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SYNTHESIS AND APPLICATION OF SILICA NANOPARTICLES ON POLYESTER BLENDED FABRIC TO IMPART SUPERHYDROPHOBICITY

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Abstract: In this study the P/C (67/33) and P/V (48/52) fabrics were treated with the silica nanoparticles and commercially available water repellent agent. The silica nanoparticles were synthesized by Stober method. The characterization of silica nanoparticles on the surface of both fabrics were done by using scanning electron microscope (SEM). The properties of P/C and P/V fabrics treated with the water repellent agent were compared with fabrics (P/C and P/V) finished with a combination of silica nanoparticles and water repellent agent. It was observed that the fabrics treated with water repellent agent showed the water repellency rating of 90 at 25gpl for P/C and for 15 gpl for P/V, while the rating of 90 was observed at 15 gpl of water repellent agent concentration when the P/C and P/V both fabrics were treated with the combination of silica nanoparticles and water repellent agent.

Keywords: Nanoparticles, Silica Nanoparticles, Superhydrophobicity, Textiles

I. INTRODUCTION

Water repellent textiles, by definition, repel water from the surface of the fabric. However, there are multiple methods by which water can be repelled from the fabric surface. A fabric surface can repel water by resisting adsorption, absorption, or penetration of water.

For many applications the ability of a finished fabric to exhibit water repellent is essential and potential applications of highly water repellent textile materials include rainwear, upholstery, protective clothing, sportswear, and automotive interior fabrics.
Superhydrophobic surfaces exist widely in nature. The lotus effect refers to the very high water repellence exhibited by the leaves of the lotus flower. Due to the invention of scanning electron microscopy today scientists becomes well known about the ability to repel the water results from superhydrophobicity, due to the combination of micrometer-scale hills and valleys and nanometre-scale waxy bumps, in combination with the reduced adhesion between surfaces and particles. The leaves of lotus have epidermal cells on their rough surface covered with wax crystals. The wax crystals provide a hydrophobic layer and the double-size structure gives the surface high roughness.

Rapidly emerging nanotechnology offers new and improved ways of imparting a range of functional performance properties of textile fabrics. Now a day's fact is that textile industry is the first manufacturing industry to come up with finished products that are enhanced through nanotechnology-based functional finishing. Nanotechnology also has revealed commercial potential for the textile industry. This is mainly due to the fact that conventional methods used to impart different properties to fabrics often do not lead to permanent effects, and will lose their functions after laundering or wearing. In contrast, nanotechnology can provide high durability for fabrics, because the nano-particles have a large surface area-to-volume ratio and high surface energy, thus presenting better affinity for fabrics and leading to an increase in durability of the function. A coating of nano-particles on fabrics will not affect physical and mechanical properties such as hand, strength, air permeability and wetting .Textile industry is the only major manufacturing industry that markets a range of end products with a nanotechnology label attached to them.

Recent research indicates that such applications may benefit from a new generation of water repellent materials that make use of the "lotus effect" to provide ultrahydrophobic textile materials. Ultrahydrophobic surfaces are typically termed as the surfaces that show a water contact angle greater than 150° with very low contact angle hysteresis. In the case of textile materials, the level of hydrophobicity is often determined by measuring the static water contact angle

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only, since it is difficult to measure the contact angle hysteresis on a textile fabric because of the high levels of roughness inherent in textile structures. Reduced contact area between the substrate and the liquid and vice versa for hydrophilic substrates.

II. PROCEDURE

A.Fabric

Type	**P/C (67/33)**	**P/V (48/52)**
Weave	Plain	Plain
Warp Count	53.96 Ne	46.69 Ne
Weft Count	49.50 Ne	41.89 Ne
EPI X PPI	108 X 75	92 X 72
GSM (g/m^2)	75.1	107

B. Chemicals

Tetraethyl orthosilicate [Si $(OC_2H_5)_4$ (TEOS)] procured from Tritech Catalyst (Pune), ethanol, ammonia solution (Laboratory Reagents) were used for the synthesis of silica nanoparticles. The distilled water was used for the synthesis of silica nanoparticles. The commercially available water repellent agent TUBIGUARD 270 L procured from CHT India Pvt. Ltd.

C. Characterization

The surface morphology of P/C and P/V fabrics treated with silica nanoparticles and combination of silica nanoparticles & water repellent agent was recorded on a Scanning Electron Microscope (SEM) (JEOL-JSN-6360). The water repellency and hydrostatic head pressure properties of treated fabrics were measured according to AATCC-22 & AATCC-127 test methods respectively. The air permeability and tearing strength properties of treated fabrics were measured according to ASTM B-737 & IS 6359:1971SP-15 respectively.

D. Synthesis of Silica Nanoparticles

The silica nanoparticles were synthesized by the alkaline hydrolysis of the tetraethyl orthosilicate followed by the dehydration, condensation reaction.

The mixture of 0.2 Mol tetraethyl orthosilicate and 2.5 Mol ethanol was prepared, then it was mixed with the mixture of 2.0 Mol distilled water, 2.5 Mol ethanol and 0.05 Mol ammonia solution. The synthesis was carried out for 3 hours at 30^0C.

E. Application of Silica Nanoparticles & the Water Repellent Agent on P/C and P/V fabrics

The previously desized, scoured & bleached P/C and P/V fabrics were padded with silica sol. The padded fabric samples were then dried at 110 ^{0}C. The dried fabric samples were cured at 150 ^{0}C for 2 mins. Then these fabric samples were again padded with the water repellent agent with varying concentration from 15gpl to 40gpl. The padded fabric samples were then dried at 110^OC and cured at 150 ^{0}C for 2 mins. Table 1 shows the composition of chemicals for the synthesis of silica nanoparticles & the table 2 shows the composition of water repellent agent and silica nanoparticles for water repellent finish as follows.

Table 1. Composition of Chemicals for the Synthesis of Silica Nanoparticles

Sr. No.	**TEOS (Mol)**	**Ethanol (Mol)**	**Water (Mol)**	**Ethanol (Mol)**	**Ammonium Hydroxide**
1	0.2	2.5	2.0	2.5	0.05

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Table 2. Composition of water repellent agent and silica nanoparticles for water repellent finish

Sr. No.	TEOS (Mol)	Ethanol (Mol)	Water (Mol)	Ethanol (Mol)	Amm. Hydroxide (Mol)	Water repellent Agent (gpl)
1	0.2	2.5	2.0	2.5	0.05	15
2	0.2	2.5	2.0	2.5	0.05	20
3	0.2	2.5	2.0	2.5	0.05	25
4	0.2	2.5	2.0	2.5	0.05	30
5	0.2	2.5	2.0	2.5	0.05	35
6	0.2	2.5	2.0	2.5	0.05	40

III. RESULTS AND DISCUSSION

A. Synthesis of Silica Nanoparticles.

Silica nanoparticles were prepared using Stober method including the hydrolysis of TEOS and the condensation of the hydrolysed silica species in the presence of an ammonia catalyst.

The reactions were as follows.

a) Hydrolysis

$Si\text{-}[OC_2H_5]_4 + 4\,H_2O \rightarrow Si\text{-}(OH)_4 + 4\,C_2H_5OH$

b) Alcohol condensation

$Si\text{-}(OH)_4 + Si\text{-}[OC_2H_5]_4 \rightarrow\ \equiv Si\text{-}o\text{-}Si \equiv + 4\,C_2H_5OH$

c) Water condensation

$Si\text{-}(OH)_4 + Si\text{-}(OH)_4 \rightarrow\ \equiv Si\text{-}o\text{-}Si \equiv + 4H_2O$

B. Effect of Concentration on Water Repellency (Spray Test)

Table 3. Effect of concentration on Water Repellency for P/C fabric

Conc. (gpl)	Spray Test Rating	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent only
15	90	80
20	90	80
25	90	90
30	90	90
35	90	90
40	90	90

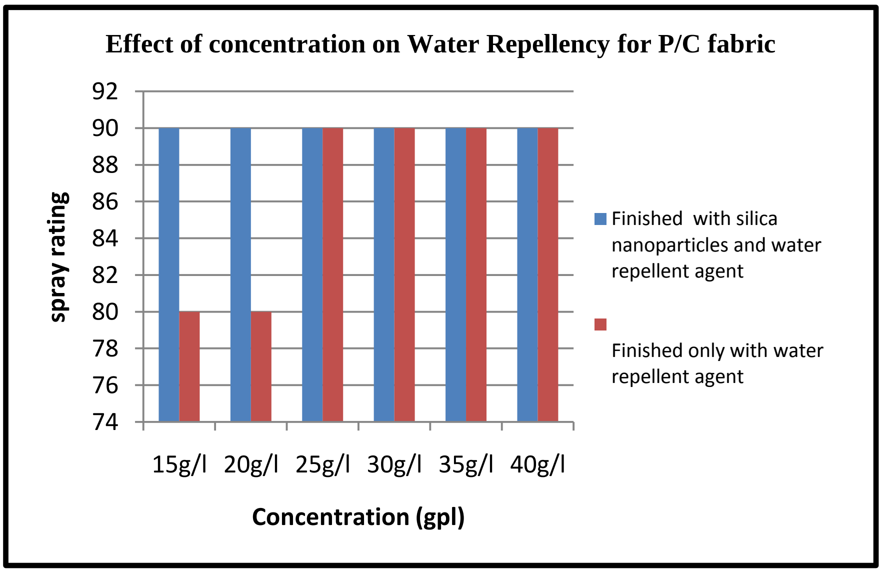

Figure 1. Effect of concentration on Water Repellency for P/C fabric

From the Table 3 and Figure 1, it is observed that, as the concentration of water repellent agent increases from 15 gpl to 40 gpl the fabric finished with only water repellent agent shows the spray rating of 80 at 15 gpl concentrations and a spray rating of 90 at 25 gpl concentrations of water repellent agent, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows the spray rating of 90 at 15 gpl concentrations of water repellent agent.

Table 4. Effect of concentration on Water Repellency for P/V fabric

Conc. (gpl)	**Spray Test Rating**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent only
15	90	90
20	90	90
25	90	90
30	100	90
35	100	90
40	100	90

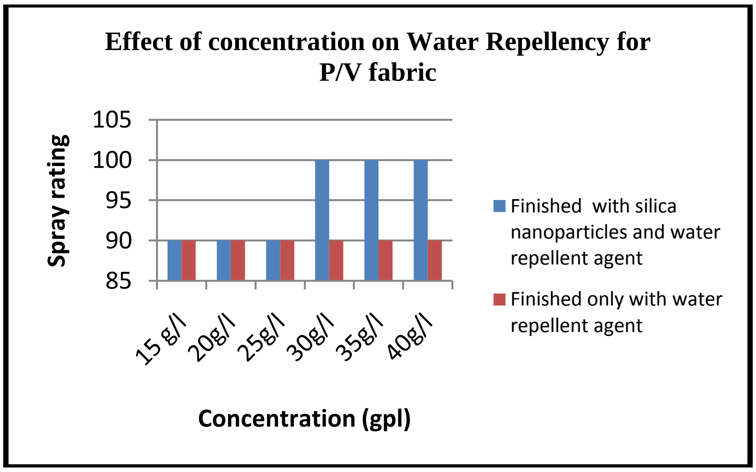

Figure 2. Effect of concentration on Water Repellency for P/V fabric

From the above Table 4 and Figure 2 it is observed that, as the concentration of water repellent agent increases from 15 gpl to 40 gpl the fabric finished with only water repellent agent shows the spray rating of 90 at 15 gpl concentrations of water repellent agent, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows the spray rating of 100 at 30 gpl concentrations of water repellent agent.

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C. Effect of Concentration on Hydrostatic Head Pressure.

Table 5. Effect of Concentration on Hydrostatic Head Pressure for P/C fabric

Conc. (gpl)	**Hydrostatic Head Pressure (cubic c.m.)**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent
15	5.5	5.0
20	6.5	6.0
25	7.5	7.0
30	7.5	7.5
35	8.5	8.0
40	8.5	8.5

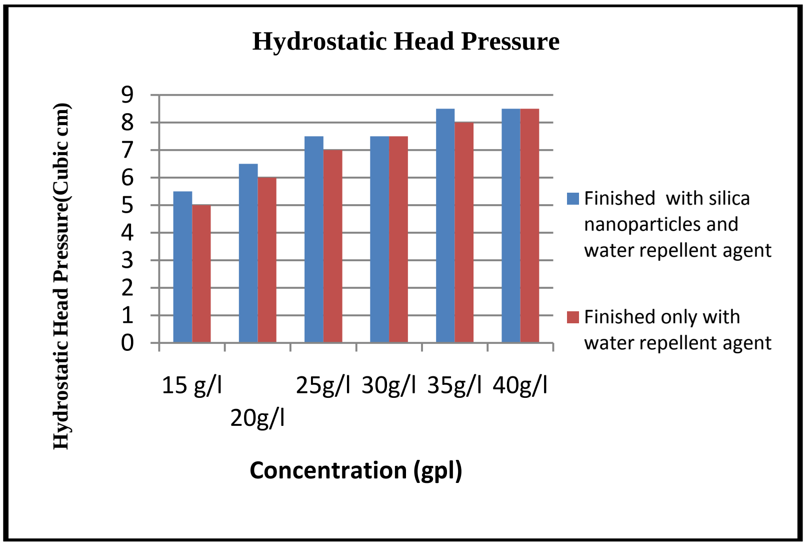

Figure 3. Effect of concentration on hydrostatic head pressure for P/C fabric

The Table 5 and Figure 3 shows that, the hydrostatic head pressure of the fabric (P/C) treated with a combination of silica nanoparticles and water repellent agent and the fabric treated with the only water repellent agent. The fabric finished with only water repellent agent shows the increase in hydrostatic head pressure from 5.0 to 8.5 for water repellent agent concentration of 15 gpl to 40 gpl respectively. The fabric finished with a combination of silica nanoparticles and water repellent agent shows an increase in hydrostatic head pressure from 5.5 to 8.5 for water repellent agent concentration of 15 gpl to 40 gpl respectively. This may be because of silica nanoparticles helps to resist the drop of water penetrating inside the fabric structure.

Table 6. Effect of concentration on Hydrostatic Head Pressure for P/V fabric

Conc. (gpl)	**Hydrostatic Head Pressure (cubic c.m.)**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent
15	11.5	8.5
20	12	8.5
25	12	9.0
30	12.5	9.5
35	13.5	11.5
40	14	12.0

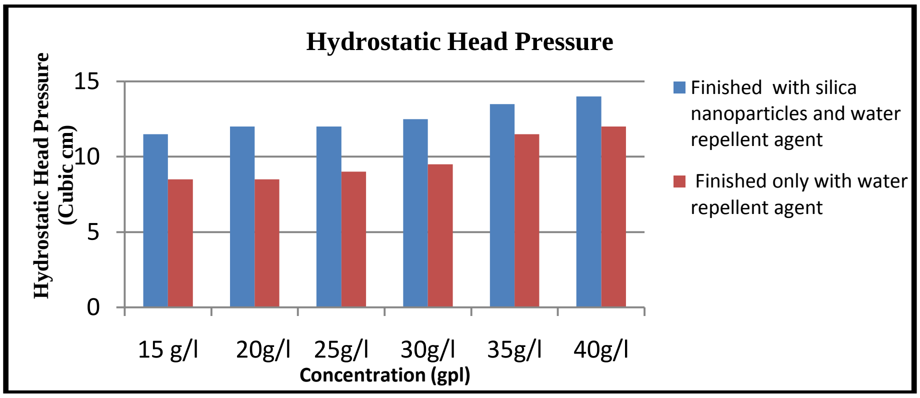

Figure 4. Effect of concentration of on hydrostatic head pressure for P/V fabric

The Table 6 and Figure 4 shows that, the hydrostatic head pressure to the fabric (P/V) treated with a combination of silica nanoparticles and water repellent agent and the fabric treated with the only water repellent agent. The fabric finished with only water repellent agent shows the increase in hydrostatic head pressure from 8.5 to 12 for water repellent agent concentration of 15 gpl to 40 gpl respectively. The fabric finished with a combination of silica nanoparticles and water repellent agent shows an increase in hydrostatic head pressure from 11.5 to 14 for water repellent agent concentration of 15 gpl to 40 gpl respectively. This may be because of silica nanoparticles helps to resist the drop of water penetrating inside the fabric structure.

D. Effect of Concentration of Water Repellent Agent on Air Permeability.

Table7. Effect of concentration of water repellent agent on air permeability for P/C fabric

Conc. (gpl)	**Air Permeability ($Cm^3/Cm^2/Sec$)**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent
15	109.85	112.75
20	108.63	110.30
25	107.54	109.44
30	106.52	107.84
35	105.96	106.42
40	103.89	105.20

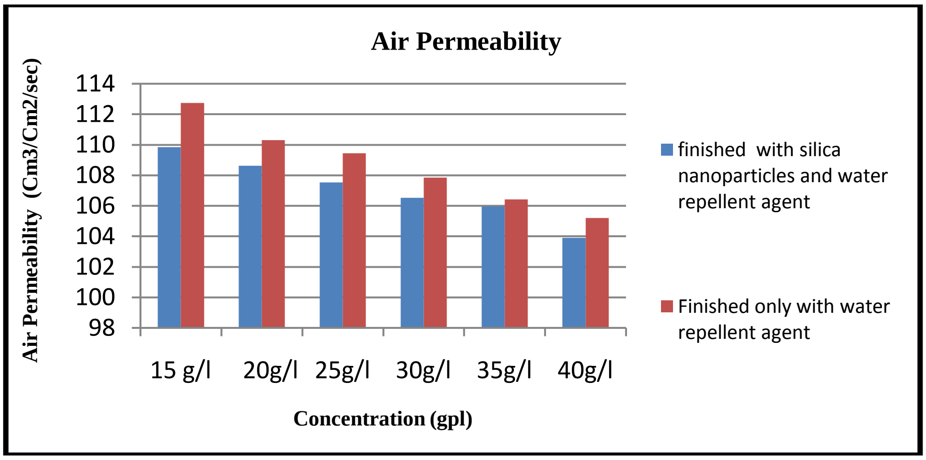

Figure 5 . Effect of concentration of water repellent agent on air permeability for P/C fabric

From the Table 7 and Figure 5, it is observed that, the fabric (P/C) finished with only water repellent agent shows the decrease in air permeability from 112.75 to 105.20 for the concentration of 15 gpl to 40 gpl of water repellent agent respectively, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows decrease in air permeability from 109.85 to 103.89 for the concentration of 15 gpl to 40 gpl of water repellent agent respectively. This may be because as the concentration goes on increasing the film is going to become continuous,

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which causes decrease in air transmission rate & also the silica nanoparticles help in filling the interstices between fibres.

Table 8. Effect of concentration of water repellent agent on air permeability for P/V fabric

Conc. (gpl)	**Air Permeability ($Cm^3/Cm^2/Sec$)**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent
15	27.04	29.42
20	25.45	29.38
25	22.59	28.96
30	20.00	27.90
35	19.88	27.26
40	18.41	26.96

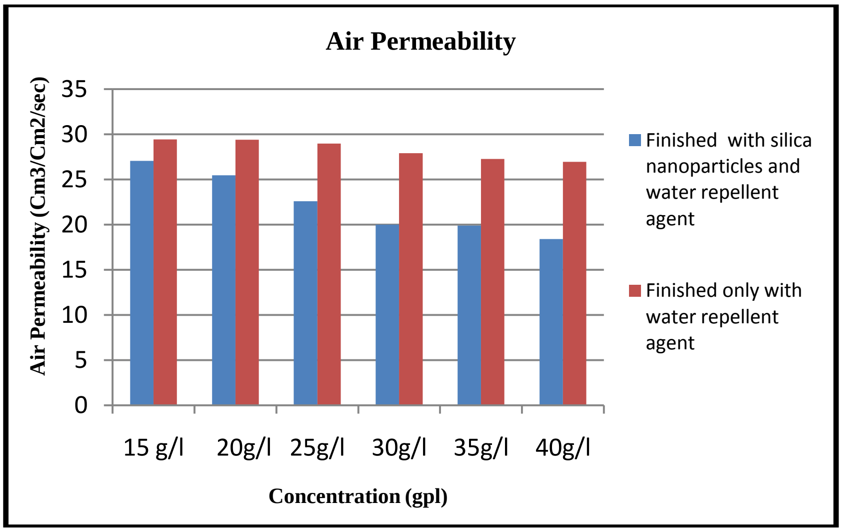

Figure 6. Effect of concentration of water repellent agent on air permeability for P/V fabric

From the Table 8 and Figure 6, it is observed that, the fabric (P/V) finished with only water repellent agent shows the decrease in air permeability from 29.42 to 26.96 for the concentration of 15 gpl to 40 gpl of water repellent agent respectively, while thefabric finished with a combination of silica nanoparticles and water repellent agent shows decrease in air permeability from 27.04 to 18.41 for the concentration of 15 gpl to 40 gpl of water repellent agent respectively. This may be because; as the concentration goes on increasing the film is going to become continuous, which causes decrease in air transmission rate. & also the silica nanoparticles help in filling the interstices between fibres.

E. Effect of Concentration of Water Repellent Agent on Tearing Strength

Table 9. Effect of concentration of water repellent agent on Tearing strength for P/C fabric

Conc. (gpl)	**Tearing Strength (gmf)**			
	Warp way		**Weft way**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent
15	1780.06	1790.22	1469.12	1482.51
20	1654.83	1767.15	1439.16	1460.62
25	1532.26	1701.12	1295.21	1400.13
30	1525.85	1640.16	1280.01	1380.17
35	1515.42	1582.21	1270.16	1370.25
40	1497.23	1536.92	1260.23	1350.86

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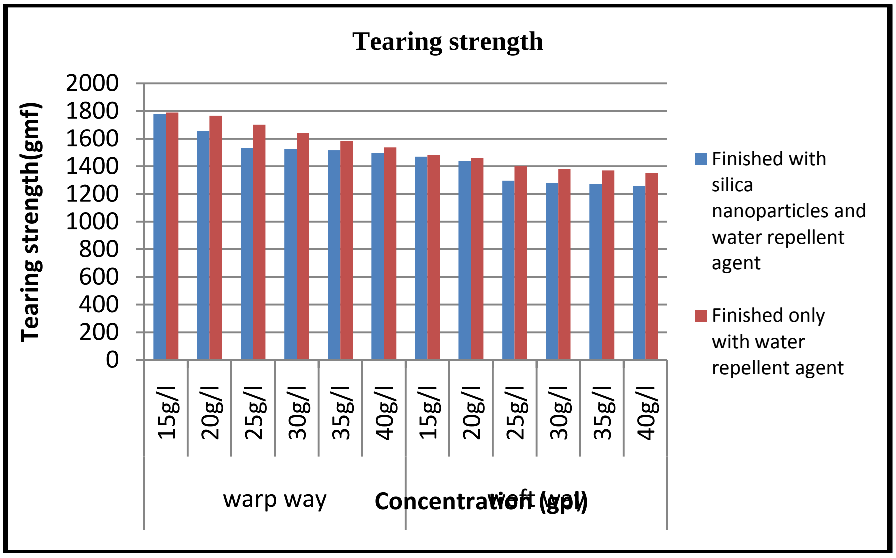

Figure 7. Effect of concentration of water repellent agent on tearing strength for P/C fabric

The above Table 9 and Figure 7 shows the tearing strength in both warp and weft way for the fabric (P/C) treated with a combination of silica nanoparticles and water repellent agent and the fabric treated with the only water repellent agent. It is observed that there is a decrease in tearing strength in warp direction from 1790.22 to 1536.92 for water repellent agent concentration of 15 gpl to 40 gpl respectively, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows the decrease in tearing strength in warp direction from 1780.06 to 1497.23 for water repellent agent concentration of 15 gpl to 40 gpl respectively. The fabric finished with only water repellent agent shows the decrease in tearing strength in weft direction from 1482.51 to 1350.86 for water repellent agent concentration of 15 gpl to 40 gpl respectively, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows the decrease in tearing strength in weft direction from 1469.12 to 1260.23 for water repellent agent concentration of 15 gpl to 40 gpl respectively. This indicates that an increase in concentration of water repellent agent along with silica nanoparticles causes an increase in the resistance for the slippage of fibres in both warp and weft direction.

Table 10. Effect of concentration of water repellent agent on tearing strength for P/V fabric

Conc. (gpl)	**Tearing Strength (gmf)**			
	Warp way		**Weft way**	
	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent	Finished with silica nano particles along with water repellent agent	Finished with water repellent agent
15	1782.52	1892.15	1678.77	1701.02
20	1750.95	1872.10	1645.54	1682.80
25	1739.03	1850.01	1630.13	1670.19
30	1672.52	1831.12	1615.15	1661.16
35	1646.61	1700.19	1600.09	1651.22
40	1597.98	1685.80	1587.01	1637.32

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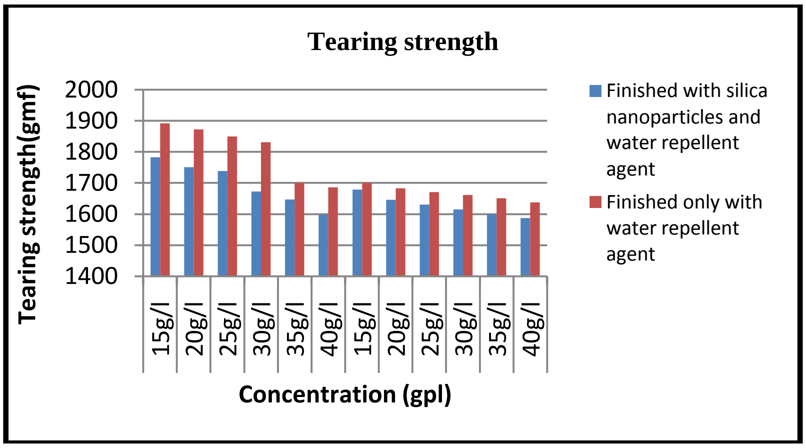

Figure 8. Effect of concentration of water repellent agent on tearing strength for P/V fabric

The above Table 10 and Figure 8 shows the tearing strength in both warp and weft way for the fabric (P/V) treated with a combination of silica nanoparticles and water repellent agent and the fabric treated with the only water repellent agent. It is observed that there is a decrease in tearing strength in warp direction from 1892.15 to 1685.80 for water repellent agent concentration of 15 gpl to 40 gpl respectively, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows the decrease in tearing strength in warp direction from 1782.52 to 1597.98 for water repellent agent concentration of 15 gpl to 40 gpl respectively. The fabric finished with only water repellent agent shows the decrease in tearing strength in weft direction from 1701.02 to 1637.32 for water repellent agent concentration of 15 gpl to 40 gpl respectively, while the fabric finished with a combination of silica nanoparticles and water repellent agent shows the decrease in tearing strength in weft direction from 1678.77 to 1587.01 for water repellent agent concentration of 15 gpl to 40 gpl respectively. This indicates that an increase in concentration of water repellent agent along with silica nanoparticles causes an increase in the resistance for the slippage of fibres in both warp and weft direction.

F. Surface Morphology

The surface morphology was characterized by using Scanning Electron Microscope (SEM). It was observed that, the P/C and P/V treated with silica nanoparticles shows the scaly appearance on the surface, which made the surface rougher & enhance the water repellency. The P/C and P/V fabrics treated with 25gpl and 15gpl concentration of both silica nanoparticles and water repellent agent shows the water repellency. The water repellent agent forms an oily layer on the silica nanoparticles just like lotus leaves. Here silica nanoparticles act as an epidermal cells and water repellent agent as waxy bumps which results in superhydrophobicity on the P/C and P/V fabrics. Figure 9 shows, the SEM image of P/C fabric treated with a combination of silica nanoparticles and water repellent agent & figure 10 shows, the SEM image of P/V fabric treated with a combination of silica nanoparticles and water repellent agent.

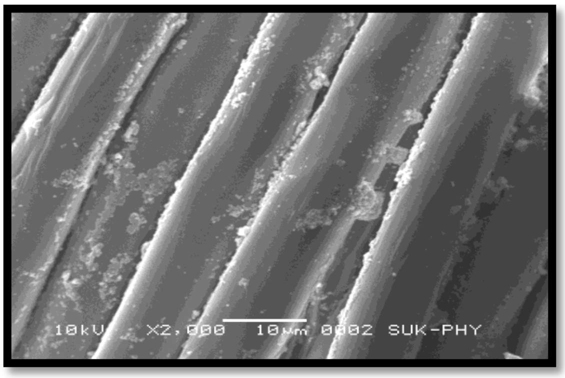

Figure 9. SEM image of P/C fabric treated with a combination of silica nanoparticles and water repellent agent at a concentration of 25 gpl

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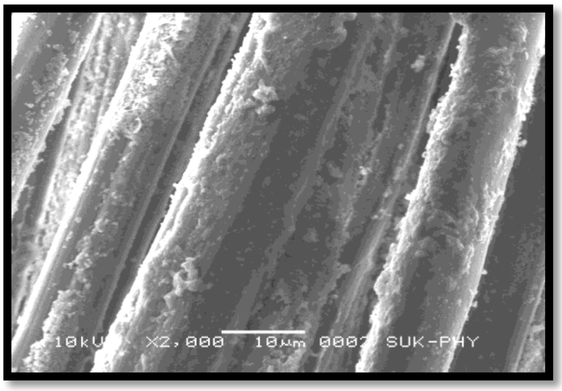

Figure 10. SEM image of P/V fabric treated with a combination of silica nanoparticles and water repellent agent at a concentration of 15gpl

IV. CONCLUSION

From the above results and discussion, it can be concluded that, as far as SEM results are concerned P/V being more hydrophobic shows more deposition of silica nanoparticles as against less deposition on P/C fabric, it means P/V may be converted into highly hydrophobic in nature.

When compared with water repellent agent and combination of silica nanoparticles with water repellent agent, the combination of silica nanoparticles and water repellent agent shows excellent results of hydrophobicity.

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