textiles are growing at a very faster rate because of recent happened deadly Covid 19 pandemic wherein textiles in the form of PPEs played a major role. On the other hand, textiles act as a carrier for germs and spread the infection very fast. Spread of infection through textiles can be controlled by using antimicrobial finishing treatment to it. Textiles are one of the primary needs for everyone. Sickness, mortality rate because of various infection spread by normal textiles can be minimized at rural area by making these textiles functional. Not only antimicrobial textiles, but there is also a growing demand for other functional properties imbued into textiles which can provide additional protection to human being such as protection from UV light, fire among the others. Therefore, there has always been a research trend to find novel finishing agents and their exploration on textile fabric to get multiple functionalities together. But demand has also grown to look for the finishing agents which should be safe, economical, and industrially viable as well as stable at temperature of integration to textiles. Thus, development of multifunctional textiles having various properties together such as antimicrobial, UPF, fragrance, colour, etc. using safe functional agent is currently of high priority research area.

At first vanillin alone and vanillin in presence of metals have been explored as multifunctional finishing agent on both the cotton and PET textiles. However, vanillin

alone has not provided durable function properties to treated fabric and elutes out after washing. But among the metal ions (Cu, Fe, Zn) studied, copper complex with vanillin (vanillin-Cu) has provided excellent functionalities such as 99% antimicrobial activity and 50+ UPF to cotton textiles. V-Cu has also been explored on PET fabric and the treated textile has provided similar functionalities to PET fabric.

Further, vanillyl alcohol has been synthesized, characterized, and explored alone on cotton and PET fabric. Vanillyl alcohol (VA) having mild fragrance than vanillin can be thought of as a better choice for many people. VA treated fabric has shown enhanced UPF (nearly 35) in case of cotton due to increased interaction with celluloses backbone, but due to small size it again washes out from cotton and PET textiles. To tackle this problem, vanillyl alcohol complex with Cu has been prepared in-situ and integrated on cotton and PET textile using high temperature high pressure (HT-HP) dyeing machine. The treated fabric has depicted improved functional properties to a greater extent, especially, antimicrobial and UV resistance property have been reached to excellent category. Treated fabric can also retain its functional properties up to 90 washes.

In the next study, Schiff base of vanillin (SB) has been synthesized through a new approach with high yield. It has been hypothesized that Schiff base having larger size would interact with the fabric strongly and be retained inside the cotton and PET structure. Integration of SB inside cotton and PET fabric has been done using HT-HP treatment process. It has been observed that treated fabrics provide good functional properties, like antimicrobial activity of nearly 90%, good UPF and beautiful colouration which confirm the effective retention of SB than vanillin and vanillyl alcohol within the textile structure. However, treated fabric properties have been observed to be retained till 3 machine wash only. To improve the wash

durability SB-Cu complex has been synthesized in-situ and integrated on cotton and PET textiles which provide excellent and durable (18 machine wash, i.e. 90 home washes) functional properties on to treated textiles.

In the next study, another new derivative, reduced Schiff base (RSB) has been synthesized using a single step process with high yield. Synthesized RSB molecules have been characterized and integrated on cotton and PET textiles. The treatment enhances the functional properties like antimicrobial activity up to 95% and antioxidant properties more than 90% for both types of fabric. Functional properties of the treated fabric have been found to be retained till 6 machine washes and thereafter the functional properties get diminished. To further enhance the functional properties, RSB-Cu has been synthesized in-situ on textiles using HT-HP process. The treated fabrics have displayed significant improvement in functional properties like increased antimicrobial property up to 99% along with excellent UPF and 50% antioxidant activity with attractive shade. The properties also have been found to be retained till 90 washes.

In a further study, vanillin reactive finish (VRF) has been synthesized and characterized. In the previous sections of the thesis work, it has been found that all the vanillin-based active ingredients retain in the textile structure due to physical interaction and properties get decreased after each wash. Thus, covalent bond formation between the cotton fabric and vanillin has been conceptualized and VRF has been synthesized successfully. The integration of VRF on cotton fabric has been done at 60°C (which is the optimized temperature) in alkaline pH. By this approach, finishing agent becomes a part of fabric and has shown long lasting functional effects. However, -OH group of vanillin is blocked in VRF and therefore antimicrobial activity of treated fabric has been found to be decreased

to 50%. Nevertheless, UPF value of treated fabric has been increased to 50 which even can be retained at a nearly 40 even after 90 washes. VRF treated fabric has advantage of having white colour and excellent UPF property. This finish has been explored on cotton fabric only.

In the later part of the thesis, comparison of all finishes in terms of multifunctional efficacy has been done in a very systematic way. Physical properties of the treated textiles have also been studied to check any deterioration of basic properties of the fabric because of finishing treatment. However, it has been proven that all finishes are safe and do not change handle of fabric as well as other useful properties like water absorbance, tensile strength and air permeability. Finally, mechanisms of UV resistance, antimicrobial activity and antioxidant activity have also been studied using UV-Visible spectroscopy, SEM and TEM analysis and DPPH test.

At the end, it has been concluded that vanillin and all other vanillin-based synthesized finishes are safe, and provide very useful functional properties to textile substrates without hampering their basic useful properties.

सार

विभिन्न अंतिम अनुप्रयोगों के लिए कार्यात्मक वस्त्रों की माँग में तेजी से वृद्धि हुई है। यह बताया गया है कि फंक्शनल टेक्स्टाइल फिनिशिंग एजेंटों का बाजार 6.1% संचयी वार्षिक वृद्धि दर (CAGR) की दर से बढ़ रहा है और यूएस में फंक्शनल टेक्स्टाइल एजेंटों का बाजार 2025 में 162.5 मिलियन अमरीकी डॉलर तक पहुंचने की उम्मीद है। कार्यात्मक वस्त्रों में हाल ही में हुई घातक कोविड 19 महामारी के कारण रोगाणुरोधी वस्त्र बहुत तेज गति से बढ़ रहे हैं, जिसमें पीपीई के रूप में वस्त्रों ने प्रमुख भूमिका निभाई है। दूसरी ओर, कपड़ा कीटाणुओं के वाहक के रूप में कार्य करता है और बहुत तेजी से संक्रमण फैलाता है। वस्त्रों के माध्यम से संक्रमण के प्रसार को रोगाणुरोधी परिष्करण उपचार का उपयोग करके नियंत्रित किया जा सकता है। कपड़ा सभी के लिए प्राथमिक जरूरतों में से एक है। इन वस्त्रों को कार्यात्मक बनाकर ग्रामीण क्षेत्र में सामान्य वस्त्रों द्वारा फैले विभिन्न संक्रमणों के कारण बीमारी दर को कम किया जा सकता है। न केवल रोगाणुरोधी वस्त्र, बल्कि वस्त्रों में निहित अन्य कार्यात्मक गुणों की भी बढ़ती मांग है जो मानव को अतिरिक्त सुरक्षा प्रदान कर सकते हैं जैसे कि यूवी प्रकाश, आग से सुरक्षा। इसलिए, एक साथ कई कार्यात्मकताओं को प्राप्त करने के लिए कपड़े पर उपन्यास परिष्करण एजेंटों और उनकी खोज को खोजने के लिए हमेशा एक शोध प्रवृत्ति रही है। लेकिन फिनिशिंग एजेंटों की तलाश के लिए मांग भी बढ़ी है जो सुरक्षित, किफायती और औद्योगिक रूप से व्यवहार्य होने के साथ-साथ वस्त्रों के एकीकरण के तापमान पर स्थिर होनी चाहिए। इस प्रकार, सुरक्षित कार्यात्मक एजेंट का उपयोग करके एंटीमाइक्रोबियल, यूपीएफ, सुगंध, रंग इत्यादि जैसे विभिन्न गुणों वाले बहुआयामी वस्त्रों का विकास वर्तमान में उच्च प्राथमिकता वाला शोध क्षेत्र है। सबसे पहले अकेले वैनिलिन और धातुओं की उपस्थिति में वैनिलिन को कपास और पीईटी वस्त्रों दोनों पर बहुआयामी परिष्करण एजेंट के रूप में खोजा गया। हालाँकि, वैनिलिन अकेले उपचारित कपड़े को टिकाऊ कार्य गुण प्रदान नहीं करता है और धोने के बाद बाहर निकल जाता है। लेकिन जिन धातु आयनों (Cu, Fe, Zn) का अध्ययन किया गया, उनमें वैनिलीन (वानीलिन-Cu) के साथ कॉपर कॉम्प्लेक्स ने सूती वस्त्रों को 99% रोगाणुरोधी गतिविधि और 50+ UPF जैसी उत्कृष्ट कार्यात्मकता प्रदान की है। पीईटी कपड़े पर वानीलिन-Cu का भी पता लगाया गया है और पीईटी (PET) कपड़े को भी समान कार्यक्षमता प्रदान की है। इसके अलावा, वैनिलिल अल्कोहल को केवल कपास और पीईटी कपड़े पर ही संश्लेषित, विशेषता और खोजा गया है। वैनिलिल अल्कोहल (वीएँ) में वैनिलिन की तुलना में हल्की सुगंध होती है, जिसे कई लोगों के लिए बेहतर विकल्प माना जा सकता है। कपास के मामले में वीएँ उपचारित कपड़े का यूपीएफ (लगभग 35) बढ़ाया है, लेकिन छोटे आकार के कारण यह फिर से कपास और पीईटी वस्त्रों को धोने के बाद बाहर निकल जाता है।

इस समस्या से निपटने के लिए Cu के साथ वैनिलिल अल्कोहल कॉम्प्लेक्स ($Va-Cu$) को इन-सीटू तैयार किया गया है और उच्च तापमान उच्च दबाव (HT-HP) रंगाई मशीन का उपयोग करके कपास और पीईटी वस्त्रों पर एकीकृत किया गया है। उपचारित कपड़े ने बेहतर कार्यात्मक गुणों को काफी हद तक चित्रित किया है, विशेष रूप से, रोगाणुरोधी और यूवी प्रतिरोध गुण उत्कृष्ट श्रेणी में पहुंच गए हैं। उपचारित कपड़े भी अपने कार्यात्मक गुणों को 90 धुलाई तक बनाए रख सकते हैं।

अगले अध्ययन में, उच्च उपज के साथ एक नए दृष्टिकोण के माध्यम से वैनिलिन (एसबी) के शिफ बेस (SB) को संश्लेषित किया गया है। यह परिकल्पना की गई है कि बड़े आकार का शिफ बेस कपड़े के साथ दृढ़ता से संपर्क करेगा और कपास और पीईटी संरचना के अंदर बना रहेगा। एचटी-एचपी उपचार प्रक्रिया का उपयोग करके कपास और पीईटी कपड़े के अंदर एसबी का एकीकरण किया गया है। यह देखा गया है कि उपचारित कपड़े अच्छे कार्यात्मक गुण प्रदान करते हैं, जैसे लगभग 90% की रोगाणुरोधी गतिविधि, अच्छा यूपीएफ और सुंदर रंगाई जो कपड़ा संरचना के भीतर वैनिलिन और वैनिलिल अल्कोहल की तुलना में एसबी के प्रभावी प्रतिधारण की पुष्टि करते हैं। हालांकि, उपचारित कपड़े के गुणों को केवल 3 मशीन धोने तक बनाए रखा गया है। धोने में सुधार करने के लिए स्थायित्व $SB-Cu$ कॉम्प्लेक्स को इन-सीटू संश्लेषित किया गया है और कपास और पीईटी वस्त्रों पर एकीकृत किया गया है जो उपचारित वस्त्रों पर उत्कृष्ट और टिकाऊ (18 मशीन वॉश, यानी 90 होम वॉश) कार्यात्मक गुण प्रदान करते हैं।

अगले अध्ययन में, उच्च उपज के साथ एकल चरण प्रक्रिया का उपयोग करके एक और नया व्युत्पन्न, $RSB-Cu$ शिफ बेस (आरएसबी) को संश्लेषित किया गया है। संश्लेषित आरएसबी (RSB) अणुओं को कपास और पीईटी वस्त्रों पर चित्रित और एकीकृत किया गया है। उपचार दोनों प्रकार के कपड़ों के कार्यात्मक गुणों जैसे रोगाणुरोधी गतिविधि को 95% तक और एंटीऑक्सीडेंट गुणों को 90% से अधिक बढ़ाता है। यह पाया गया है कि उपचारित कपड़े के कार्यात्मक गुण 6 मशीन वॉश तक बने रहते हैं और उसके बाद कार्यात्मक गुण कम हो जाते हैं। कार्यात्मक गुणों को और बढ़ाने के लिए, एचटी-एचपी प्रक्रिया का उपयोग करके वस्त्रों पर आरएसबी-सीयू को संश्लेषित किया गया है। उपचारित कपड़ों ने कार्यात्मक गुणों में उल्लेखनीय सुधार प्रदर्शित किया है जैसे कि उत्कृष्ट UPF के साथ 99% तक बढ़ी हुई रोगाणुरोधी संपत्ति और आकर्षक छाया के साथ 50% एंटीऑक्सीडेंट गतिविधि। गुणों को 90 धुलाई तक भी बनाए रखा गया है। एक और अध्ययन में, वैनिलीन रिएक्टिव फिनिश (वीआरएफ) को संश्लेषित और विशेषता दी गई है। थीसिस के काम के पिछले खंडों में, यह पाया गया है कि सभी वैनिलीन-आधारित सक्रिय तत्व शारीरिक संपर्क के कारण कपड़ा संरचना

म बने रहेंते ह और प्रत्येक धोने के बाद गुण कम हो जाँते ह। इस प्रकार, सूती कपड़े और वैनिलीन के बीच सहसंयोजक बंधन गठन की अवधारणा की गई है और वीआरएफ (VRF) को सफलतापूर्वक संश्लेषित किया गया है। सूती कपड़े पर वीआरएफ का एकीकरण क्षारीय पीएचें म 60 डिग्री सेल्सियस (जो कि अनुकूलित तापमान है) पर किया गया है। इस दृष्टिकोण से, परिष्करण एजेंट कपड़े का एक हिस्सा बन जाता है और लंबे समय तक चलने वाले कार्यात्मक प्रभाव दिखाता है। हालांकि, वैनिलिन का -OH समूह VRF म अवरुद्ध है और इसलिए उपचारित कपड़े की रोगाणुरोधी गतिविधि म कमी पाई गई है। फिर भी, उपचारित कपड़े की यूपीएफ त्रैल्य बढ़ाकर 50 कर दी गई है, जिसे 90 धुलाई के बाद भी लगभग 40 पर रखा जा सकता है। वीआरएफ उपचारित कपड़ें म सफेद रंग और उत्कृष्ट यूपीएफ गुण होने का लाभ है। यह परिसज्जा केवल सूती कपड़े पर खोजी गई है।

थीसिस के बाद के भागें म, बहुक्रियाशील प्रभावकारिता के संदर्भ म सभी फिनिश की तुलना बहुत व्यवस्थित तरीके से की गई है। उपचारित वस्त्रों के भौतिक गुणों का भी अध्ययन किया गया है ताकि परिष्करण उपचार के कारण कपड़े के बुनियादी गुणों म किसी भी तरह की गिरावट को रोका जा सके। हालांकि, यह साबित हो चुका है कि सभी फिनिश सुरक्षित ह और कपड़े के हडल के साथ-साथ अन्य उपयोगी गुणों जैसे जल अवशोषण, तन्य शक्ति और वायु पारगम्यता को नहीं बदलेंते ह। अंत म, यूवी-विज़िबल स्पेक्ट्रोस्कोपी, एसईएम (SEM) और टीईएम (TEM) विश्लेषण और डीपीपीएच परीक्षण का उपयोग करके यूवी प्रतिरोध, रोगाणुरोधी गतिविधि और एंटीऑक्सिडेंट गतिविधि के तंत्र का भी अध्ययन किया गया है। अंत म, यह निष्कर्ष निकाला गया है कि वैनिलिन और अन्य सभी वैनिलिन-आधारित संश्लेषित खत्म सुरक्षित ह, और उनके बुनियादी उपयोगी गुणों म बाधा डाले बिना कपड़ा सबस्ट्रेट्स को बहुत उपयोगी कार्यात्मक गुण प्रदान करेंते ह।

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ABBREVIATION AND SYMBOLS

CAGR	Cumulative annual growth rate
UV-Vis	Ultraviolet-visible
PET	Polyethylene terephthalate
HT-HP	High temperature high pressure
UPF	Ultra-violet protection factor
VA	Vanillyl alcohol
SB	Schiff base of vanillin
RSB	Reduced Schiff base of vanillin
AATCC	American Association of Textile Chemists and Colorists
V-Cu	Vanillin-copper
VA-Cu	Vanillyl alcohol-copper
SB-Cu	Schiff base-copper
RSB-Cu	Reduced schiff base-copper
FDA	Food and Drug Administration
KL	Kraft lignin
CAS no	Chemical abstracts service number
g/ml	Gram per millilitre
TPSA	Topological polar surface area
RFD	Ready for dyeing
GSM	Gram per square meter
NMR	Nuclear magnetic resonance
IR	Infrared radiation
<i>E. coli</i>	Escherichia coli
<i>S. aureus</i>	Staphylococcus aureus
XRD	X-ray diffraction
CC	Control cotton
CP	Control polyester
SEM-EDX	Scanning electron microscopy-energy dispersive X-ray analysis
cm ⁻¹	Centimeter inverse
DPPH	2,2-diphenyl-1-picrylhydrazyl
K/S	Colour strength
Tg	Glass transition temperature
VRF	Vanillin reactive finish

HAT	Hydrogen atom transfer
cfu	colony forming unit
BCR	bacteria colony reduction