

**NANOSURFACE MODIFICATION OF POLYESTER
FABRICS FOR MOISTURE MANAGEMENT**

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NANOSURFACE MODIFICATION OF POLYESTER FABRICS FOR MOISTURE MANAGEMENT

by

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CERTIFICATE

This is to certify that the thesis entitled '**Nanosurface modification of polyester fabrics for moisture management**' being submitted by **Ms. Rashi Agarwal** to the **Indian Institute of Technology Delhi** for the award of degree **Doctor of Philosophy**, is a record of bonafide research work carried out by her. She has worked under our guidance and supervision and fulfilled the requirements for the submission of 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 share of polyester (polyethylene terephthalate, PET) in textiles has been continuously increasing over the last several years due to its high mechanical durability and low cost and has reached ~80%. However, polyester suffers from many problems and most of them arise from its poor hydrophilicity and inability to get functionalized as it lacks sufficient functional groups on its surface. This poor hydrophilicity causes poor absorption, wicking and evaporation of moisture from its surface. Further, the lack of polar groups on the PET surface restricts the formation of a conductive layer which results in the accumulation of static charges on its surface. For the past few years, nanotechnology has emerged as an option to functionalize textiles with improved performance without adversely affecting permeability or hand feel of the fabric. This can also be used to alter the surface properties of polyester for various applications. Nano-SiO₂ and nano-TiO₂ particles have been selected for such modification as they are widely used in cosmetics and food industry. However, the application of these nanoparticles (NPs) on textiles face many challenges such as agglomeration, dispersion, attachment and wash durability.

In this study, SiO₂ and TiO₂ NPs were synthesized using simple sol-gel method. Both synthesized and commercially available SiO₂ and TiO₂ NPs of different sizes and at varying concentrations were mixed with a commercially available nonreactive aliphatic hydrophilic polyester resin and applied on the fabric using a simple pad-dry-cure method to nanomodify the PET substrate. The treated PET fabrics were characterized for changes in the surface morphology and chemical composition both in as-applied state and after several cycles of laundry washes. The treatment effectively and durably enhanced the hydroxyl group concentration on the surface of PET fabric even with the application at a very low

concentration of ~1 wt%, which is very critical for its industrial viability. This finishing methodology opens up the possibility of imparting durable functionalities such as photocatalytic, UV absorption, antistatic, and/or moisture management properties to otherwise inert hydrophobic PET fabric.

These nanomodified PET fabrics were explored for their moisture management properties i.e., wetting, wicking, and evaporation. The influence of type of NPs, their particle size and concentration on the surface morphology and moisture management behavior of PET was investigated. The composite of NPs and resin exhibited radical improvement in the moisture management behavior of PET substrate, which is crucial for its industrial viability for active wear. At an add-on of < 2 wt%, this nano-finishing agent led to remarkable enhancement in wettability, wickability, and evaporation of moisture. Based on the surface morphology and chemical characteristics of the treated fabrics, a mechanism has been proposed to explain the role of NPs in the enhancement of moisture management properties. It has been shown that in the presence of resin, the NPs form a durable network on the surface of PET fibers and play a synergistic role in moisture and heat management of the finished fabric. It was further shown that the presence of large number of surface OH groups with low binding energy with water molecules on NPs changes the overall dynamics of evaporation by improving absorption, wicking properties thereby making the hydrophobic fabric comfortable as active wear. The properties were observed to be significantly improved with titania NPs as compared to silica NPs treated samples. Infrared thermography has been used to demonstrate the potential of such combination to attain enhanced comfort properties.

Further, the influence of morphology of silica NPs on surface nano modification of PET fibers and its effect on moisture management, UV protection, and antistatic properties of resultant fabric was investigated. Two different types of silica NPs, one sol-gel synthesized solid spherical NPs (SiNsol) and the other having mesoporous structure (MS-silica) were durably applied over the PET fabric surface at different concentrations in combination with a hydrophilic resin. Though both NPs showed enhancement in the properties, MS-silica NPs were observed to show significantly higher improvement compared to SiNsol NPs owing to their high surface area, large number of surface OH groups, and better dispersibility at higher concentration (1 wt%).

Finally, a rapid and simple process of synthesis to obtain water dispersible aromatic polyester resin is reported. The resin was prepared using glycolysis of polyethylene terephthalate (PET) with polyhydric alcohol (pentaerythritol, PENTA) in the presence of zinc acetate as a catalyst. The influence of process parameters, such as the molar ratio of PENTA to PET, reaction time, and catalyst concentration on water dispersibility and chemical structure of the synthesized resin was investigated. The characterization of resin confirmed the formation of a complex mixture of oligo-ester-diols with large number of hydroxyl groups as confirmed by FTIR, ^1H and ^{13}C NMR, and MALDI-TOF MS. The obtained resins, when applied as it is or in combination with NPs, imparted enhanced moisture management properties as compared to only resin along with good wash durability to the otherwise inert hydrophobic PET fabric.

सार

पिछले कई वर्षों में वस्त्रों में पॉलिएस्टर (पॉलीइथाइलीन टेरेफ्थैलेट, पीईटी) की हिस्सेदारी इसकी उच्च यांत्रिक स्थायित्व और कम लागत के कारण लगातार बढ़ रही है और बढ़कर ~ 80% तक पहुंच गयी है। हालांकि, पॉलिएस्टर कई समस्याओं से ग्रस्त है और उनमें से अधिकतर उत्पन्न होते हैं इसकी खराब हाइड्रोफिलिसिटी और क्रियाशील होने में असमर्थता क्योंकि इसकी सतह पर पर्याप्त कार्यात्मक समूहों का अभाव है। यह खराब हाइड्रोफिलिसिटी के कारण पॉलिएस्टर की सतह से नमी का खराब अवशोषण, विकिंग और वाष्पीकरण होता है। इसके अलावा, पीईटी सतह पर ध्रुवीय समूहों की कमी के कारण प्रवाहकीय परत का निर्माण नहीं हो पाता जिसके परिणामस्वरूप पीईटी सतह परस्थिर आवेशों का संचय होता है। विगत वर्षों में, नैनोटेक्नोलाजी विकल्प के रूप में उभरी है जो पॉलिएस्टर के नकारात्मक प्रभाव को निष्फल करने में सक्षम है। इसका उपयोग विभिन्न प्रकार के पॉलिएस्टर के सतह गुणों को बदलने के लिए भी किया जा सकता है। नैनो-सिलिका और नैनो-टाइटेनिया कणों को इस तरह के संशोधन के लिए चुना गया है क्योंकि ये व्यापक रूप से सौंदर्य प्रसाधन और खाद्य उद्योग में उपयोग किए जाते हैं। हालांकि, नैनोकणों (एनपी) का वस्त्रो पर उपयोग को कई चुनौतियों का सामना करना पड़ता है जैसे कि ढेर, फैलाव, लगाव और धोने स्थायित्व।

इस अध्ययन में, सिलिका और टाइटेनिया एनपी को सरल सोल-जेल विधि का उपयोग करके संश्लेषित किया गया। दोनों संश्लेषित और व्यावसायिक रूप से उपलब्ध विभिन्न आकारों के सिलिका और टाइटेनिया एनपी और अलग-अलग सांद्रता को व्यावसायिक रूप से उपलब्ध गैर-प्रतिक्रियाशील स्निग्ध हाइड्रोफिलिक पॉलिएस्टर रेसिन के साथ मिलाया गया और साधारण पैड-ड्राई-क्योर विधि का उपयोग करके कपड़े पर लगाया गया ताकि पीईटी सबस्ट्रेट का नैनोमोडिफिकेशन किया जा सके। नैनो संशोधित पीईटी वस्त्र के सतह परिवर्तन और रासायनिक संरचना में आये

परिवर्तन को लक्षित किया गया । इन परिवर्तनों का नैनो संशोधन के तथा कई चक्रों में कपड़ों की धुलाई के उपरान्त विवरण किया गया । यहाँ तक की बहुत कम सांद्रता ~ 1 wt% के आवेदन के साथ भी पीईटी कपड़े की सतह पर प्रभावी ढंग से और टिकाऊ रूप से हाइड्रॉक्सिल समूह की सांद्रता को पीईटी सतह पर बढ़ाया, जो की औद्योगिक व्यवहार्यता के लिए बहुत महत्वपूर्ण है। यह परिष्करण पद्धति की मदद से हाइड्रोफोबिक पीईटी कपड़े को टिकाऊ कार्यात्मकता जैसी की फोटोकैटलिटिक, यूवी अवशोषण एंटीस्टैटिक और नमी प्रबंधन गुण प्रदान कर सकते हैं।

इन नैनोमॉडिफाइड पीईटी कपड़े को उनके नमी प्रबंधन गुणों जैसे की अवशोषण, विकिंग और वाष्पीकरण के लिए जाँच किया गया । एनपी के प्रकार, उनके कण आकार और सांद्रता का प्रभाव पीईटी की सतह आकारिकी और नमी प्रबंधन व्यवहार पर जाँचा गया । एनपी और रेसिन के सम्मिश्रण ने पीईटी सब्सट्रेट के नमी प्रबंधन में आमूल-चूल सुधार प्रदर्शित किया, जोकि सक्रिय वस्त्र की औद्योगिक व्यवहार्यता के लिए महत्वपूर्ण है । <2 wt% के ऐड-ऑन पर, इस नैनो-फिनिशिंग एजेंट ने गीलापन, विकबिलिटी और नमी के वाष्पीकरण में उल्लेखनीय वृद्धि की । सतह आकारिकी और उपचारित कपड़ों की रासायनिक विशेषताओं के आधार पर नमी प्रबंधन गुणों की वृद्धि में एनपी की भूमिका को समझाने के लिए एक तंत्र प्रस्तावित किया गया । यह दिखाया गया है कि रेसिन की उपस्थिति में, एनपी पीईटी फाइबर की सतह पर एक टिकाऊ नेटवर्क बनाते हैं और उनकी नमी और गर्मी प्रबंधन गुणों में सहक्रियात्मक भूमिका निभाते हैं । आगे दिखाया गया एनपी की सतह पर OH समूहों की बड़ी संख्या की उपस्थिति जिनका पानी के साथ कम बाध्यकारी ऊर्जा ने वाष्पीकरण की समग्र गतिशीलता में परिवर्तन करके हाइड्रोफोबिक कपड़े को आरामदायक बना दिया । सिलिका की तुलना में टाइटेनिया एनपी के साथ गुणों में काफी सुधार देखा गया । इन्फ्रारेड थर्मोग्राफी का उपयोग इस संयोजन के क्षमता को प्रदर्शित करने एवं वर्धित आराम प्राप्त करने हेतु किया गया ।

इसके अलावा, सिलिका एनपी के आकारिकी का पीईटी फाइबर के नैनो संशोधन पर प्रभाव का अध्ययन किया गया। इसके उपरान्त इस नैनो संशोधित फाइबर का नमी प्रबंधन, यूवी संरक्षण और एंटीस्टैटिक गुणों की जांच की गई। दो अलग-अलग प्रकार के सिलिका एनपी, एक सोल-जेल संश्लेषित ठोस गोलाकार एनपी (SiNsol) और अन्य मेसोपोरस संरचना (मस-सिलिका) वाले स्थायी रूप से पीईटी कपड़े की सतह पर विभिन्न सांद्रता में हाइड्रोफिलिक रेसिन के साथ संयोजन में लगाए गए। हालांकि दोनों एनपी ने गुणों में वृद्धि दिखाई, सिलिका एनपी की तुलना में एमएस-सिलिका एनपी में उनके उच्च सतह क्षेत्र, बड़ी संख्या में सतह OH समूह, और उच्चतर सांद्रता (1 wt%) पर बेहतर फैलाव के कारण काफी अधिक सुधार दिखा।

अंत में, पानी फैलाने योग्य पॉलिएस्टर रेसिन प्राप्त करने के लिए संश्लेषण की एक तेज़ और सरल प्रक्रिया बताई गयी है। पॉलीहाइड्रिक अल्कोहल (पेंटाएरिथ्रिटोल, पेंटा) के उपयोग से पॉलीइथाइलीन टैरेफ्थैलेट (पीईटी) का ग्लाइकोलाइसिस उत्प्रेरक के रूप में जिंक एसीटेट की उपस्थिति में करके रेसिन तैयार किया गया। प्रक्रिया मापदंडों का प्रभाव, जैसे कि पेंटा से पीईटी का दाढ़ अनुपात, प्रतिक्रिया समय, और उत्प्रेरक सांद्रता संश्लेषित रेसिन के पानी के फैलाव और रासायनिक संरचना पर जांच की गई। रेसिन के विवरण ने जटिल ओलिगो-एस्टर-डायोल का मिश्रण के गठन बड़ी संख्या में हाइड्रॉक्सिल समूहों के साथ FTIR, ^1H और ^{13}C एनएमआर और मालदी-टीओएफ एमएस द्वारा की गई। निष्क्रिय हाइड्रोफोबिक पीईटी कपड़े पर संश्लेषित रेसिन एनपी के साथ अकेले रेसिन की तुलना में ज्यादा नमी प्रबंधन गुण के साथ अच्छे धोने के स्थायित्व प्रदान करता है।

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LIST OF SYMBOLS AND ABBREVIATIONS

Symbol/Abbreviation	Expanded Form/Term
Γ	interfacial tension
Θ	contact angle
AA	acrylic acid
AA-g-PET	acrylic acid grafted PET
Aam	acrylamide
AFM	atomic force microscopy
Ag NPs	silver nanoparticles
BHET	bihydroxy ethylene terephthalate
CTAB	cetyl trimethyl ammonium bromide
DAT	drop absorbency time
DETA	diethylenetriamine
DMT	dimethyl terephthalate
EB	electron beam
EG	ethylene glycol
GCA	glycerin contact angle
GTMAC	glycidyl tri-methyl ammonium chloride
MCC	Microcrystalline cellulose
MS-Silica	mesoporous silica
NPs	nanoparticles
P	developed capillary tube pressure
P25 TiO ₂ NPs	P25 titania nanoparticles

PEG	polyethylene glycol
PENTA	pentaerythritol
PET	poly(ethylene terephthalate)
PP	polypropylene
PVA	poly(vinyl alcohol)
QAC	Quaternary ammonium salts
R_a	arithmetic mean of the variance in surface height
R_c	capillary tube radius
R_q	root mean square average of surface roughness
SiNsol	silica nano sol
SiO ₂ NPs	fumed silica nanoparticles
TA	terephthalic acid
TEOS	tetraethyl orthosilicate
TETTA	triethylenetetramine
T_g	glass transition temperature
T_m	melting temperature
TiNsol	titania nano sol
TTEPA	tetraethylenepentamine
TTIP	titanium isopropoxide
UV	Ultraviolet
UPF	UV protection factor
WCA	water contact angle
WVTR	water vapor transmission rate
ZnO	zinc oxide