JANUS PARTICLES FOR TEXTILE APPLICATIONS

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by

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DEDICATED TO MY LATE FATHER

CERTIFICATE

This is to certify that the thesis entitled **'Janus particles for textile applications'** being submitted by **Ms. Kamlesh Panwar** 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

Nanoparticles (NPs), due to their higher surface area to volume ratio and unique features, have found tremendous importance for various applications. On textiles, silver nanoparticles (AgNPs) and titania (TiO_2) NPs have been widely explored for imparting antimicrobial and self cleaning activities, respectively. However, these isotropic NPs face several challenges such as dispersion, agglomeration, attachment, and durability etc. which ultimately restrict their application on textiles. AgNPs and TiO_2 NPs in the form of Janus particles may be used to overcome some of the above–mentioned challenges. These Janus particles may possess NPs on one half and functionality suitable for attachment with textile substrate on the other half.

Silica (SiO_2) particles have been one of the mostly used base materials for the synthesis of Janus particles due to their high thermal stability, mechanical strength and stability in solvents. In the first part of the study, SiO_2 NPs with size ranging from ~100 to 800 nm were synthesized by Stöber method. SiO_2 particles of diameter ~550 nm were used for further studies. In order to synthesize Janus particles, the SiO_2 particles were functionalized with amine, thiol and epoxy groups.

Silver–silica (Ag–SiO_2) Janus particles were synthesized by depositing AgNPs on one half of the SiO_2 particles and retaining varying functionalities i.e. amine, thiol and epoxy on the other half surface of these particles using Pickering emulsion method. These Janus particles were explored for their antimicrobial activity. Due to their easy dispersibility, Janus particles with AgNPs of diameter ~3 nm showed much lower Minimum Bactericidal Concentration (MBC) as compared to conventional redispersed isotropic AgNPs powder having AgNPs of almost same diameter. The functionalized Ag–SiO_2

Janus particles were attached on cotton fabric by exhaustion followed by curing. Due to the presence of various functionalities on one–half of Ag–SiO_2 Janus particles, these could be attached to cellulose substrates for imparting durable antimicrobial property.

The Ag–SiO_2 Janus particles with ~3 nm sized AgNPs were also evaluated for surface enhanced Raman scattering (SERS) activity. Also, the particles were attached on a cellulosic film to fabricate a macroscopic SERS (M–SERS) substrate. Raman spectrum of Rhodamine B (RhB) showed an enhancement factor of $\sim 5 \times 10^4$ for dye tagged on both Janus particles and M–SERS. In comparison, AgNPs with almost the same diameter, even after aggregation, did not show enhancement of Raman signals. The enhancement was attributed to in–situ reduction of silver precursor on the SiO_2 surface leading to deposition of AgNPs and generation of a large number of fixed hot–spots in a controlled manner.

Titania–silica (TiO_2–SiO_2) Janus particles with high photocatalytic activity were synthesized using Pickering emulsion method. The photocatalytic activity of Janus particles, calcined at two different temperatures, was compared with that of calcined SiO_2–TiO_2 core–shell particles and commercially available P25 TiO_2 NPs. Janus particles calcined at 450 °C (TiJanus@450) showed significantly higher adsorption (by almost 2–14×) and rate of degradation (by almost 6.6–211×) of a photostable Solophenyl green dye compared to other TiO_2 structures. The higher activity of TiJanus@450 was attributed to its unique electronic structure which showed significantly higher average carrier lifetime of 1.98 ns compared to 0.18 ns of other structures. Various TiO_2 nanostructures including TiJanus@450, commercially available P25 TiO_2 NPs and synthesized TiO_2 nanosol were applied on cotton fabrics and evaluated for their photocatalytic activity. TiJanus@450 treated cotton, with TiO_2 content almost half of that present in P25 TiO_2 treated cotton, showed significantly higher photocatalytic activity with good wash durability and better retention of mechanical properties. These properties could be attributed to the characteristic morphology of Janus particles and the presence of SiO_2–TiO_2 interface that prevented direct contact of TiO_2 with textile surface.

Further, the thermal regulation property of TiJanus@450 was investigated after applying Janus particles on a cotton substrate. The coated fabric was evaluated for both near infrared (NIR) reflection and heat build–up behavior. TiJanus@450 treated cotton fabric, showed significantly higher NIR reflection (i.e. 79% at 800–1100 nm) and better thermal regulation than TiO_2 treated cotton and control cotton fabrics. The enhanced properties were attributed to the unique morphology of Janus particles, which suggests that these structures can be used as an effective substitute for the widely studied multilayered structures of high and low refractive indices materials.

Finally, a facile method for the synthesis of amphiphilic SiO_2 Janus particles by low temperature atmospheric pressure plasma is reported. The amphiphilic SiO_2 Janus particles were prepared by masking method, where half of the surface of SiO_2 particles was embedded inside a poly(styrene) (PS) film deposited on a glass slide. The masked SiO_2 particles were treated with He/Floron low temperature atmospheric pressure plasma to modify the exposed surface. The amphiphilic nature of Janus particles was confirmed using fluorescent microscopy by tagging the hydrophilic part of the Janus particles with a fluorescent dye. These amphiphilic Janus particles may find applications as surfactants and surface modifying agents.

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List of Symbols and Abbreviations

Symbol/Abbreviation	Expanded Form/Term
α	absorption coefficient
θ	Bragg angle
C_{SERS}	concentration of Rhodamine B in sample
C_{bulk}	concentration of Rhodamine B powder
E	enhancement factor
ν	frequency
β	full width at half maxima
I_A	intensity of anatase (101) peak
I_{SERS}	intensity of peak in sample
I_{bulk}	intensity of peak of Rhodamine B powder
I_R	intensity of rutile (110) peak
h	Planck’s constant
K	Scherrer constant
A	UV absorbance
λ	wavelength
X_A	weight fraction of anatase
APTMS	(3–aminopropyl) trimethoxysilane
ATRP	atom transfer radical polymerization
DI	double deionized
DDAB	didodecyldimethylammonium bromide
FITC	fluorescein isothiocyanate

GPS	(3–glycidyloxypropyl) trimethoxysilane
AuNPs	gold nanoparticles
HEC	hydroxyethyl cellulose
MPTMS	(3–mercaptopropyl) trimethoxysilane
MBC	minimum bactericidal concentration
NPs	nanoparticles
NIR	near infrared
PAM	poly(acrylamide)
P(AAm–co–AA)	poly(acrylamide–co–acrylic acid)
PDMAEMA	poly[2–(dimethylamino) ethyl methacrylate]
PDVB	poly(divinylbenzene)
PEO	poly(ethylene oxide)
PMMA	poly(methyl methacrylate)
PNIPAM	poly(N–isopropylacrylamide)
PS	poly(styrene)
PS–b–P4VP	poly(styrene)–b–poly(4–vinylpyridine)
P(S–BIEM)	poly(styrene–2–(2–bromoisobutyryl oxy) ethyl methacrylate)
P4VP	poly(4–vinylpyridine)
PVP	poly(vinylpyrrolidone)
RhB	Rhodamine B
SiO_2	silica
AgNPs	silver nanoparticles
Ag–SiO_2	silver–silica

SERS	surface enhanced Raman scattering
TBOT	tetrabutyl orthotitanate
TEOS	tetraethyl orthosilicate
TiO_2	titania
TiO_2–SiO_2	titania–silica
TTIP	titanium isopropoxide
UV	ultraviolet