

Fabrication of Super hydrophobicity of cotton fabric treated with silica Nano particles and water repellent agent

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Abstract: To obtain the super hydrophobic water-repellent cotton fabrics, cotton fabrics were treated with silica nanoparticles and/or a cost-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 300 nm. 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 0.3 wt% 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 75° can be obtained even at the very low WR agent concentration of 0.1 wt%. Therefore, super hydrophobic cotton fabrics could be obtained the combined treatment of silica nanoparticle and WR agent, which is cost effective compared with fluorinate silane treatment.

Keywords— super hydrophobic cotton, silica nanoparticles, water repellent, contact angle.

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 hydrophilicity and structural instability upon contact with water.[1] 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. Modifying fiber surfaces by changing their roughness and chemistry is considered to be the only way to achieve water repellent natural fibers.[2] 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. SiO₂) immobilization on fiber 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. Some of these non-wettable textiles have been successfully demonstrated in applications like filtration, oil-water separation and pattern able wetting .[3, 4]

MATERIALS AND METHOD

Materials

Cotton Fabrics

Plain woven and bleached 100% cotton fabric with 135 ± 5 g/m² mass density and having 56/cm warp and 40/cm weft threads was purchased from a local market.[5]

Silica Silica

Silicon dioxide, also known as silica, is an oxide of silicon with the chemical formula SiO₂, 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.

Density	2.648(α quartz), 2.196 (amorphous) $\text{g}\cdot\text{cm}^{-3}$
Melting point	1,713 °C (3,115 °F; 1,986 K) <i>Table Error! No text of specified style in document.-1</i>
Boiling point	2,950 °C (5,340 °F; 3,220 K)
Thermal conductivity	12(\parallel c-axis), 6.8 (c-axis), 1.4 (am.) $\text{W}/(\text{m}\cdot\text{K})$
Refractive index (nD)	1.544 (o), 1.553 (e)(p4.143)

Table 2 Properties of Silicon dioxide[5].

polyethylene glycol (PEG/4000)

is a polyether compound derived from petroleum with many applications, from industrial manufacturing to medicine. PEG is also known as **polyethylene oxide (PEO)** or **polyoxyethylene (POE)**, depending on its molecular weight. The structure of PEG is commonly expressed as $\text{H}-(\text{O}-\text{CH}_2-\text{CH}_2)_n-\text{OH}$. [6]

Ethanol (C₂H₅OH)

Ethanol (also called ethyl alcohol).

It is a simple alcohol with the chemical formula C₂H₆O. Its formula can be also written as CH₃-CH₂-OH or C₂H₅OH (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.[7]

Ammonium hydroxide (NH₄OH)

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.[8]

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.[9]

Methods

Preparation of silica nanoparticles

The spherical silica nanoparticles were prepared by Using Polmeel machine.

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

Resulting in powder spherical silica and tow scale.

150 nm and 300nm.



Figure 1: Polmeel machine

Preparation of PEG Solutions

Immersion PEG in water and heating at 150 °C

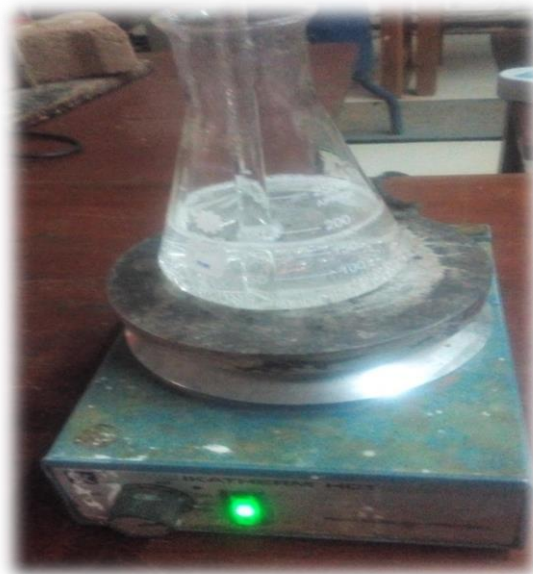


Figure 2: Heating Process

PEG Content

The PEG content of each sample calculated from equation 3-1

$$\text{PEG content (\%)} = \frac{\text{weight of the composite samples} - \text{weight of Pure fabric}}{\text{weight of the composite sample}} \times 100 \dots$$

Concentration contain

Concentration of a substance in a quantity solute present in given quantity of solution.

Measurement of concentration using Presley device.



Figure 3 Presley device

Solution B1:

Concentration=18.5%

Sediment =1.136033

Solution B2:

Solution Preparation

Sample	H ₂ O(ml)	SiO ₂ (g)	NH ₃ OH(ml)	EtOH(ml)	PEG(ml)
Sol1	70	57	10	170.8	63
Sol2	70	57	15	170.8	63

Table 2 Components and compositions for preparing silica nanoparticles.

The mixtures were stirred continually using magnetic stirrer at room temperature 30 °C for 1 h. Speed of magnetic stirrer 3507 r.p.m



Figure 4: Magnetic stirrer

Preparation of super hydrophobic cotton fabrics

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

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

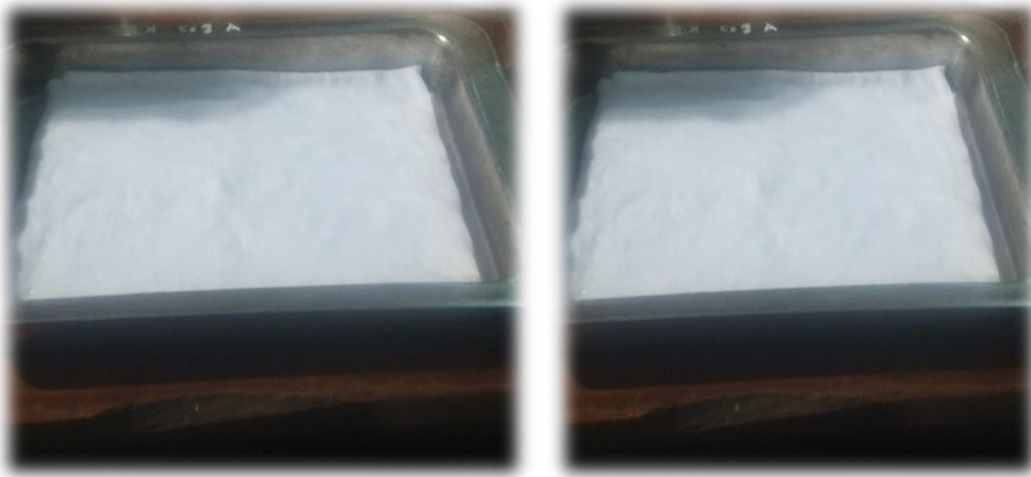
-The fabrics were then dried at 80°C for 3 min and cured at 160°C for 3 min with a preheated 50°C for 30 min to immobilize silica nano-particles on the cotton fabric.

-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 at 180°C for 3 h.



(B1)

(B2)

Figure 5: Cotton fabrics were immersed in a solution of silica nanoparticlesat/PEG

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 time



Figure 6: Drying process and curing

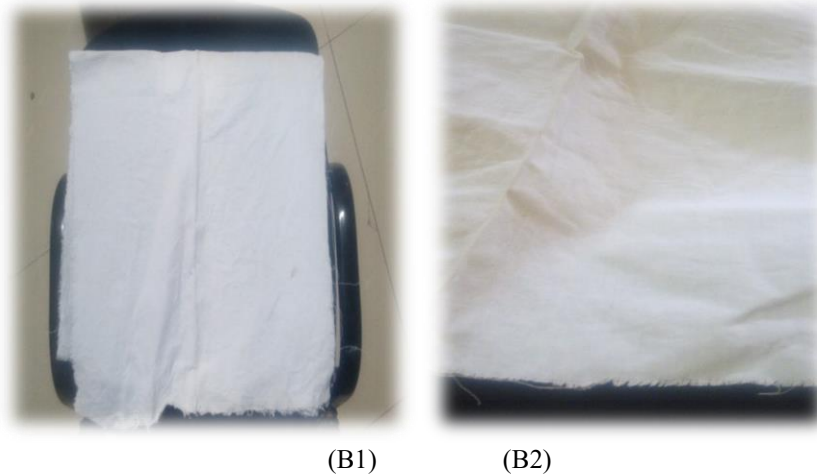


Figure 7: Fabric after treatment sample

Characterization

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. Irrespective if you are a quilter or a simple buyer, knowing to identify fabrics is truly important since that can help you charting out proper care instructions.[10, 11]

Fourier Transform Infrared (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.[12] FTIR mode was preferred since it allows the chemical analysis of the surface enabling a better characterization of the different coatings employed.[12] In FTIR spectrometer the calculated penetration depth of FTIR into the sample varies between $\sim 0.55 \mu\text{m}$ at 4000 cm^{-1} and $\sim 3.30 \mu\text{m}$ at 400 cm^{-1} . [13]

5 Resistance to water penetration

- 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 $20 \times 20 \text{ cm}^2$ dimension

Results and discussion

Experimental analysis

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

Characterization of the synthesized silicon nanoparticles using FTIR.

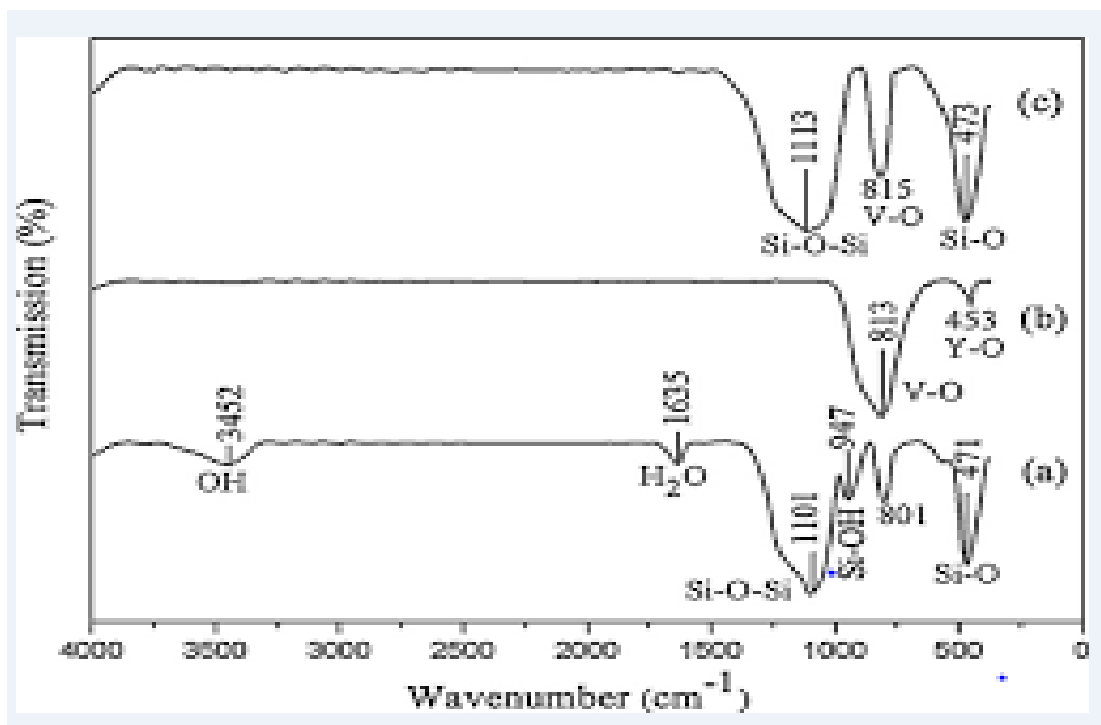


Figure 8: FTIR spectra of untreated cotton fabric and cotton fabrics assembled with (PEG/SiO₂)_n mu. FTIR analysis also gives a set of peak values unique for the sample along with information of the plant peptides that are present in the sample as the plant extract acts as a reducing agent figure. FTIR analysis is used to confirm the presence of plant peptides visible due to the bending produced by amide bonds.

Resistance to water penetration

For theoretical estimation of needed for penetration of water into waterproof porous structure *Table*

<i>A</i>	<i>W2</i>	<i>W1</i>	<i>W</i>	<i>B1</i>	<i>W2</i>	<i>W1</i>	<i>W</i>	<i>B2</i>	<i>W2</i>	<i>W1</i>	<i>W</i>
1	74	86	11	1	79.6	84	4.4	1	82.3	84.3	2.5
2	74.3	85	10.7	2	81.9	83	1.1	2	79	84	5
3	75.9	85	9.1	3	81	83	1.6	3	81	84.7	3.7

Table 4-3 compared the water penetration of the treated and untreated fabric.

To verify above theoretical values hydrostatic head experiments were performed for all treated samples including B1 and B2 (*Table 4-3*). Untreated fabric (A) being highly hydrophilic did not impregnate any water allowing continuous.

Conclusions

Synthesized silica nanoparticles via the sol-gel process and prepared super 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 NH₄OH catalyst.

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