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Hydrophobicity of Cotton Fabric Treated With Silica Nanoparticles

عدم قابلية الامتصاص لقماش قطني معالج بجسيمات السيليكا النانوية

Thesis Submitted as Partial Fulfillment for a degree Master of Fiber and polymer Engineering

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آيات من الذكر الحكيم

(هَذَا خَلْقُ اللَّهِ فَأَرُونِي مَاذَا خَلَقَ الَّذِينَ مِن دُونِهِ بَلِ الظَّالِمُونَ فِي ضَلَالٍ مُّبِين)

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(وَآتَاكُم مِّن كُلِّ مَا سَأَلْتُمُوهُ وَإِن تَعُدُّواْ نِعْمَتَ اللّهِ لاَ تُحْصُوهَا)

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C E R T I F I C A T E

This is to certify that the thesis entitled hydrophobicity of cotton fabric treated with silica nanoparticles submitted by

Rahma Aissa Hassan Adam

In partial fulfillment of the requirements for the award of the degree of Master of Fiber and polymer *Science* engineering with Sudan University of Science and Technology. To the best of my knowledge, the matter embodied in thesis has not been submitted to elsewhere for the award of any degree.

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LIST OF ACRONYMS & ABBREVIATIONS

WR	Water Repellent
CVA	Chemical vapor deposition
TEOS	Tetra Eth Oxy Silane
CA	Contact Angles
WCA	Water contact Angle
EPS	Environmental Protection Agency
PFOA	Perfluorooctanoic acid
PFOS	PerFluoro Octanoic Sulfonate
PFHxA	PerFluoro Hexanoic Acid
DTMS	Dodecyl Tri Methoxy Silane
PVA	Poly Vinyl Alcohol
LbL	Layer-by-Layer
EtOH	Ethanol
PEG	Poly Ethylene Glycol
FTIR	Fourier Transform Infrared

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LbL	Layer-by-Layer
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FTIR	Fourier Transform Infrared

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A B S T R A C T

To obtain the hydrophobicity cotton fabrics, cotton fabrics were treated with silica nanoparticles and/or accost effective Water Repellent agent (WR agent).Two different silica nanoparticles were synthesized via a sol–gel process and their shapes, sizes, and compositions were characterized. It was found that silica particles are spherical and have diameters of 150 and 300nm.

For the cotton fabrics treated with the WR agent alone the water contact angles on the fabric surface remained lower than 20°approximately at the WR agent concentration of 0.3wt% or less.

Silica nanoparticle treatment itself did not change the hydrophilic surface of cotton fabric indicating that water drops were absorbed into fabrics due to the hydroxyl groups on both cotton and silica nanoparticle surfaces.

However for the cotton fabrics treated with both silica nanoparticles and the WR agent a contact angle above 105°can be obtained even at the very low WR agent concentration of 0.1wt%.

Therefore,hydrophobic cotton fabrics could be obtained the combined treatment of silica nanoparticle and WR agent, which is cost effective com-pared with fluorinate silane treatment.

Keywords — Hydrophobicity cotton, silica nanoparticles, water Repellent (WR), Contact Angle (CA).

مسخلص البحث

للحصول على الأقمشة القطنية المقاومة للماء ، تمت معالجة هذه الأقمشة القطنية بجسيمات السيليكا النانوية و / أو عامل طارد للماء بتكلفة فعالة.

تم تصنيع اثنين من جسيمات السيليكا النانوية المختلفة عبر عملية سول-جيل وتم تحديد أشكالها وأحجامها وتركيبها. وجد أن جزيئات السيليكا كروية ويبلغ قطرها ١٥٠ و ٣٠٠ نانومتر.

وحده ، ظلت زوايا التلامس بالماء على سطح WR بالنسبة للأقمشة القطنية المعالجة بعامل البالغ ٠.٣٪ بالوزن أو أقل WR القماش أقل من ٢٠ درجة تقريبًا عند تركيز عامل.

لم تتغير معالجة جسيمات السيليكا النانوية نفسها سطح الماء المحبب للنسيج القطني مما يشير إلى أن قطرات الماء قد تم امتصاصها في الأقمشة بسبب مجموعات الهيدروكسيل على كل من أسطح القطن وجسيمات السيليكا النانوية.

ومع ذلك ، بالنسبة للأقمشة القطنية المعالجة بكل من جسيمات السيليكا النانوية وعامل طارد الماء ، يمكن الحصول على زاوية تلامس أعلى من ١٠٥ درجة حتى عند تركيز عامل طارد الماء المنخفض جدًا بنسبة ٠.١٪ بالوزن.

لذلك ، يمكن الحصول على الأقمشة القطنية المقاومة للماء المعالجة المشتركة لجسيمات السيليكا النانوية وعامل طارد الماء، وهو فعال من حيث التكلفة مقارنة بمعالجة السيلان بالفلور.

الكلمات المفتاحية:-

قطن مقاوم للماء ، جزيئات السيليكا النانوية ، طارد الماء(WR) ، زاوية التلامس(CA)

CHAPTER 1

INTRODUCTION

Textiles are intensively used materials in daily life. However, direct outdoor use of textiles including the synthetic ones such as nylon, polyester, acrylic etc. for weather protection and water proofing require surface treatment or multilayer approaches .Textiles made from natural fibers such as cotton, wool, silk etc. are particularly unsuitable for weathering owing to their inherent hydrophobicity and structural instability upon contact with water. Nevertheless, recently increased environmental awareness as well as potential large scale applicability of some techniques towards water proofing natural fibers have increased exploitation of these inexpensive natural fibers for applications with requirements of water and oil repellency (Zahid et al., 2018).

Modifying surfaces by changing their roughness and chemistry is considered to be the only way to achieve water repellent natural fibers. This is still a challenging task and requires an efficient combination of low surface energy chemistry with hierarchical micro/nano-scaled roughness. The most common approaches reported in the literature involve utilization of silicone chemistry with nanoparticle (i.e. SiO_2) immobilization on fabric surfaces Depending on the type of incorporated nanoparticles, multifunctional fabrics can be realized by one-step process.

In order to achieve water repellent textiles, various fabrication techniques such as solution immersion and sol-gel methods. The treatment was made in such a way that the open porous structured was preserved allowing breathability. The ease of application technique, industrial scale availability of the low-cost polymers and the non-toxic ingredients would allow this fabric treatment to be implemented in large scale textile treatment facilities(Deeksha et al., 2020).

Silica nanoparticles is being used in developing countries to help treat disease and prevent health issues, and also being applied to or developed for application to a variety of industrial processes (Vishwakarma et al., 2018).

Many fundamental researches on hydrophobic surfaces are conducted on such rigid solid substrates as mostly used silicon wafers, glass slides, and metal surfaces. These substrates might limit the practical application as well as the large-scale production of hydrophobic surfaces. In recent years, hydrophobic surfaces on fabric have been obtained by a number of different approaches (Srivastava, 2020, Enieb and Diab, 2017, Wang et al., 2021).In this work; we adopted the traditional fabric finishing process to prepare hydrophobicity surfaces of cotton fabric traded of silica nanoparticles.

1.1 Problem statement

- Cotton fabric is an ideal place for settling and growing pathogenic bacteria because of its porous and hydrophilic structure.
- Hydrophilic is also of importance especially in some specific applications like medical usage , human tissue, antifouling nanoparticles of SiO_2 have been developed for super hydrophobic surface on the cotton fabric .

1.2 Objective

The main objectives of this study were to obtain the hydrophobic water-repellent cotton fabrics, Prepare new prototype from cotton fabrics with silica nanoparticles and study the influence of the cotton fabrics Treatment with silica nanoparticles on the properties.

1.3 Research Hypothesis

- The first hypothesis is limited by the needs for materials (Pure cotton fabric, SiO_2 nanoparticles and PVA/PEG).
- The second hypothesis stable process parameters to be sure that all the structures are produced with the same.
- The third hypothesis requires a surface treatment in order to improve the quality of cotton fabric.

- The fourth hypothesis requires a mechanical and chemical test in order to determine properties of cotton fabric such as tensile strength, contact angle, FTIR.
- The fifth hypothesis requires more experimentation in the wet of a hydrophobicity cotton fabric.

CHAPTER 2

LITERATU RESURVEY

In recent years, super hydrophobic surfaces have attracted great attention for their wide applications in water-repellency, self-cleaning, anti-sticking and anti-fouling Surface wettability is governed by both the chemical composition and the geometric structure. In nature, the unusual super hydrophobicity of lotus leaves, with static water contact angles larger than 180◦and sliding angles less than 10˚, is known to originate from the combination of micro- and nanoscale hierarchical structures and low surface energy materials on the surface For fabric surfaces, they have the natural micrometer scale roughness coming from the fibers themselves and the woven structure. The surface morphology of the silica nanoparticle assembled fibers can be tailored by the assembly cycles, which makes it available to study the effect of the surface morphology on the static contact angle and the angle hysteresis (Sarshar et al., 2013, Zhao et al., 2010, Grundke et al., 2015, Zhang et al., 2011, Gou and Guo, 2019).

The presence of the hydrophobic nanoparticles, however, will prevent water from penetrating hills (Barthlott et al., 2016).To simulate or produce such super hydrophobic surface on substrates, among different methods (such as chemical vapor deposition phase inversion electro spinning, electro wetting lithography and etching) the sol-gel method seems more conventional to be used on textile materials, due to easy processing and

acceptable treatment conditions (e.g., low temperature). In this method, hydrolysis and condensation reactions of the precursor material are carried out to form a nanocolloidal solution, and a network of nanoparticles will be formed on the substrate through the gradual evaporation of the solvent. The precursors are often based on metal organic compounds such as acetylacetonate, or metal alkoxides like tetraethoxysilane Si $(OC_2H_5)_4$ (TEOS), titanium isopropoxide Ti $(OC_3H_7)_4$, and Al $(OC_4H_9)_3$ (Lin et al., 2018, Ahlers et al., 2019). According to its natural properties cotton fabric is among the very popular textiles. Producing hydrophobic surface on cotton fabric will guarantee its dryness and cleanness which is considered as desired features, in particular on its outside facet .Furthermore, cotton fabric is an ideal place for settling and growing pathogenic bacteria because of its porous and hydrophilic structure. So, hydrophilic is also of importance, especially in some specific applications like medical usage. Like many other particles, the desired properties of copper may be improved by reducing its size to nanoscale. Hence, these nanoparticles can be developed and applied in various new fields, such as water purification, medical science, human tissue, antifouling (Innovativi). Few researches have been focused on developing two abovementioned properties on cellulosic substrates like cotton fabric, simultaneously.

On the other hand, nanoparticles of copper and core shell SiO_2/Cu have been less developed for textile finishing. The current aimed to fabricate

hydrophobic surface on the cotton fabric, by introducing Cu nanoparticles into the silica sols. It was expected that due to their chemical activities, such nanoparticles would change the morphology and arrangement of silica nanostructure, and in addition activity on cotton fabrics (Berendjchi et al., 2011).Textiles are intensively used materials in daily life. However, direct outdoor use of textiles including the synthetic ones such as nylon, polyester, acrylic etc. for weather protection and water proofing require surface treatment or multilayer approaches (Zahid et al., 2018). Textiles made from natural fibers such as cotton, wool, silk etc. are particularly unsuitable for weathering owing to their inherent hydrophobicity and structural instability upon contact with water. Nevertheless, recently increased environmental awareness as well as potential large scale applicability of some techniques towards water proofing natural fibers have increased exploitation of these inexpensive natural fibers for applications with requirements of water and oil repellency (Karim et al., 2020, Tausif et al., 2018). Modifying fiber surfaces by changing their roughness and chemistry is considered to be the only way to achieve water repellent natural fibers. This is still a challenging task and requires an efficient combination of low surface energy chemistry with hierarchical micro/nanoscaled roughness. The most common approaches reported in the literature involve utilization of fluorine or silicone chemistry with nanoparticle (i.e. SiO_2, ZnO, and TiO_2) immobilization on fiber surfaces.

Depend on the type of incorporated nanoparticles, multifunctional fabrics can be realized by one-step process. In order to achieve water repellent textiles, various fabrication techniques such as solution immersion, plasma modification, layer by layer assembly, and chemical vapor deposition and sol-gel methods have been implemented. Some of these non-wet table textiles have been successfully demonstrated in applications like filtration, oil-water separation and pattern able wetting (Mattsson, 2013).

Fluorinated organic polymers were comprehensively studied in the past for cotton textiles because of their combined water and oil repellency properties. Fluor polymers, particularly with ≥ C-8 fluorinated side chains exhibit very low surface free energy (≤ 18 dynes/cm). Although environmental protection agency (EPA) has restricted use of fluoropolymers/fluorotelomers having fluorinated side chains with C-8 or longer, many reports are still found in the literature utilizing flu chemicals with C-8 chemistry. For instance, Tang et al. fabricated oleo phobic/hydrophobic cotton woven fabric using hyper branched polyester/poly-urethane with C-13 fluorinated side chains. As a result of their treatment, contact angles (CA) of 146°, 122° and 102° for water, hexadecane and decane were demonstrated, respectivel (Stratakis, 2012). On the other hand, Yu et al. treated cotton woven fabrics to achieve hydrophobic textiles by per fluorinated silane coupling agent having C-8 fluorinated carbon atoms. A static water contact angle (WCA) of 133° was

achieved without surface roughness modification. The WCA was increased to 145° by incorporating silica nanoparticles, satisfying the Cassie and Baxter's non-wetting model for rough hydrophobic surfaces (Xiang and Liu, 2021). Reported hydrophobic woven cotton fabrics with WCA of 170° through multistep treatment. Their process included pre-fictionalizations of fiber surfaces with epoxy, subsequent solution dipping in nanoparticle suspension for roughness and eventually coating with H_1, H_1, H_2, H_2-perfluorodecyltrichlorosilane and satiric acid to render them super hydrophobic. Despite their excellent water and oil repellency, fluoropolymers or heavy fluorosilanes with C-8 chemistry or higher cause various environmental concerns (Zahid et al., 2017). They decompose into perfluorooctanoic acid (PFOA) or perfluorooctanoic sulfonate (PFOS) which are persistent and bio-accumulate and likely to be carcinogenic as indicated by Environmental Protection Agency (EPA). Hence, they were banned (U.S. EPA 2010/15 PFOA Stewardship Program) and new generation fluoropolymers or silanes with C-6 or lower fluorinated side chains that can degrade into environmentally safe perfluorohexanoic acid (PFHxA) with rapid bio-elimination rate were developed (Zahid et al., 2017, De Preux Gallone and Sassi, 2018). Although in terms of water and oil repellency C-6 fluorinated side chains perform poorer than C-8 counterparts, proper application, chemical modification and thermal treatment of coatings with C-6 fluorinated side chains have also been

successfully used to create water and oil/solvent repellent coatings as well as fibrous non-woven mats In the meantime; silicone chemistry based fabric treatments have become more popular due to abovementioned concerns and the fact that silicone polymers are more flexible and softer than per fluorinated acrylic polymers. For instance, long chain non fluorinated alkyls lanes generally exhibit good hydrophobicity. fabricated water repellent cotton fabrics using dodecyltrimethoxysilane (DTMS) with SiO_2 nanoparticle and ZnO nanorod array inclusions where WCAs of 153° and 159° were obtained, respectively (Vitale et al., 2015, Oh, 2011). rendered different textile substrates (polyester, silk, polyacrylonitrile, wool and cotton) super hydrophobic by chemical vapor deposition of polymethylsilsesquioxane (PMSQ) monofilament's and achieved 25° roll off angle for cotton textiles Gao et al. also prepared highly hydrophobic treatments for cotton and PET woven fabrics by using silica nanoparticles (30 to 71 nm) and hexadecyltrimethoxysilane (HDTMS) (Zahid et al., 2017). A super hydrophobic cotton fabric with WCA 155° was produced once the average silica nanoparticle size was approximately 50 nm.

A similar treatment using HDTMS and nanoparticles was also applied to PET achieving a WCA of 143°. They argued that the reduction in hydrophobicity on PET was due to the smoother surface of PET fibers compared to cotton over which the hydrophobic alkylsilane was applied (Zahid et al., 2017, Zahid et al., 2019). In addition, some long chain alkyl

polymer treatments were also implemented to avoid fluorinated coatings. For instance, recently. Adopted a sustainable approach to fabricate water repellent cotton fabrics by grafting long alkyl chains fatty acids onto the cotton fibers. Water contact angles close to 140° were reported. Although silicone chemistry is preferred over fluorine chemistry for natural textile finishes, it is challenging to produce silicone based non-wet table textile finishes with self-cleaning ability (small water droplets roll-off with slight tilting), therefore, we propose that non-toxic C-6 chemistry can be used in tandem and in much less quantities together with the silicone treatments towards significantly lowering droplet shedding (roll-off) angles and increasing hydrophobic durability (Zahid et al., 2017, Wang et al., 2018). Hence, in this study, we demonstrate that the pretreatment of cotton fibers with a dilute C-65 fluoropolymer solution considerably enhances the hydrophobic performance of the main silicone treatment. Furthermore, incorporation of SiO_2 nanoparticles in the C-6 polymer solution helps decrease water rolls off angles by creating the necessary texture. Encapsulation of this texture (nanocomposite pretreatment) with the silicone polymer ensures that under both wear abrasion and washing with ultrasonic agitation, the nanoparticles are not removed from the fiber surfaces (Deng, 2018). In recent years, super hydrophobic surfaces have attracted great attention for their wide applications in water-repellency, self-cleaning, anti-sticking and anti-fouling Surface wettability is governed by

both the chemical composition and the geometric structure. In nature, the unusual super hydrophobicity of lotus leaves, with static water contact angles larger than 150˚and sliding angles less than 10˚, is known to originate from the combination of micro- and nanoscale hierarchical structures and low surface energy materials on the surface For fabric surfaces, they have the natural micrometer-scale roughness coming from the fibers themselves and the woven structure. Inspired by lotus leaves, researchers have aimed to generate secondary nanoscale structures by incorporating carbon nanotubes (Moradi, 2014).

Gold particles, silica nanoparticles, ZnO nanorods, or copper crystallites onto the micrometer-scale fibers to fabricate hydrophobic fabrics. Among them, however, contact angle hysteresis (defined as the difference between the advancing and the receding contact angles) was rarely mentioned, and some works have considered super hydrophobic fabrics purely on the basis of the criterion of static contact angle larger than 150◦, which is not sufficient to guarantee a low sliding angle for self-cleaning behavior. Electrostatic layer-by-layer (LbL) assembly is a versatile technique based on alternative adsorption of oppositely charged polyelectrolyte's, inorganic nanoparticles, macromolecules or even supra molecular systems on charged substrates to build up multi-layered composite films in a controlled manner Since the LbL assembly technique is independent of the size and topography of substrates, and uniform multilayer can be

formed on substrates with different spatial structures, charged fibers can be used. As substrates to conduct the electrostatic assembly Compared with previously reported methods like dip coating and padding used to generate nanostructures on fibers, the LbL assembly technique has the advantage of being able to tailor the surface morphology of the nanostructures by controlling the assembly cycles, and also the advantage of good durability because of the electrostatic interactions between the negatively charged silica nanoparticles and thepolycations. that for hydrophobic fabric, its durability against washing remains agreat challenge; Daoud et al reported the static contact angle of cotton fabric decreased from 141◦to 105◦after 10 wash cycles, also reported the dramatic decrease in the static contact angle of super hydrophobic cotton fabric from 155˚to about 105˚after10 wash cycles ,were port on the fabrication of super hydrophobic cotton fabrics by electrostatic assembly of silica nanoparticles and polycations on cotton fibers and sub-sequent treatment with fluoroalkyl silane.

The two main problems that were found by practical application:

- Unorganized treat for cotton fabric.
- hydrophilic competition

Cotton fabric is an ideal place for settling and growing pathogenic bacteria because of its porous and hydrophilic structure.

Hydrophilic is also ofimportance especially in some specific applications like medical usage, human tissue, antifouling .nanoparticles of SiO_2 having been developed for super hydrophobic surface on the cotton fabric.

2.1 Hydrophilic substances

2.1.1 What is Hydrophilic?

Are polar in nature Like dissolves like theory governs the fact that hydrophilic substances tend to readily dissolve in water or polar solvents while hydrophobic substances are poorly soluble in water or polar solvents.We all have seen the example of hydrophilic substances in our daily lives. Each one of us has seen that sometimes water spread out evenly on a surface while in certain cases it forms small droplets. It's because certain surfaces are water-loving or hydrophilic and hence water spreads out while in the case of poorly hydrophilic substances (or hydrophobic substances) it forms tiny droplets as these surfaces repel water (Abbott and Hansen, 2008).

2.1.2 Chemistry behind Hydrophilicity

Hydrophilic molecules or Hydrophilic moieties are basically polar compounds that have ionic groups. The polar nature of these hydrophilic molecules enables them to readily absorb water or polar solvent and

eventually getting dissolved in polar solvents like water. Being a polar protic solvent, water is capable of forming a hydrogen bond (-H—-OH-). Hydrophilic molecules are polar in nature and easily form a hydrogen bond with water thereby getting dissolved in water. Notably, these interactions between the hydrophilic molecule and water are thermodynamically favored. In general, hydrophilic substances can easily form hydrogen bonds with polar solvents like water, alcohol (Bourlinos et al., 2003).The hydrophilicity of any surface varies as per the functional group and ability for hydrogen bonding: non-polar < polar, no hydrogen bonding < polar, hydrogen bonding < hydroxylic, ionic. Hydrophilicity is significantly influenced by the number of sites and the structure and density of the interphase area (Kachwal et al., 2020).

2.1.3 Measurement of Hydrophobicity

Contact angle measurement is a major parameter to quantify the hydrophobicity of a substance, which is further indicative of wettability. Hydrophilic substances possess good wettability.

Wettability is the ability of the liquid to remain in contact with the solid surface. The degree of wettability is measured using a contact angle (Lu et al., 2016,Deng, 2020 #91).

2.1.4 Applications of Hydrophilic Substances

Hydrophilic polymers and molecules are widely utilized in the field of physics, chemistry, engineering, biomedical, drug delivery, food,

pharmaceuticals, paint, textiles, paper, constructions, adhesives, coatings, water treatment, dispersing and suspending agents, stabilizers, thickeners, gellants, flocculants and coagulants, film-formers, humectants, binders and lubricants, personal care, building products, detergents, oil field products, and mineral processing, etc. Hydrophilic polymers exhibit good water vapor permeability due to ionic groups. Clothing or apparel that is required to be breathable is made up of hydrophilic fibers. surface of medical devices to reduce bacterial adhesion onto the surface of the medical device (Thombare et al., 2016).

2.2 Cotton

Is a soft, fluffy staple fiber that grows in a boll, or protective case, around the seeds of the cotton plants of the genus Gossypium in the mallow family Malvaceae. The fiber is almost pure cellulose. Under natural conditions, the cotton bolls will increase the dispersal of the seeds. Manually decontaminating cotton before processing at an Indian spinning mill (2010) the plant is a shrub native to tropical and subtropical regions around the world, including the Americas, Africa, Egypt and India. The greatest diversity of wild cotton species is found in Mexico, followed by Australia and Africa. Cotton was independently domesticated in the Old and New Worlds. The fiber is most often spun into yarn or thread and used to make a soft, breathable textile. The use of cotton for fabric is known to date to prehistoric times; fragments of cotton fabric

dated to the fifth millennium BC have been found in the Indus Valley Civilization, as well as fabric remnants dated back to 6000 BC in Peru. Although cultivated since antiquity, it was the invention of the cotton gin that lowered the cost of production that led to its widespread use, and it is the most widely used natural fiber cloth in clothing today (Belt and Cotton).

Current estimates for world production are about 25 million tonnes or 110 million bales annually, accounting for 2.5% of the world's arable land. India is the world's largest producer of cotton. The United States has been the largest exporter for many years. In the United States, cotton is usually measured in bales, which measure approximately 0.48 cubic meters (17 cubic feet) and weigh 226.8 kilograms (500 pounds) (Broudy, 1993).

Figure 2-4 Cotton ready for harvest

2.2.1 What Is Cotton Fabric?

Cotton fabric is one of the most commonly used types of fabrics in the world. This textile is chemically organic, which means that it does not contain any synthetic compounds. Cotton fabric is derived from the fibers surrounding the seeds of cotton plants, which emerge in a round, fluffy formation once the seeds are mature; Cotton fiber is the purest source of cellulose and the most significant naturalfiber. The economic significance of cotton in the global market is evident byits majority share (over 50%) among fibers for apparel and textile goods. Both the market value and the quality of cotton products are directly related to fiber quality Competition with other fibers is affected by innovations and commercialization of other fibers including micro denier (polyesters and nylons), elastomeric (spandex), and lyocell fibers, among others. Fundamental understanding of the fibers (structural formation during development, chemistry, physics), significant improvement in fiber quality as well as in process innovation and product differentiation are critical to uphold the interfile competitiveness of cotton fibers and the share of cotton fibers in the global apparel and other textile markets.

Part I of this book focuses on the chemical and physical properties of cotton fibers. The most essential cotton fiber qualities related to mechanical processing, i.e. traditionally yarn spinning, weaving, and knitting, are length, strength, fineness and their distributions. The

ranking importance of these fiber qualities varies with the type of yarn spinning method, such as ring ,rotor, and air-jet. These fiber qualities also determine the yarn strength, yarn regularity, handle and luster of fabrics. For chemical processing such as scouring, dyeing and finishing, fiber structure related to maturity, or the level of development, plays a major role. This is largely due to the impact of the no cellulosic cell wall components and the cellulose in the secondary cell wall on these chemical processes.(Tausif et al., 2018).The earliest evidence for the use of cotton fibers in textiles is from the Mehrgarh and Rakhigarhi sites in India, which date to approximately 5000BC.The Indus Valley Civilization, which spanned the Indian Subcontinent from 3300 to 1300 BC, was able to flourish due to cotton cultivation, which provided the people of this culture with readily available sources of clothing and other textiles (ADNAN ALI and IMRAN SARWAR, 2010).While cotton cultivation was widespread in both Arabia and Iran, this textile plant didn't make its way to Europe in full force until the late middle Ages. Before this point, Europeans believed that cotton grew on mysterious trees in India, and some scholars during this period even suggested that this textile was a type of wool that was produced by sheep that grew on trees(Bulliet, 2009,Geczy, 2013 #84). Studying over 200 species of water repellent plants, Neinhuis and Barthlott found an ideally wonderful hydrophobic effect on lotus (Nelumbonucifera) leaves which leads to

supreme self-cleaning properties, so-called lotus effect. The rough structure of lotus leaves (hills and valleys template) causes a reduced contact area with water.

2.3 Overview of nanotechnology

Nanotechnology (or "nanotech") is the use of matter on an atomic, molecular, and supramolecular scale for industrial purposes. The earliest, widespread description of nanotechnology referred to the particular technological goal of precisely manipulating atoms and molecules for fabrication of macro scale products, also now referred to as molecular nanotechnology (dos Santos Ramos et al., 2020, Jayavarthanan et al., 2017).A more generalized description of nanotechnology was subsequently established by the National Nanotechnology Initiative, which defined nanotechnology as the manipulation of matter with at least one dimension sized from1 to 100 nanometers. This definition reflects the fact that quantum mechanical effects are important at this quantum realm scale, and so the definition shifted from a particular technological goal to a research category inclusive of all types of research and technologies that deal with the special properties of matter which occur below the given size threshold (Jayavarthanan et al., 2017). It is therefore common to see the plural form "nanotechnologies" as well as "nanoscale technologies" to refer to the broad range of research and applications whose common trait is size. Nanotechnology as defined by size is naturally broad, including fields of

science as diverse as surface science, organic chemistry, molecular biology, semiconductor physics, energy storage engineering micro fabrication and molecular engineering. The associated research and applications are equally diverse, ranging from extensions of conventional device physics to completely new approaches based upon molecular self-assembly from developing new materials with dimensions on the nanoscale to direct control of matter on the atomic scale. Scientists currently debate the future implications of nanotechnology (Jadhav et al., 2021).Nanotechnology may be able to create many new materials and devices with a vast range of applications, such as in nanomedicine, nanoelectronics, biomaterials energy production, and consumer products. On the other hand, nanotechnology raises many of the same issues as any new technology, including concerns about the toxicity and environmental impact of nonmaterial's and their potential effects on global economics, as well as speculation about various doomsday scenarios (Dhanapal). These concerns have led to a debate among advocacy groups and governments on whether special regulation of nanotechnology is warranted a nanoparticle or ultrafine particle is usually defined as a particle of matter that is between 1 and 100 nanometers (nm) in diameter. The term is sometimes used for larger particles, up to 500 nm or fibers and tubes that are less than 100 nm in only two directions. At the lowest range, metal particles smaller than 1 nm are usually called atom clusters instead (Dhanapal).

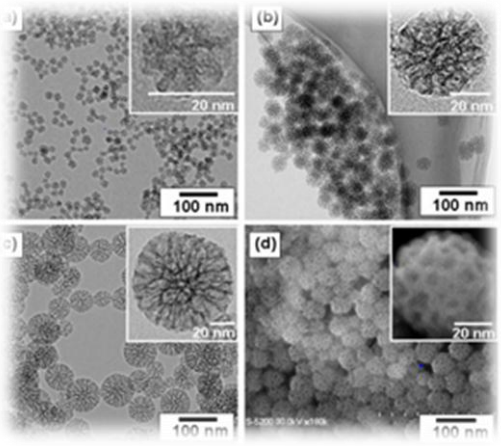


Figure (2-1): TEM (a, b, and c) images of prepared mesoporous silica nanoparticles with mean outer diameter: (a) 20nm, (b) 45nm, and (c) 80nm. SEM (d) image corresponding to (b) (Batnyam, 2021, Ghamami and Ghammamy, 2019).

The insets are a high magnification of mesoporous silica particle. Nanoparticles are usually distinguished from microparticles (1-1000 μm), "fine particles" (sized between 100 and 2500 nm), and "coarse particles" (ranging from 2500 to 10,000 nm), because their smaller size drives very different physical or chemical properties, like colloidal properties and optical or electric properties. Being more subject to the Brownian motion, they usually do not sediment like colloidal particles that conversely are usually understood to range from 1 to 1000 nm. Being much smaller than the wavelengths of visible light (400-700 nm), nanoparticles cannot be seen with ordinary optical microscopes, requiring the use of electron microscopes. For the same reason dispersions of nanoparticles in

transparent media can be transparent whereas suspensions of larger particles usually scatter some or all visible light incident on them. Nanoparticles also easily pass through common filters, such as common ceramic candles, so that separation from liquids requires special nanofiltration techniques (Giri et al., Krebs et al., 2008).

The properties of nanoparticles often differ markedly from those of larger particles of the same substance. Since the typical diameter of an atom is between 0.15 and 0.6 nm, a large fraction of the nanoparticle's material lies within a few atomic diameters from its surface. Therefore, the properties of that surface layer may dominate over those of the bulk material. This effect is particularly strong for nanoparticles dispersed in a medium of different composition since the interactions between the two materials at their interface also become significant (Preining, 1998).

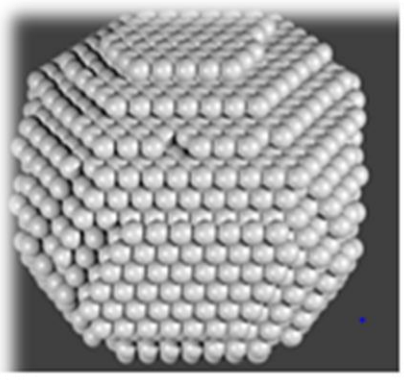


Figure (2-2): Idealized model of a crystalline nanoparticle of platinum, about 2 nm in diameter, showing individual atoms (Alayoglu et al., 2009).

Nanoparticles occur widely in nature and are objects of study in many

sciences such as chemistry, physics, geology and biology. Being at the transition between bulk materials and atomic or molecular structures, they often exhibit phenomena that are not observed at either scale. They are an important component of atmospheric pollution, and key ingredients in many industrialized products such as paints, plastics, metals, ceramics, and magneticarticles. The production of nanoparticles with specific properties is an important branch of nanotechnology (t Amount Percent and Over).

In general, the small size of nanoparticles leads to a lower concentration of point defects compared to their bulk counterparts, but they do support a variety of dislocations that can be visualized using high resolution electron microscopes. However, nanoparticles exhibit different dislocation mechanics, which, together with their unique surface structures, results in mechanical properties that are different from the bulk material (Wasisto et al., 2019).Anisotropy in a nanoparticle leads to a lot of changes in the properties of the nanoparticles. Non-spherical nanoparticles of gold, silver, and platinum due to their fascinating optical properties are finding diverse applications and are of great interest in the field of research. Non-spherical geometries of nanoprisms give rise to high effective cross-sections and deeper colors of the colloidal solutions. The possibility of shifting the resonance wavelengths by tuning the particle geometry is very interesting for using these nanoparticles in the fields of molecular labeling, for bimolecular assays, trace metal detection, and nontechnical applications.

Anisotropic nanoparticles display a specific absorption behavior and stochastic particle orientation under unpolarized light, showing a distinct resonance mode for each excitable axis. This property can be explained based on the fact that on a daily basis there are new developments being made in the field of synthesis of these nanoparticles for preparing them in high yield (Pearce et al., 2021, Eustis, 2006).

2.3.1 Class Nanotechnology

There are three main classes or types of intentionally produced nanomaterials:

- Carbon-based
- Metal-based
- And nanocomposites

- Carbon based non materials are intentionally produced fullerenes.

These include carbon nanotubes and buck balls.

Whereas buck balls are spherical in shape, a nanotube is cylindrical, with at least one end typically capped with a hemisphere of the buck ball structure. Their name is derived from their size, since the diameter of a nanotube is on the order of a few nanometers (approximately 50,000 times smaller than the width of a human hair), while they can be up to several centimeters in length (Berg, 2010).

-There are two main types of nanotubes:

- Single-walled nanotubes (SWNTs)

- And multi-walled nanotubes (MWNTs).

Figure (2-3): Rotating single walled zigzag carbon nanotube (Hussain and Naeem, 2019, Hussain et al., 2019).

2.3.1.1 Synthesis of Carbon nanotubes

Techniques have been developed to produce carbon nanotubes in sizable quantities including

1- Arc discharge

2-Laser ablation

3-High-pressure carbon monoxide disproportionate

4- Chemical vapor deposition (CVD)

2.3.2 Structure Nanotechnology

Nanotechnology refers to manipulating the structure of matter on a length scale of some small number of nanometers, interpreted by different people at different times as meaning anything from 0.1 nm (controlling the arrangement of individual atoms) to 100 nm or more (anything smaller than micro technology) (El-Diasty and Regab, 2013).

2.3.3 Advantages of Nanotechnology

Nanotechnology is the study of controlling matter on a nuclear, atomic and supramolecular scale. Such fields include organic chemistry, micro fabrication, molecular biology, atomic science, surface science, and semiconductor material science (Roco et al., 2000).

2.3.4 Applications of nanotechnology

A large amount of research is being devoted to the development of nano composites of different types for various applications, including structural materials, high performance coatings, catalysts, electronics, photonics and biomedical systems. In commercial products, although most applications are limited to the bulk use of passive nano materials (Abdin et al., 2013). Examples include titanium dioxide and in sunscreen, cosmetics and some food products; silver nanoparticles in food packaging, clothing, disinfectants and household appliances such as Silver nanocarbon nanotubes for stain-resistant textiles; and cerium oxide as a fuel catalyst.

The Project on Emerging Nanotechnologies estimated that over 1300 manufacturer identified nanotech products are publicly available, with new ones hitting the market at a pace of 3–4 per week.

2.3.5 Fabrication of nanotechnology

There are four main types of intentionally produced non materials:

The two basic approaches to making nanostructures involve either:

• top-down approach whereby an existing solid is gradually reduced

In size using some external radiation and/or chemical methods.

•And a bottom-up approach whereby the nanostructure is built atom by atom from scratch.

CHAPTER 3

MATERIALS AND METHODS

3.1. **Materials**

3.1.1 Cotton Fabrics

Plain woven and bleached 100% cotton fabric with 135 ± 5 g/m^2 mass density and having 56/cm warp and 40/cm weft threads was purchased from a local market

3.1.2 Silica

Silicon dioxide, also known as silica, is an oxide of silicon with the chemical formula SiO_2, most commonly found in nature as quartz and in various living organisms. In many parts of the world, silica is the major constituent of sand. Silica is one of the most complex and most abundant families of materials, existing as a compound of several minerals and as a synthetic product. Notable examples include fused quartz, fumed silica, silica gel, and aero gels. It is used in structural materials, microelectronics (as an electrical insulator) and as components in the food and pharmaceutical industries.(Boussaa et al., 2016, Naveen et al., 2018)

▶ Safety Data Sheet for Silicon dioxide 113126.(Das et al., 2019)

-Material Safety Data Sheet or SDS for Silicon dioxide 113126 from - Merck for download or viewing in the browser.

-Catalog Number 113126.

-Product Name Silicon dioxide.

Chemical formula	**SiO_2**
Molar mass	60.08 g/mol
Density	2.648(α quartz),2.196 (amorphous) g • cm^{-3}
Melting point	1,713 ° C (3,115 ° F; 1,986 K) *T*
Boiling point	2,950 ° C (5,340 ° F; 3,220 K)
Thermal conductivity	12(|| c-axis), 6.8 (c-axis), 1.4 (am.) W/(m·K)

Table (3-1) Properties of Silicon dioxide (Naveen et al., 2018).

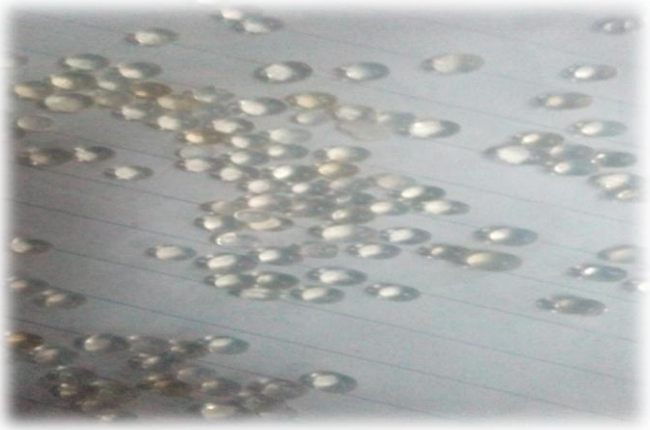


Figure 3-2: Silica

3.1.3 Poly Vinyl Alcohol

(PVA) synthetic polymer, It is used in papermaking, coating, textile warp sizing as a thickener and emulsion stabilizer in PVA.

- Chemical Product

MSDS Name: Polyvinyl alcohol, 75 - 100% hydrolyzed, M.W. 89.000 – 95000.

- Appearance: powder

3.1.4 Polyethylene Glycol/400

PEG is a polyether compound derived from petroleum with many applications, from industrial manufacturing to medicine (Bismark et al., 2018).

Name	**Chemical formula**	**Density**	**Molar mass**	**Melting Point**
PVA	C_2H_4O	1.19 – 1.31 g/cm^3	44.05g/mol	*392 F^0 , 200 C^0*
PEG	$C_{2n}H_{4n+2}O_{n+1}$	*1.125 g/cm^3*	44.05n + 18.02 *g/mol*	*395 F^0,200 C^0*

Table 3-2 Properties of PVA and PEG (Bismark et al., 2018).

3.1.5 Ethanol (C_2H_5OH)

Ethanol (also called ethyl alcohol).

It is a simple alcohol with the chemical formula C_2H_6O. (An ethyl group linked to a hydroxyl group), and is often abbreviated as EtOH. Ethanol is a volatile, flammable, colorless liquid with a slight characteristic odor (Nemec et al., 2017).

Molar mass (g/mol)	Density (g • cm^{-3})
46.07	0.789

Table 3-3 Properties of Ethanol (Li et al., 2005).

3.1.6 Ammonium hydroxide (NH_4OH)

Ammonia solution, also known as ammonia water ammonium hydroxide ammoniac liquor, ammonia liquor, aqua ammonia, aqueous ammonia, or (inaccurately) ammonia, is a solution of ammonia in water, is used to redacting agent . It can be denoted by the symbols NH_3 (aq). Although the name ammonium hydroxide suggests an alkali with composition [NH4+][OH−], it is actually impossible to isolate samples of NH_4OH (Fernelius, 2009).

Molar mass (g/mol)	Density (g • cm^{-3})
35.05	0.91

Table 3-4 Properties of ammonium hydroxide

3.1.7 Distilled water

Distilled water is water that has been boiled into vapor and condensed back into liquid in a separate container. Impurities in the original water that do not boil below or near the boiling point of water remain in the original container. Thus, distilled water is a type of purified water (https :).

3.2. Method

3.2.1. Preparation of silica nanoparticles

The spherical silica nanoparticles were prepared by using boll mill machine.

Speed r.p.m for 3h and granule grinding

Resulting in powder spherical silica and tow scale mesh (150 nm and 300nm).

Figure 3-2: boll mill machine

3.2.2. Preparation of PVA Solutions

Immersion (10.25g) PVA in (300ml) water and heating at 150 ˚C for 20 min under stirring depends on the grade of PVA used.

Note: dissolution of PVA on water must be 95 % to avoid the water evaporation at 200 c^0

Figure 3-3: Heating Process

3.2.3 Solution Preparation

Solutions of silica nanoparticles were prepared firstly a mixture of 57 g SiO_2 counting 63 ml PVA/PEG are mixing and 170.8ml ethanol was prepared, then it was mixed with another mixture of 70 ml distilled water 170.8 ml ethanol, NH_4OH was used as a catalyst to control the particle size (*Table 3-5)*. The mixtures were stirred continually using magnetic stirrer at room temperature 30 °C for 1 h.

Speed of magnetic stirrer 3507 r.p.m.

Figure0-4: Magnetic stirrer

Sample	H_2O(ml)	SiO_2(g)	NH_3OH(ml)	EtOH(ml)	PVA/PEG(ml)	
Sol_1	70	57	10	170.8	63	150nm
Sol_2	70	57	15	170.8	63	300nm

Table 3-5 Components and compositions for preparing silica nanoparticles.

3.2.4. Preparation of super hydrophobic cotton fabrics

-The cotton fabrics were immersed in a solution of silica nanoparticles at 30°C for 5 min while stirring.

-The wet fabrics were squeezed using a pressure, resulting in A wet-pick-up of 70%.

-The fabrics were then dried at 80°C for 3 min and cured at 160°C for 3 min with a preheated 50°C for 30 min to immobilize silica nano-particles on the cotton fabric.

-A laboratory Cleaning (DL-2002, Daelim Engineering, Korea) was used to remove the residual silica particles not immobilized on the fabric.

-A standard laundering condition 2 g of detergents in 400 ml of distilled water while stirring at 40°C for 30 min was applied (ISO 105-A01).

-For the impartment of hydrophobic property to the hydrophilic cotton fabrics previously applied with silica nanoparticles they were immersed in an aqueous solution at the WR agent concentration of 0.1–1.0 wt. % at 30°C For 5 min while stirring and then squeezed.

-Subsequently the fabrics were dried and cured simultaneously at180°C for 3 h.

(B1) *(B2)*

Figure 3-5: Cotton fabrics were immersed in a solution of silica nanoparticles

3.2.5 Drying process and curing

After compositing process, the samples were put into the refrigerator with -180˚C for 3 h for drying. *(Figure 3-7*) shows the method of drying process.

Note: coating process the fabric was done three times

Figure 3-6: Drying process and curing

(B1) (B2)

Figure 3-7: Fabric after treatment sample

3.3 Characterization

All characteristic was determined according to Standard ISO 7198: 1998[78]. The results were evaluated and analyzed all tests were done ten times and the average of each test were obtained.Samples were prepared of 50*50 cm, and then the weight gram per meter square.

3.3.1 Flame test

When it comes staying ahead in style, fabrics definitely play a significant role. With different kinds of fabrics, including cotton, linen, rayon and georgette, it becomes primarily necessary to understand and identify one fabric from the other, especially if you want to make your presence in the occasion a lasting one. In fact, not just for the end consumers, identification of fabrics is equally important for quilters, who need to sew with only 100% cotton fabrics (Hargrave, 1997, McKelvey and Wickell, 2002).

3.3.2 H_2SO_4 test

A correlation of the degradation of cellulose in aqueous alkali solution was established with its so-called "amorphous" content. To achieve this end, cellulose was regenerated under different preparative conditions from its cup ammonium solution and cotton linter was acid-hydrolyzed into a fibrous form and the pulp was physically milled to powder.(Pielichowska and Blazewicz, 2010)

3.3.3 PVA/PEG Content

The PVA/PEG content of each sample calculated from equation (3-1)

PVA/PEG content (%)

$$= \frac{\boldsymbol{weight\ of\ the\ composite\ samples - weight\ of\ Pure\ fabric}}{\boldsymbol{weight\ of\ the\ composite\ sample}} \boldsymbol{X100} \quad \textit{... (0-1)}$$

3.3.4FourierTransformInfrared(FTIR)

Spectroscopy Infrared spectra of samples were obtained with FTIR spectrometer (FTIR- 8400s). All spectra were recorded in the range from 4000 to 400 cm^{-1} with 4 cm^{-1} resolution, accumulating 128 scans. To ensure the reproducibility of obtained spectra three samples of each type were measured. FTIR mode was preferred since it allows the chemical analysis of the surface enabling a better characterization of the different coatings employed. [68] In FTIR spectrometer the calculated penetration depth of FTIR into the sample varies between ~0.55 μm at 4000 cm^{-1} and

~3.30 µm at 400 cm-[1]

3.3.5 Water contact angles

Static water contact angle (WCA) and water shedding angle (WSA) or droplet angle were measured with a contact angle instrument (OCAH-200 Data Physics, Germany).

Contact angle refers to a method of calculating surface free energy by evaluating the interface of a liquid and a solid surface.

A contact angle (also referred to as a wetting angle) is formed when a drop of liquid is placed on a material surface and the drop forms a dome shape on the surface. The angle formed between the surface and the line tangent to the edge of the drop of the water is called the contact angle (Subedi, 2011).

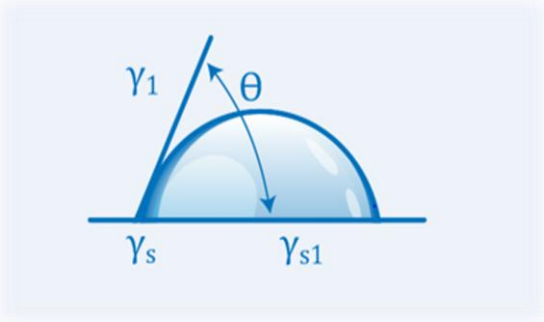


Figure 3-8: contact angle

Figure 3-8: contact angle in the illustration above. As the drop of water spreads across a surface and the dome becomes flatter, the contact angle becomes smaller. If the drop of water beads up on the surface (as you might see on a water resistant article of clothing or a waxed car) the dome becomes taller and the angle becomes larger (Holmes, 2000).

3.3.5.1 Contact Angle Measurement:

When the angle the drop makes with surface is measured, the resulting angle indicates whether the drop of water is more attracted to itself or to the surface it is on. Unseen forces on the surface of the material are acting on the water drop as soon as contact is made (Spori, 2010).

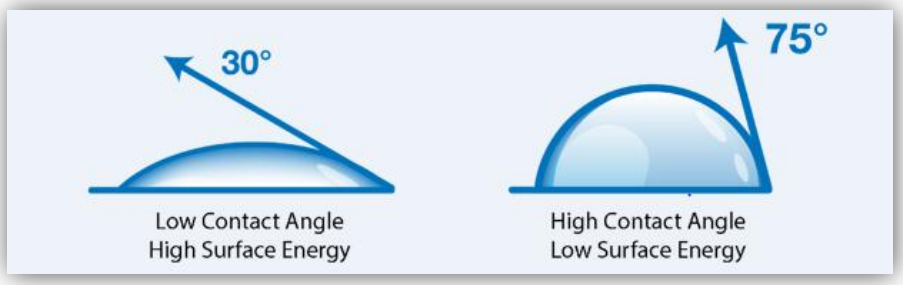


Figure 3-9: surface energy in contact angle (Kozbial et al., 2014).

Figure 3-10: if these forces are strong, their pull on the water drop will cause the drop to "wet out" or spread further over the surface. If the forces are not stronger than the attractive forces the drop has for itself, then the drop will constrict into a shape as close to a sphere as it can (Reece, 2008). These two forces are working in tandem on the drop creating an angle that can be measured.

In many manufacturing and assembly processes a liquid or a melted material needs to spread out adequately on a surface. A coating needs to flow into small crevices and thoroughly cover the surface of circuit boards.

3.3.5 Resistance to water absorption

-Waterproof properties of the treated fabrics were quantified by measuring hydrostatic due to water rise over the fabrics.

-The hydrostatic head supported by a fabric is a measure of the resistance to the passage of water through the fabric.

-The impregnation specimen in water for 30 min.

- Three specimens from each sample material were measured.

-a fabric sample with 20×20 cm2 dimension.

3.3.6 Tensile strength

-For mechanical characterization (stress-strain curves), an Instron (3365 Instron, USA) instrument was used. Tree specimens from each material were measured.

-A dog bone shaped sample of 30 mm length along the axis of warp threads and 5 mm width was cut by a mechanical cutter.

-Samples were mounted in pneumatic clamps and a constant rate of extension 5 mm/min was used for each measurement at room conditions.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Experimental analysis

4.1.1 Effect of flame for fabric

- **Flame:** Burning Quickly, orange/yellow flame
- **Odor:** paper burning like odor
- **Residue:** Light and feathery gray ash, ash is black if mercerized
- **Approaching Flame:** Does not shrink away, Scorches, ignites quickly
- **Removed From Flame:** Continues to burn rapidly has afterglow
- After burning it's converting into ashes gray or black powder

Figure 5-1 Flam test of cotton

4.1.2 Effect H_2SO_4 Solubility Test

Cotton is a robust fiber and alkalis have no to little effect on cotton but yes cotton is soluble in Sulphuric acid with 70% solution at room temperature.

4.1.3 Concentration contain

Concentration of a substance (Solutions of silica nanoparticles) in a quantity solute present in given quantity of solution.

Measurement of concentration using Presley device.

Figure 5-2 Presley device

Solution B_1:

Concentration=18.5%

Sediment =1.136033

Solution B_2:

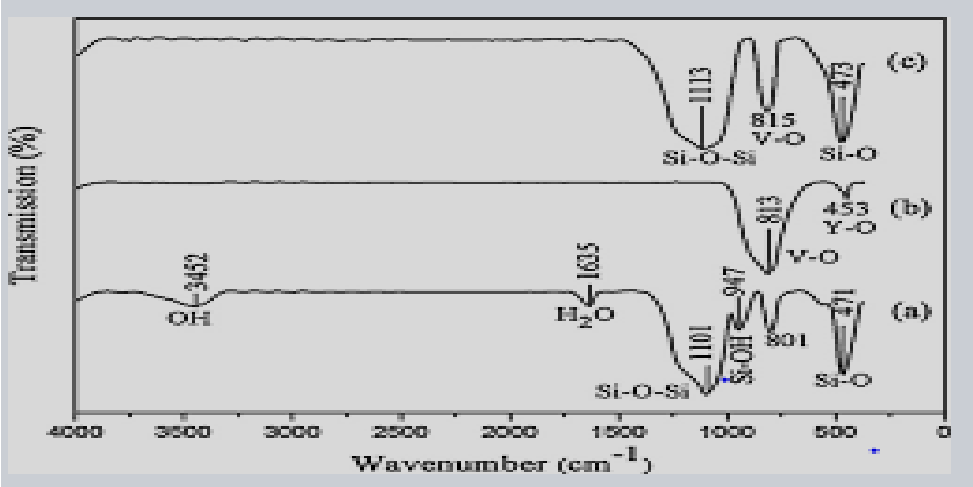
Concentration=15%

Sediment =1.35033

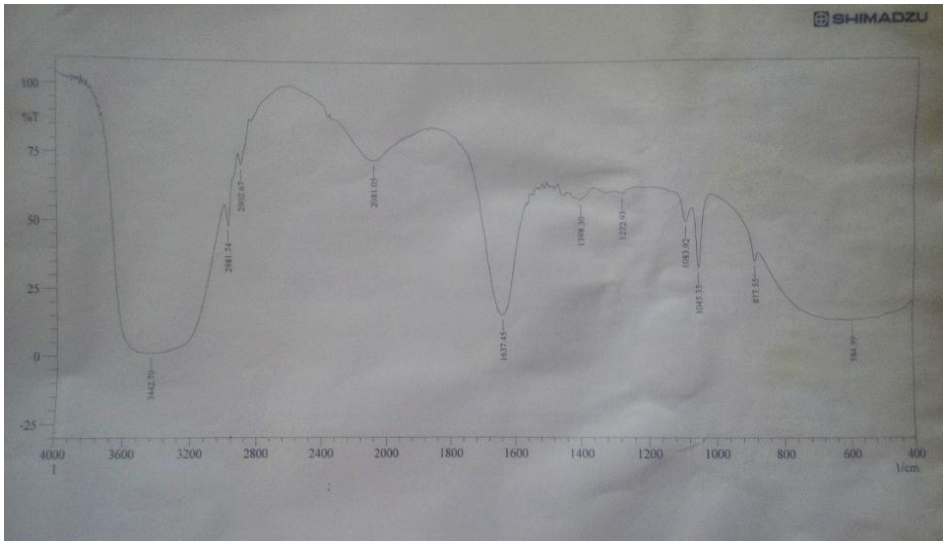
4.1.3 PVA/PEG Content =

(84-56)/84=33%

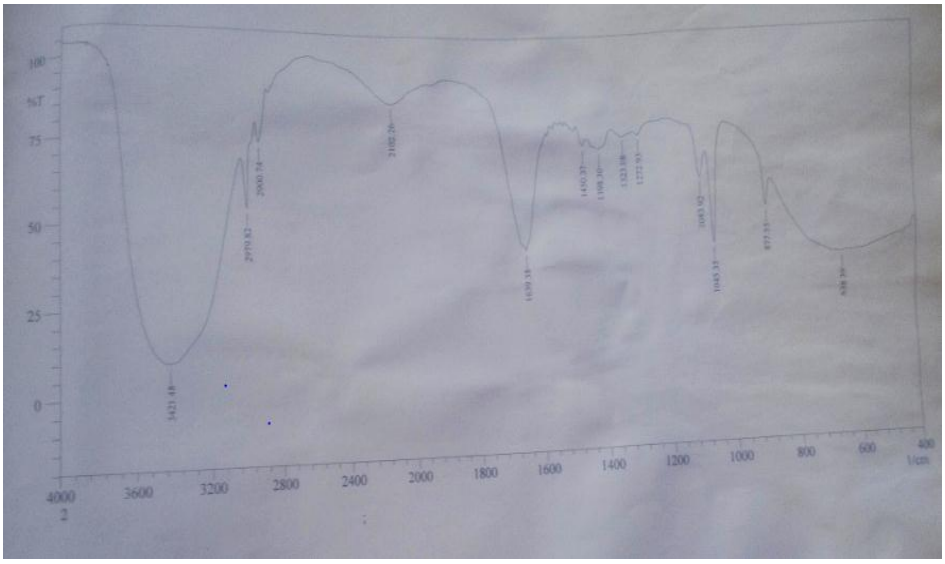
4.1.4 Characterization of the synthesized silica nanoparticles using FTIR.



(a)



(b)



(c)

Figure 5-3: Fourier Transform Infrared (FTIR) (a) stander of cotton fabric coated with SiO_2, (b) and (c) cotton fabrics coated with (PEG and PVA /SiO_2) nanoparticles.

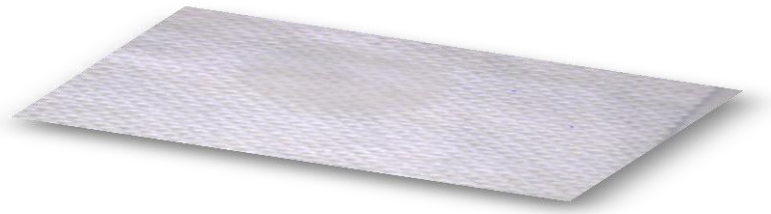
FTIR analysis also gives a set of peak values unique for the sample along with information of the plant peptides that are present in the sample as the plant extract acts as a reducing agent figure 5-3. FTIR analysis is used to confirm the presence of plant peptides visible due to the bending produced by amide bonds.fabrics treated with silicone on the top of the nanocomposite treatment, there exists a linear decreasing trend in the ratio obtained by dividing signal area (under the curve) of the O-H stretching peak at 3452 cm^{-1}.The decrease in this ratio can directly increase in hydrophobicity of the treatments and also allows optimizing the required nanoparticle concentration for droplet.

4.1.5 Wettability

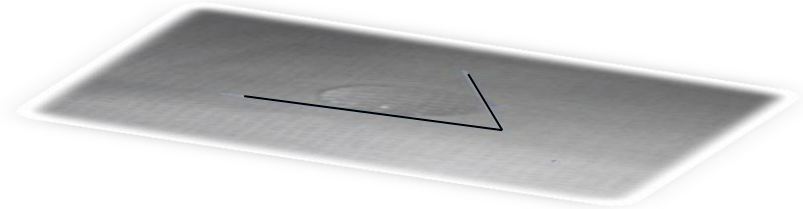
Cellulosic materials are well known for their super wettability. Hence untreated natural cotton fibers can take up water many times of their own dry weight. This extreme wettability comes from high surface area of cotton fibers which are made up of hydrophilic cellulosic polymer. Therefore, it was not possible to measure WCA on pristine cotton fabric.

Wettability is the amount to which a liquid can spread on a surface determined by the intermolecular forces between the surface and the liquid.

A specimen is subjected to a steadily increasing pressure of water on one face, under standard conditions, until penetration occurs in three places. The water pressure may be applied from below or from above the test specimen. The hydrostatic head supported by a fabric is a measure of the



(a)



(b)

Figure 5-3: (a) and (b) contact angle of treated and un treated fabric

This measurement allows us to understand the relationship between the liquid drop and the surface. -Young's equation mathematically expresses the relationship between the surface energy of the solid surface (Υ_s), the surface tension (which is a term for surface energy of liquid phase substances) of the liquid (Υ_l) and the surface energy of the interface (Υ_{sl}).

Young's Equation: $\Upsilon_s = \Upsilon_{sl} + \Upsilon_l \cos\theta$ …….. (1)

$\Upsilon_{sl} = F_{s1}/2L_{s1}$…… (2)

$\Upsilon_l = F1/2L_1$…….. (3)

Sample	Υ_{sl}(N/m)	Υ_{l}(N/m)	cos θ	Υs(N/m)
B1	8.232	9.770	120	10.76
B2	8.244	9.860	115	4.07

Table 4-1 cotton fabrics treated with the rater replant agent

Sample	Υ_{sl}(N/m)	Υ_{l}(N/m)	cos θ	Υs(N/m)
B1	8.33	9.770	150	-.13
B2	8.526	9.59	155	-.17

Table 4-2 cotton fabrics treated with both silica nanoparticles and the water replant agent

Silica nanocomposites treatment also produced similar problems against contact with large water droplets Such hydrophobicity performance indicators (i.e., wetting by larger volume water droplets >10 μ L) are not common in the literature but are important for the real performance (Ware et al., 2018).

Based on these observations the bilayer method (polymer nanocomposite as the base layer and silicone as the outer) was used to ensure that the fabrics are not wet when in contact with larger water droplets rather than small static droplets.Most reported hydrophobic textiles demonstrate low droplet angles (≤ 10°) when large droplet volumes of more than 40 μ L are used (Zahid et al., 2017).

4.1.6 Resistance to water absorption

For theoretical estimation of needed for absorption of water into waterproof porous structure *Table (4-3*)

A	W_2	W_1	W	B_1	W_2	W_1	W	B_2	W_2	W_1	W
1	75	86	11	1	79.6	84	4.4	1	82.3	84.8	2.5
2	74.3	85	10.7	2	81.9	83	1.1	3	79	84	5
3	75.9	85	9.1	3	81.4	83	1.6	3	81	84.7	3.7

Table 4-3 compared the water absorption of the treatment and treatment fabric.

To verify above theoretical values hydrostatic head experiments were performed for all treated samples including B_1 and B_2 (*Table 4-3*). Untreated fabric (A) being highly hydrophilic did not impregnate any water allowing continuous.

4.1.7 Mechanical Characteristics

Mechanical strength and flexibility of textile fabrics are prime parameters for quality and hence it is important to achieve water repellency without altering the fabric's mechanical characteristics to a significant extent.

For longitudinal direction the sample was held on device as shown in Figure 5-3, with the following setting:

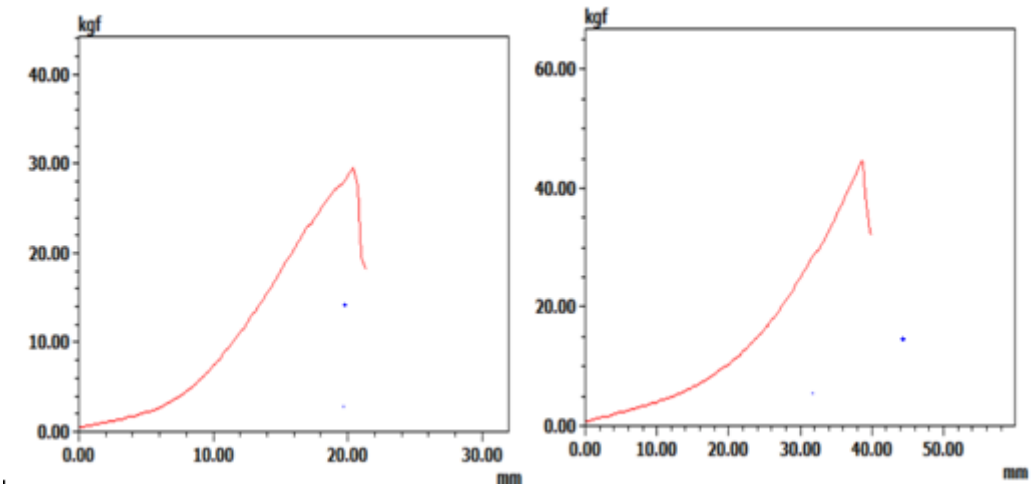
Sample Dimension 30*5cm, Length between clamps and the results were average of 3 times testing

Figure 5-4: fabric tensile strength testing method

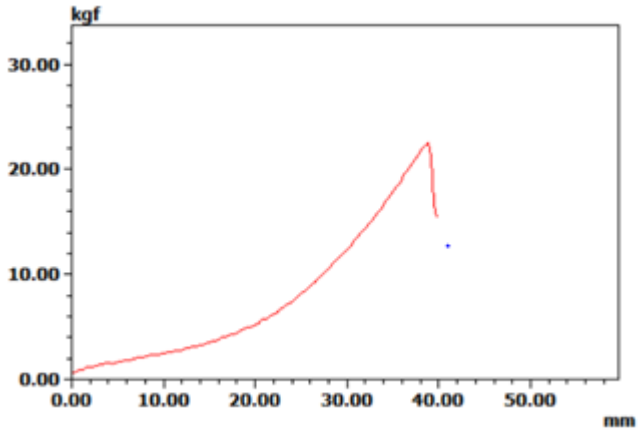
In this study typical stress-strain were obtained from calculations equation (1) (2) Young's Modulus equation (3) from all treated fabrics B_1, B_2 and untreated A (see supporting information Figure and *table belows*)

Sample a	**F_{max}**	**Sample b_1**	**F_{max}**	**Sample b_2**	**F_{max}**
S_1	27.52	S_1	50.62	S_1	36.80
S_2	24.17	S_2	43.34	S_2	32.49
S_3	29.50	S_3	44.55	S_3	30.65
Mean	27.06	**Mean**	46.17	**Mean**	33.1
Stdev	2.69	**Stde v**	3.90	**Stde v**	3.15

Table 4-4 compared the F_{max} of the treatment and untreatment fabric



(A) (B_1)



Maximum force

Elongation

(B_2)

Figure 5-5 Mechanical characterization: Representative maximum force of an untreated and (B_1, B_2) treated (A) samples and percent elongation at break.

Stress=F/A…….. (1)

Strain= (L_1-L_2)/l1…… (2)

Young's Modulus=stress/strain…… (3)

Sample	**Stress(N/m^2)**	**Strain**	**Young's Modulus(Mpa)**
A	180.4	26.7	6.74
B_1	307.4	46.7	6.58
B_2	220.6	40	5.52

Table 4-5 stress-strain of these fabrics and Young's modulus

The stress-strain of these fabrics extends up to 25% strain. This could be mainly attributed to the very low dry pick up weight (1%) of the PVA/PEG treatment. As mentioned earlier this was intentional in order to maintain a minimum amount of (PVA/ PEG) chemistry.

Best hydrophobic performance it suffices to argue that as a result of this treatment no significant deterioration or loss in the mechanical characteristics (Young's modulus and maximum elongation at break) of the original fabric occurs.

CHAPTER 5

CONCLUSION AND PROSPECTS

5.1 Conclusion

Synthesized silica nanoparticles via the sol–gel process and prepared hydrophobic cotton fabrics by the combined applications of the silica nanoparticle and a cost effective WR agent. The synthesized silica particles were characterized to have average diameters of 150 and 300 nm, depending on the concentration of NH_4OH catalyst. For the cotton fabrics treated with silica nanoparticles of average diameter 300 nm, water contact angles above 105°could be easily obtained even with a very low WR agent concentration of 0.1wt% at which no hydrophobicity was exhibited for the neat cotton fabric treated with the WR agent only. Overall, it is considered that the combined treatment of silica nanoparticles and cost-effective WR agent can be a practical process to obtain cotton fabrics with super hydrophobicity.

5.2 Scope for Future Work

-The ease of application technique industrial scale availability of the low-cost polymers and the non-toxic ingredients would allow this fabric treatment to be implemented in large scale textile treatment facilities.

-Moreover developed fabrics can be further tested in applications such as filtering, oil-water separation and as special bags for organic oil spills

cleaning.

- Use freezing dryer in drying process.

-Apply the different treatments was performed by Scanning Electron Microscopy (SEM) in order to evaluate the chemical characterization .

REFERENCES

ABBOTT, S. & HANSEN, C. M. 2008. *Hansen solubility parameters in practice*, Hansen-Solubility.

ABDIN, Z., ALIM, M. A., SAIDUR, R., ISLAM, M. R., RASHMI, W., MEKHILEF, S. & WADI, A. 2013. Solar energy harvesting with the application of nanotechnology. *Renewable and sustainable energy reviews,* 26**,** 837-852.

ADNAN ALI, M. & IMRAN SARWAR, M. 2010. Sustainable and Environmental freindly fibers in Textile Fashion (A Study of Organic Cotton and Bamboo Fibers). University of Borås/Swedish School of Textiles.

AHLERS, M., BUCK-EMDEN, A. & BART, H.-J. 2019. Is dropwise condensation feasible? A review on surface modifications for continuous dropwise condensation and a profitability analysis. *Journal of advanced research,* 16**,** 1-13.

ALAYOGLU, S., ZAVALIJ, P., EICHHORN, B., WANG, Q., FRENKEL, A. I. & CHUPAS, P. 2009. Structural and architectural evaluation of bimetallic nanoparticles: a case study of Pt− Ru core− shell and alloy nanoparticles. *Acs Nano,* 3**,** 3127-3137.

BARTHLOTT, W., MAIL, M. & NEINHUIS, C. 2016. Superhydrophobic hierarchically structured surfaces in biology: evolution, structural principles and biomimetic applications. *Philosophical Transactions*

of the Royal Society A: Mathematical, Physical and Engineering Sciences, 374**,** 20160191.

BATNYAM, N. 2021. CURRENT TRENDS IN NANOELECTRONICS IN MONGOLIA. *ББК 81+ 81 Я41***,** 23.

BELT, C. & COTTON, K. Plant fiber from the genus Gossypium.

BERENDJCHI, A., KHAJAVI, R. & YAZDANSHENAS, M. E. 2011. Fabrication of superhydrophobic and antibacterial surface on cotton fabric by doped silica-based sols with nanoparticles of copper. *Nanoscale research letters,* 6**,** 1-8.

BERG, J. C. 2010. *An introduction to interfaces & colloids: the bridge to nanoscience*, World Scientific.

BISMARK, S., ZHIFENG, Z. & BENJAMIN, T. 2018. Effects of differential degree of chemical modification on the properties of modified starches: Sizing. *The Journal of Adhesion,* 94**,** 97-123.

BOURLINOS, A. B., GOURNIS, D., PETRIDIS, D., SZABó, T., SZERI, A. & DEKáNY, I. 2003. Graphite oxide: chemical reduction to graphite and surface modification with primary aliphatic amines and amino acids. *Langmuir,* 19**,** 6050-6055.

BOUSSAA, S., KHELOUFI, A., ZAOURAR, N. B. & KERKAR, F. 2016. Valorization of Algerian Sand for Photovoltaic Application. *Acta Physica Polonica, A.,* 130.

BROUDY, E. 1993. *The book of looms: a history of the handloom from*

ancient times to the present, UPNE.

BULLIET, R. W. 2009. *Cotton, climate, and camels in early Islamic Iran: a moment in world history*, Columbia University Press.

DAS, G., PATRA, J. K., PARAMITHIOTIS, S. & SHIN, H.-S. 2019. The sustainability challenge of food and environmental nanotechnology: Current status and imminent perceptions. *International journal of environmental research and public health,* 16**,** 4848.

DE PREUX GALLONE, L. & SASSI, F. 2018. Economics of Pollution Interventions.

DEEKSHA, P., DEEPIKA, G., NISHANTHINI, J., HIKKU, G., JEYASUBRAMANIAN, K. & MURUGESAN, R. 2020. Super-hydrophobicity: Mechanism, fabrication and its application in medical implants to prevent biomaterial associated infections. *Journal of Industrial and Engineering Chemistry*.

DENG, X. 2018. Toughening of natural-fibre composites using nano-and microcrystalline cellulose particles.

DHANAPAL, R. Journal of science ENGINEERING.

DOS SANTOS RAMOS, M. A., DOS SANTOS, K. C., DA SILVA, P. B., DE TOLEDO, L. G., MARENA, G. D., RODERO, C. F., DE CAMARGO, B. A. F., FORTUNATO, G. C., BAUAB, T. M. & CHORILLI, M. 2020. Nanotechnological strategies for systemic

microbial infections treatment: A review. *International Journal of Pharmaceutics*, 119780.

EL-DIASTY, A. I. & REGAB, A. Future contributions of nanotechnology to EOR in Egypt. Offshore Middle East (OME) Conference & Exhibition, Doha, Qatar, 2013. e23.

ENIEB, M. & DIAB, A. 2017. Characteristics of asphalt binder and mixture containing nanosilica. *International Journal of Pavement Research and Technology,* 10**,** 148-157.

EUSTIS, S. 2006. *Gold and silver nanoparticles: characterization of their interesting optical properties and the mechanism of their photochemical formation.* Georgia Institute of technology.

FERNELIUS, W. C. 2009. *Inorganic syntheses*, John Wiley & Sons.

GHAMAMI, M. & GHAMMAMY, S. 2019. Fractal Dimensions Analysis and Morphological Investigation of Nanomedicine by Machine-Learning Methods. *Science and Technology of Polymers and Advanced Materials*. Apple Academic Press.

GIRI, A., SHEIKH, A., TATHE, P., SITAPHALE, G. & BIYANI, K. Solid Lipid Nanoparticles: A Brief Review.

GOU, X. & GUO, Z. 2019. Surface topographies of biomimetic superamphiphobic materials: design criteria, fabrication and performance. *Advances in colloid and interface science,* 269**,** 87-121.

GRUNDKE, K., PöSCHEL, K., SYNYTSKA, A., FRENZEL, R., DRECHSLER, A., NITSCHKE, M., CORDEIRO, A., UHLMANN, P. & WELZEL, P. 2015. Experimental studies of contact angle hysteresis phenomena on polymer surfaces—Toward the understanding and control of wettability for different applications. *Advances in colloid and interface science,* 222**,** 350-376.

HARGRAVE, H. 1997. *From fiber to fabric: The essential guide to quiltmaking textiles*, C&T Publishing Inc.

HOLMES, D. A. 2000. Waterproof breathable fabrics. *Handbook of technical textiles,* 282315.

HUSSAIN, M. & NAEEM, M. N. 2019. Rotating response on the vibrations of functionally graded zigzag and chiral single walled carbon nanotubes. *Applied Mathematical Modelling,* 75**,** 506-520.

HUSSAIN, M., NAEEM, M. N. & TAJ, M. 2019. Vibration characteristics of zigzag and chiral functionally graded material rotating carbon nanotubes sandwich with ring supports. *Proceedings of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science,* 233**,** 5763-5780.

INNOVATIVI, M. Growth and Characterization of ZnO and SiC Nanowires.

JADHAV, A. S., URKUDE, R. P. & JANGAM, S. S. 2021. CURRENT

REVIEW ON NANOPARTICLES: PROPERTIES, APPLICATION AND TOXICITY.

JAYAVARTHANAN, R., NANDA, A. & BHAT, M. A. 2017. The impact of nanotechnology on environment. *Materials Science and Engineering: Concepts, Methodologies, Tools, and Applications.* IGI Global.

KACHWAL, V., SHARMA, P. K., SARMAH, A., CHOWDHURY, S. & LASKAR, I. R. 2020. A multistimuli responsive heteroleptic iridium (iii) complex: role of hydrogen bonding in probing solvent, pH and bovine serum albumin (BSA). *Journal of Materials Chemistry C,* 8**,** 6605-6614.

KARIM, N., AFROJ, S., LLOYD, K., OATEN, L. C., ANDREEVA, D. V., CARR, C., FARMERY, A. D., KIM, I.-D. & NOVOSELOV, K. S. 2020. Sustainable personal protective clothing for healthcare applications: a review. *ACS nano,* 14**,** 12313-12340.

KOZBIAL, A., LI, Z., CONAWAY, C., MCGINLEY, R., DHINGRA, S., VAHDAT, V., ZHOU, F., D'URSO, B., LIU, H. & LI, L. 2014. Study on the surface energy of graphene by contact angle measurements. *Langmuir,* 30**,** 8598-8606.

KREBS, F. C., THOMANN, Y., THOMANN, R. & ANDREASEN, J. W. 2008. A simple nanostructured polymer/ZnO hybrid solar cell—preparation and operation in air. *Nanotechnology,* 19**,** 424013.

LI, D.-G., ZHEN, H., XINGCAI, L., WU-GAO, Z. & JIAN-GUANG, Y. 2005. Physico-chemical properties of ethanol–diesel blend fuel and its effect on performance and emissions of diesel engines. *Renewable energy,* 30**,** 967-976.

LIN, Y., CHEN, H., WANG, G. & LIU, A. 2018. Recent progress in preparation and anti-icing applications of superhydrophobic coatings. *Coatings,* 8**,** 208.

LU, X., PENG, Y., GE, L., LIN, R., ZHU, Z. & LIU, S. 2016. Amphiphobic PVDF composite membranes for anti-fouling direct contact membrane distillation. *Journal of Membrane Science,* 505**,** 61-69.

MATTSSON, E. 2013. *Plasma treated water and oil repellent textiles-combining multi-scale surface roughness and low surface energy.*

MCKELVEY, S. & WICKELL, J. 2002. *Quilting and Color Made Easy*, Rodale.

MORADI, S. 2014. *Super-hydrophobic nanopatterned Interfaces: Optimization and manufacturing.* University of British Columbia.

NAVEEN, E., RAMANAN, N., ARVIND, R., DINESH, I., MAYANDI, A. & NAVEEN, D. 2018. Analysis of Mechanical and Wear Properties of Al-SiO2 Composite Material. *Journal of Innovation in Mechanical Engineering,* 1**,** 1-5.

NEMEC, P., MALCHO, M. & PALACKA, M. 2017. INFLUENCE OF

TEMPERATURE ON DIELECTRIC BREAKDOWN OF WORKING FLUID VAPOR IN HEAT PIPE. *Machines. Technologies. Materials.,* 11**,** 178-180.

OH, J. M. 2011. *Electrolyte incorporation into composite electrodes for proton-exchange membrane fuel cells and lithium-ion batteries.* Clemson University.

PEARCE, A. K., WILKS, T. R., ARNO, M. C. & O'REILLY, R. K. 2021. Synthesis and applications of anisotropic nanoparticles with precisely defined dimensions. *Nature Reviews Chemistry,* 5**,** 21-45.

PIELICHOWSKA, K. & BLAZEWICZ, S. 2010. Bioactive polymer/hydroxyapatite (nano) composites for bone tissue regeneration. *Biopolymers***,** 97-207.

PREINING, O. 1998. The physical nature of very, very small particles and its impact on their behaviour. *Journal of aerosol science,* 29**,** 481-495.

REECE, E. 2008. *Field Work: Modern Poems from Eastern Forests*, University Press of Kentucky.

ROCO, M. C., WILLIAMS, R. S. & ALIVISATOS, P. 2000. *Nanotechnology research directions: IWGN workshop report: vision for nanotechnology in the next decade*, Springer Science & Business Media.

SARSHAR, M. A., SWARCTZ, C., HUNTER, S., SIMPSON, J. &

CHOI, C.-H. 2013. Effects of contact angle hysteresis on ice adhesion and growth on superhydrophobic surfaces under dynamic flow conditions. *Colloid and Polymer Science,* 291**,** 427-435.

SPORI, D. M. 2010. *Structural influences on self-cleaning surfaces.* ETH Zurich.

SRIVASTAVA, V. 2020. Pollution Control: A brief review of conventional and nano technological approaches.

STRATAKIS, E. 2012. Nanomaterials by ultrafast laser processing of surfaces. *Science of Advanced Materials,* 4**,** 407-431.

SUBEDI, D. 2011. Contact angle measurement for the surface characterization of solids. *Himalayan Physics,* 2**,** 1-4.

T AMOUNT PERCENT, R. & OVER, C. MAJOR MULTI-USER RESEARCH FACILITIES. *Geosciences,* 15**,** 8.00-40.0.

TAUSIF, M., JABBAR, A., NAEEM, M. S., BASIT, A., AHMAD, F. & CASSIDY, T. 2018. Cotton in the new millennium: Advances, economics, perceptions and problems. *Textile Progress,* 50**,** 1-66.

THOMBARE, N., JHA, U., MISHRA, S. & SIDDIQUI, M. 2016. Guar gum as a promising starting material for diverse applications: A review. *International journal of biological macromolecules,* 88**,** 361-372.

VISHWAKARMA, K., UPADHYAY, N., KUMAR, N., TRIPATHI, D. K., CHAUHAN, D. K., SHARMA, S. & SAHI, S. 2018. Potential

applications and avenues of nanotechnology in sustainable agriculture. *Nanomaterials in plants, algae, and microorganisms.* Elsevier.

VITALE, A., BONGIOVANNI, R. & AMEDURI, B. 2015. Fluorinated oligomers and polymers in photopolymerization. *Chemical reviews,* 115**,** 8835-8866.

WANG, J., XIAO, F. & ZHAO, H. 2021. Thermoelectric, piezoelectric and photovoltaic harvesting technologies for pavement engineering. *Renewable and Sustainable Energy Reviews,* 151**,** 111522.

WANG, Y., WANG, X., XIE, Y. & ZHANG, K. 2018. Functional nanomaterials through esterification of cellulose: a review of chemistry and application. *Cellulose,* 25**,** 3703-3731.

WARE, C. S., SMITH-PALMER, T., PEPPOU-CHAPMAN, S., SCARRATT, L. R., HUMPHRIES, E. M., BALZER, D. & NETO, C. 2018. Marine antifouling behavior of lubricant-infused nanowrinkled polymeric surfaces. *ACS applied materials & interfaces,* 10**,** 4173-4182.

WASISTO, H. S., PRADES, J. D., GULINK, J. & WAAG, A. 2019. Beyond solid-state lighting: Miniaturization, hybrid integration, and applications of GaN nano-and micro-LEDs. *Applied Physics Reviews,* 6**,** 041315.

XIANG, S. & LIU, W. 2021. Self‐Healing Superhydrophobic Surfaces: Healing Principles and Applications. *Advanced Materials Interfaces*, 2100247.

ZAHID, M., ATHANASSIOU, A. & BAYER, I. S. 2018. Multifunctional and Responsive Textile Nanocomposites for High Value Applications. *University of Genova-Istituto Italiano di Tecnologia*, 1-123.

ZAHID, M., HEREDIA-GUERRERO, J. A., ATHANASSIOU, A. & BAYER, I. S. 2017. Robust water repellent treatment for woven cotton fabrics with eco-friendly polymers. *Chemical Engineering Journal,* 319**,** 321-332.

ZAHID, M., MAZZON, G., ATHANASSIOU, A. & BAYER, I. S. 2019. Environmentally benign non-wettable textile treatments: A review of recent state-of-the-art. *Advances in colloid and interface science,* 270**,** 216-250.

ZHANG, F.-Y., ADVANI, S. G. & PRASAD, A. K. 2011. Advanced high resolution characterization techniques for degradation studies in fuel cells. *Polymer Electrolyte Fuel Cell Degradation,* 365.

ZHAO, Y., TANG, Y., WANG, X. & LIN, T. 2010. Superhydrophobic cotton fabric fabricated by electrostatic assembly of silica nanoparticles and its remarkable buoyancy. *Applied Surface Science,* 256**,** 6736-6742.