FABRICATION OF SUPERHYDROPHOBIC SURFACE WITH SILICA/PDMS NANOCOMPOSITE USING DIP COATING

by

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A Research Study Submitted in Partial Fulfilment of the Requirements for the Degree of Master of Engineering in Nanotechnology

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AUTHOR'S DECLARATION

I, Rahul Guptha Myadam, declare that the research work carried out for this research was in accordance with the regulations of the Asian Institute of Technology. The work presented in it, is my own and has been generated by me as the result of my own original research, and if external sources were used, such sources have been cited. It is original and has not been submitted to any other institution to obtain another degree or qualification. This is a true copy of the research, including final revisions.

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ABSTRACT

Silica (SiO_2) is one of the very interesting inorganic compound materials which have unique physio-chemical properties and wide industrial applications. In this research we used silica nanoparticles surface modified with hydrophobic functional groups to prepare a nanocomposite using polydimethylsiloxane (PDMS) as matrix and use the nanocomposite to fabricate superhydrophobic surface using dip coating technique. The silica nanoparticles, silica/PDMS nanocomposite and the superhydrophobic surfaces are characterized by electron microscopy to study their morphology. Water contact angle on the superhydrophobic surface is studied by analysing surface wettability measurement methods. Static and Sliding water contact angles are investigated for different concentrations of silica nanoparticles in the nanocomposite. A water contact angle of 154° is obtained for silica/PDMS nanocomposite film which is almost 28% higher than the plain PDMS indicating improved hydrophobicity of the PDMS layer.

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LIST OF ABBREVIATIONS

SiO_2	= Silicon Dioxide
PDMS	= Polydimethyl Siloxane
KH-550	= 3-Aminopropyltriethoxysilane
KH -570	= 3-Methacryloxypropyltrimethoxysilane
WCA	= Water Contact Angle

CHAPTER 1
INTRODUCTION

1.1 Background of the Study

Polydimethylsiloxane (PDMS) is one of the organosilicon compounds that are commonly known as silicones. It is the broadly used silicon-based organic polymer, because of its versatility and properties. This led to its usage in various applications, such as electronics, automobile, medical devices, microfluidic systems, and various household applications. The chemical formula for PDMS is $CH_3[Si(CH_3)_2O]_n\ Si(CH_3)_3$, where n represents the number of repetitive monomer units, having molecular formula: $(C_2H_6OSi)_n$. Because of its transparency, flexibility, bio compatibility, chemical inertness, and ease of handling, PDMS is a common choice for many applications.

Silica (SiO_2) is the base material of silicones that from where it was originally derived. Silica is nothing but glass and it also has wide industrial applications across the world. Silica chemistry is well-studies throughout the history and has been used widely. Nanoparticles of silica is also getting tremendous public interest in the recent times because of their tunable properties, stability, and ease of synthesis. Some studies published recently showed silica/PDMS mixture to modify the wettability of the nanocomposite surface to make them more hydrophobic and durable(Chang et al., 2015). Such surfaces provide less drag resistance and self-cleaning properties which is desired for many applications.

1.2 Statement of the Problem

Although PDMS surface is hydrophobic and it can produce decent amount of hydrophobicity to keep a surface dry, it is still not adequate to obtain super hydrophobicity with pure PDMS. Therefore, various additives are used to further modify PDMS to obtain super hydrophobicity. Most of these additives are organic compounds that either show short-term stability or less compatibility with PDMS. Sometimes they are also toxic to the environment. To avoid such issues, silica nanoparticles are adopted due to their stability against various natural and chemical factors. In addition to that, silica is the base material from where PDMS is prepared and therefore its compatibility with PDMS is very high.

However, silica surface is hydrophilic, and they are therefore difficult to mix in PDMS to get a uniform mixture. Hence, additional surface modification of silica nanoparticles is required to functionalize them with hydrophobic functional groups to get a uniform silica/PDMS nanocomposite. Techniques applied to develop silica/PDMS nanocomposite films can also be hurdle for large scale production of these superhydrophobic surfaces. Most of the techniques developed in the lab, e.g. spin coating or drop-casting techniques, are suitable for small scale coating and sometimes required sophisticated equipment.

Spray coating is a solution to obtain large area coating in short time and this technique has been adopted in industries also due to its simplicity. However, spray coating produces large amount of waste which is not desired by the industries. To address this, we propose to explore the dip coating technique which has the potential for large area coating with high yield, and the technique does not produce large amounts of waste and requires less maintenance.

1.3 Objectives

The primary objective of this research is to develop a silica/PDMS nanocomposite and fabricate a superhydrophobic surface using the nanocomposite. In order to attain the primary objective, following specific objectives are planned:

1. Prepare silica/PDMS nanocomposite using silica nanoparticles modified with silane coupling agents.
2. Prepare silica/PDMS nanocomposite thin films on glass substrates using dip coating method.
3. Investigate surface wettability of the silica/PDMS nanocomposite thin film and compare water contact angle for different concentrations of silica nanoparticles in the silica/PDMS nanocomposite.

1.4 Scope

This research study involves preparation of silica/PDMS nanocomposite thin films using dip coating technique. The nanocomposite will be prepared by using simply mixing technique using surface modified silica nanoparticles with PDMS matrix.

Thin films will be Prepared on Glass Substrate and Dip Coating technique will be used to prepare the thin films. Water contact angle will be analysed using sessile drop method.

The Study will Investigate the effect of amount of silica nanoparticles in the silica/PDMS nanocomposite on the surface wettability of the thin films. Surface wettability will be investigated using water as the solvent.

1.5 Report Outline

Chapter 1- Introduction to the research field

Chapter 2- Literature review on relevant systems, academic projects, and commercial projects

Chapter 3- Description about the methodology used in the research study

Chapter 4- Description of results obtained and their discussion

Chapter 5- Conclusions and future recommendations

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

Super hydrophobic surfaces have recently grabbed the attention of researcher's for its unique properties like anti-contamination and self-cleaning (M. Zhang et al., 2016). However, the study of artificial superhydrophobic surface has been inspired after the deep analysis of lotus leaf surface which is the excellent example of super hydrophobic surface given to the mankind by the nature.

Some recent reviews have described that there is a fast advancement in preparation of superhydrophobic surfaces due to its interesting, phenomenal properties and functional applications. different deposition techniques will affects the influence of the relative roughness of the surface and its wettability nature, which will be explained in detail in the following sections of the report(Li et al., 2007). The structures of 0-Dimensional and 1-Dimensional geometric in the range of micrometre to nanometre scale and their chemical composition will influence the hydrophobic nature of solid surfaces. Therefore, Super hydrophobic surfaces can be fabricated by the superficial modification of surface roughness, having lower surface energy.

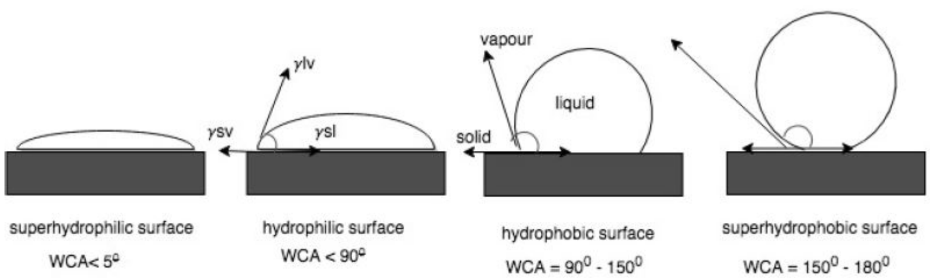
Compounded materials with polymeric matrix and ceramics as filler material have been modified and enhanced by the current research going on. This attributed to the development of nano particle's functioning process for super hydrophobic applications. The coating techniques i.e., spin or dip coatings have been used, further, for the deposition of coatings with varied properties, due to the improvement in the dispersion of nano particles in the given matrix.

2.2 Surface Wettability

Surface regime is nothing but the classification of surface, this surface classification is purely based on “Contact angle” that is the angle measured with the liquid, when a liquid-vapor interface meets a solid surface.

Figure 2.1

Surface Classification Based on Water Contact Angle



Note. Classification of Various Surfaces Based on Water Contact Angle.

The term hydrophilic represents water-loving nature. The surfaces having contact angle of 90° or less than 90° are termed as “hydrophilic” in nature. If the angle is less than 5°, then the surfaces were considered as super-hydrophilic. In similar, the term Hydrophobicity represents the property of surfaces to repel water(Huhtamäki et al., 2018). For this kind of surfaces, the contact angle varies from 90° to 150°. Such surfaces repel or reduce the interaction with the water droplet, to avoid to get wet. If the adhesive nature of surfaces towards liquid drop is superficial with the contact angle greater than 150°, they are treated as super hydrophobic. These coatings, at nano scale, are make the water droplets to roll off.

They maintain the interactions with water as minimum as possible. Superhydrophobic surfaces have the hysteresis of contact angle to be limited such that the sliding angle will be less than 10°. Lower surface energy and good surface roughness are the main criteria for the materials to attain super hydrophobicity.

2.3 Silicon Dioxide

SiO_2 is commonly used material for the fabrication of rough surfaces, which is one of the most important property of super hydrophobicity(Li et al., 2007). The synthesis of SiO2 nano particles, at 0D,1D,2D,3D level, can be done through the techniques such as Sol-Gel, Precipitation, Solvo-thermal and Hydrothermal approaches. The interest towards the effect of morphology on super hydrophobicity, made the researchers to use microwave irradiation method (Kang et al., 2016). As the SiO_2 is non-toxic in nature, it can be used in numerous industrial, cosmetic, and medical device applications.

SiO_2 nanoparticles are mostly known for their low cost, low environmental impact, and excellent photoelectric performance(Cai et al., 2018), SiO_2 can be synthesized using various methods, but Sol-gel technique, is the most widely followed route for the fabrication of hydrophobic and super hydrophobic surfaces(J. Zhang et al., 2018).

In this respect it is a very good strategy to modify the morphology of the outer most surface of the sol-gel route-based coating by using silane coupling agents with different alkyl chains. To achieve the better mechanical resistance, new trends are evolving based on the modification of sol-gel coatings with inorganic nanoparticles (Chang et al., 2015).

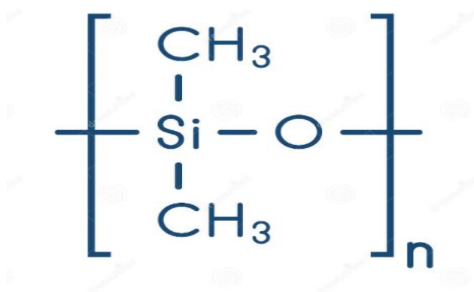
2.4 PDMS

To develop the composite material based coatings, Polydimethylsiloxane (PDMS) gives the better properties, as the ((Si-O-Si)n) main chain has higher bond angle and bond energy. These properties made the coating better in terms of elasticity, thermal stability and superhydrophobicity (Prertkaew et al., 2012). The dispersion and interaction of filler particles in the matrix of PDMS composites can be improved concern to the functionality of oxides, which is not available for the functionality to SiO_2.

The major aim of this research is to develop the nanocomposite with Functionalized SiO_2/PDMS for creating a super hydrophobic surface by the dip-coating technique to obtain the coatings and to contribute the advantages of functional SiO_2 nano fillers in PDMS polymer matrixx for self-cleaning applications.

Figure 2.2

Structure of Polydimethylsiloxane (PDMS)



Note. Structural Representation of PDMS.

2.4.1 Advantages of the Usage of PDMS in Super Hydrophobic Coatings

The value of surface tension for PDMS is as low as 20 mN/m, due to the presence of CH_3 groups on each of the silicon atom, and surface tension of PDMS is comparatively less than the PVDF (30.3mN/m) and PTFE (23.9mN/m). Multiscale structures can be easily developed by the presence of PDMS in the coatings. These coatings can be coated on the substrates using range of coating methods like dip coating, spin coating, and spray coating etc., to achieve super hydrophobicity (Wang et al., 2021).

PDMS coatings have longer durability because of their excellent wear, chemical resistance, and resilience and the cost of PDMS is very economical, comparatively, cheaper than the fluorinated compounds.

2.5 Various Coating Techniques for the Fabrication of Super Hydrophobic Surface

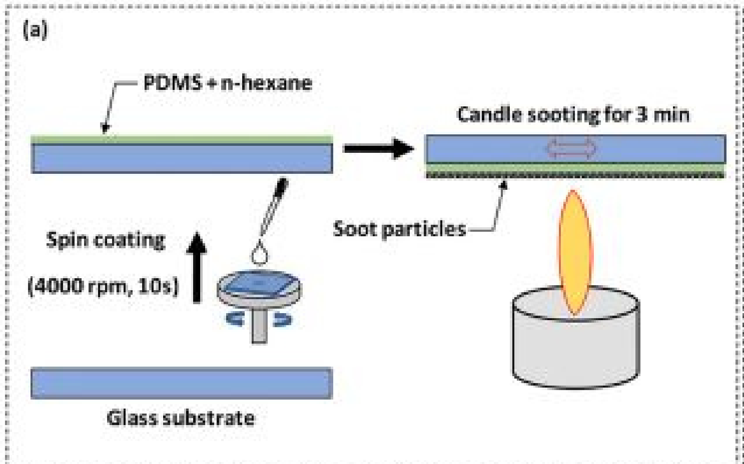
The fabrication of superhydrophobic surfaces can be done by different coating such as 1) Spin-coating 2) Dip-coating 3) Spray coating and etc. here in the following sections we discuss few of those coating techniques.

2.5.1 *Spin-Coating Technique*

It is the unique technique to coat thin films on the substrates with reproducibly uniform thickness over a bulk area having diameter greater than 30 cm.

Figure 2.3

A Schematic Diagram of Spin Coater Developing a Super Hydrophobic Surface with PDMS Using Solvent n-hexane(Wang et al., 2021)**.**



Note. Schematic Diagram of Spin Coater.

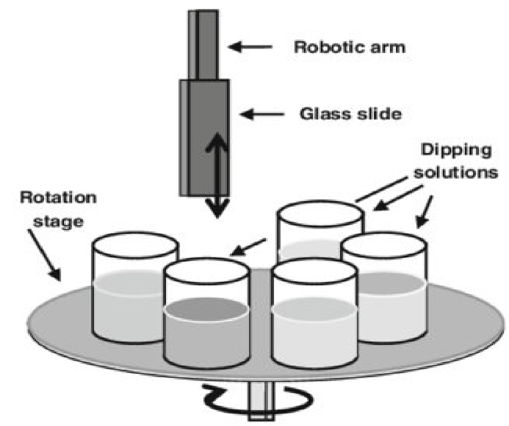
2.5.2 ***Dip-Coating Technique***

It is the most widely followed technique to fabricate the super hydrophobic coatings, since this technique is cost-economic, waste-free, and easily scalable, it gives controllable thickness. Using this technique, The substrates like cotton, glass, fabric, polyurethane sponge are fully dipped in the solution containing bare PDMS or PDMS with nanoparticles(Santiago et al., 2020). This leads to the formation of super hydrophobic coatings on the substrate when the substrate is lifted from the solution container and air-dried.

Dip-coating method is widely preferable for the fabrication of super hydrophobic surfaces. This method is also highly recommendable in modifying the planar sheets or meshes, and in 3D structural materials like cotton wools and sponges.

Figure 2.4

Schematic Diagram of Multi Vessel Dip Coater



Note. Schematic representation of Multi-Vessel Dip-coater System.

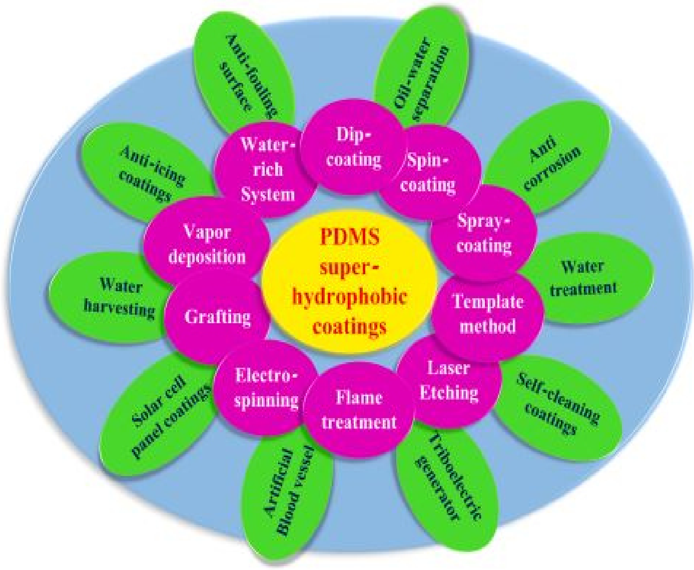
Table 2.1

Various Coating Techniques used for Super Hydrophobic Application

Coating Technique	Substrate	Nano Particles	Solvent	WCA
Dispersion Technique	Glass	ZnO, Stearic acid, Methyl polysiloxane, PDMS(Santiago et al., 2020)	Ethanol	160°
Drop Coating	Glass	1-dodecanethinol	Ethanol, n-hexane	159°
Vapor Deposition	Glass	APS, ZnO , PDMS	Ethanol, n-hexane	160°
Spin Coating	Glass	SiO_2 , PDMS ,DTS	n-hexane	161 °
Dip Coating	Glass	APTES, SiO_2, PDMS	Ethanol	145°

Figure 2.5

Various Preparation Techniques and Applications of Super Hydrophobic Coatings.



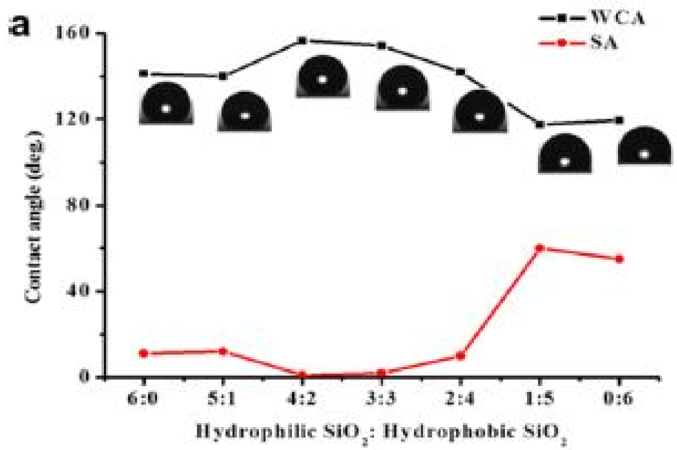
Note. Schematic representation of Superhydrophobic surface and its applications.

2.6 Super Hydrophobicity Using SiO_2

Xiao Gong, Shuang He has developed superhydrophobic coatings using spray-coating technique with combination of readily available silicon dioxide and polydimethylsiloxane (PDMS). In these coatings this group has used two types of silicon dioxide namely hydrophilic and hydrophobic silicon dioxide and they had coated on wide types of substrates like glass, paper and plastic and they achieved the contact angle of 156.4˚and sliding angle of less than 5˚.

Figure 2.6

Static Water Contact angle of Hydrophobic and Hydrophilic SiO_2 with Combination of PDMS



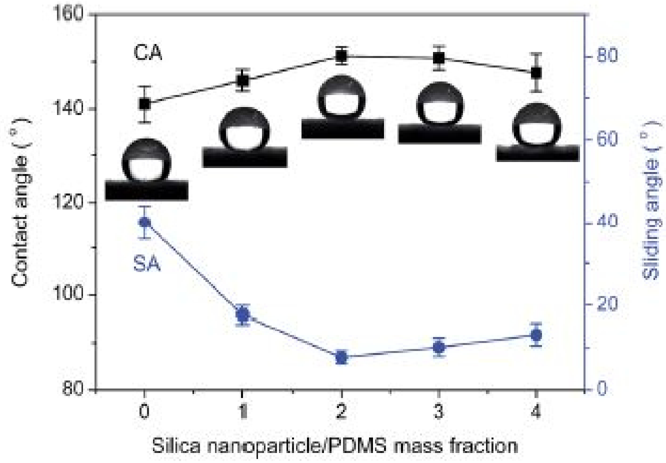
Note. Graphical representation of Static water contact angle of hydrophilic/hydrophobic SiO_2 .

2.7 Super Hydrophobicity of SiO_2 on Wooden Substrate

Chang, Kunkun Tu, wang and Jun liang has developed superhydrophobic coatings with combination of Silicon dioxide and PDMS on the wooden substrate using dip-coating technique. They achieved the Sliding angle of less than 10˚and contact angle of 152˚. The major aim of their research is to develop a durable super hydrophobic wood with self-cleaning properties(Chang et al., 2015).

Figure 2.7

Static and Dynamic Contact Angle of Wooden Substrate Developed with the Combination of SiO_2 and PDMS.



Note. Graphical Representation of Static and Dynamic Water Contact Angle of hydrophilic/hydrophobic SiO_2 and PDMS.

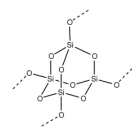
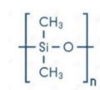
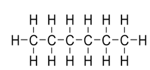
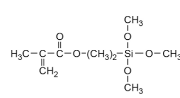
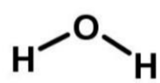
CHAPTER 3
METHODOLOGY

3.1 Methods and Materials

The primary aim of this research is to is to develop a silica/PDMS nanocomposite and fabricate a superhydrophobic surface using the nanocomposite. Table 3.1 below lists the basic materials that were used in this study.

Table 3.1

Material List of Chemical Compounds

Materials	Chemical Formula	Chemical Structure	What is it
Silicon Dioxide	SiO_2		Inorganic Compound
PDMS	$(C_2H_6OSi)_n$		Hydrophobic Constituent
Hexane	C_6H_{14}		Solvent
3-Methacryloxypropyl rimethoxysilane	$C_{10}H_{20}O_5Si$		Functional Agent
DI Water	H_2O		Works as Solvent

Note: This table explains about the list of materials that will be used to fabricate the silica/PDMS nanocomposite and the superhydrophobic surface using the nano composite.

3.2 Preparation of SILICA (SiO_2) Dispersion

In this study we plan to use commercially available silica nanoparticles functionalized with KH550 and KH570 silane coupling agents. These silica nanoparticles are in powder form and therefore prior to mixing them with PDMS, a uniform dispersion of the silica nanoparticles in hexane was prepared. First silica nanoparticles were heated slightly in vacuum oven at 120 °C for 12 hours to get rid of any adsorbed moisture on them. After that certain amount of silica nanoparticles will be added to 100 mL of hexane.

The mixture will be magnetically stirred for about 30 minutes at room temperature to get a fairly uniform mixture. It will be then transferred to ultrasonic vibration for another 1 hour to obtain a uniform dispersion of silica nanoparticles in hexane. The amount of silica nanoparticles will be varied in the dispersion based on its quantity in the PDMS matrix which will be varied between 0.1% to 5 % by wt.

3.3 Preparation of the SILICA/PDMS Nano Composite

To prepare the silica/PDMS nanocomposite silicone elastomer Sylgard 184 from Dow Corning will be used. Sylgard 184 contains two parts: Part-A the elastomer and Part-B the curing agent. To prepare the nanocomposite Part-A will be added to the silica-hexane dispersion under continuous stirring. The amount of Part-A will be adjusted in order to obtain various Silica: PDMS ratio which will be varied between 0.1% to 10% by wt.

The mixture will be then subjected to ultrasonic vibration to obtain a homogeneous mixture. After about 1 hour of ultrasonic vibration, Part-B will be added to the mixture. The amount of Part-B compared to Part-A will be maintained at 1 to 10 ratio(Chang et al., 2015), respectively. The ultrasonic vibration will be continued for another 10 minutes. The obtained silica-Sylgard 184 mixture will be then cured at 80 °C for at least 2 hours to obtain the silica/PDMS nanocomposite.

3.4 Preparation of SILICA/PDMS Nano Composite Thin Film

3.4.1 Cleaning of Glass Substrate

To develop the silica/PDMS nanocomposite thin films, glass will be used as the substrate. First glass substrates will be cleaned using ultrasonic vibration with soap water, followed by ethanol, then after with acetone, and finally with deionized (DI) water for 15 minutes in each case. The cleaned glass substrates will be then dried in a oven at 90 °C for about 1 hour prior to further experimentation**.**

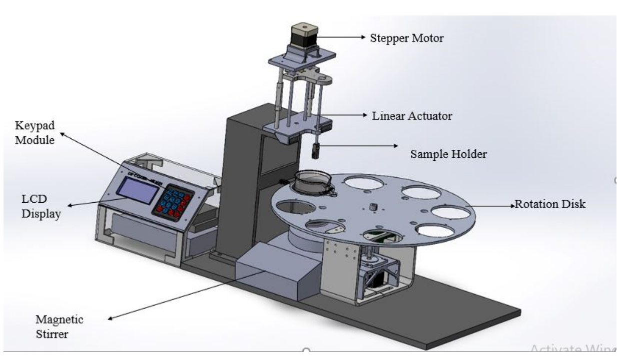
3.4.2 Preparation of SILICA/PDMS Nanocomposite Thin film

First, the silica/PDMS mixture will be prepared using the same process described in section 3.3. Only modification will be that the final curing step at 80 °C will not be performed here. The amount of silica nanoparticles to PDMS will be varied between 0.1% to 10 % by wt. Using a custom-made dip coating machine (Figure 3.1), the cleaned glass substrates will be vertically dipped into the silica/PDMS mixture to apply a thin film layer on its surface.

The process of dip coating will be investigated to understand the film formation and its surface wettability. Table 3.2 describes the various parameters that will be varied during the dip coating technique in order to study the coating process. After the coating of the glass substrate with the silica/PDMS mixture, the substrates will be subjected to curing at 80 °C for 2 hours to obtain the soft silica/PDMS thin film on the glass substrate.

Figure 3.1

Dip Coater System



Note. Schematic representation of Multi-Vessel Dip-Coater system.

Table 3.2

Parameters that will be Investigated During the Dip Coating Process of the SILICA/PDMS Thin films

Parameters	Range
Ratio of silica nanoparticles to PDMS elastomer	0.1 – 10 % by wt.
Dipping speed	1 mm/s to 10 mm/s
Dipping time	30 s to 300 s
Repeating dip cycles	1 to 10
Drying time between each cycle	10 minutes

3.5 Characterizations of the SILICA/PDMS Thin Film

The as-obtained silica/PDMS nanocomposite thin films on glass substrates will be investigated by using optical microscopes to observe the surface topography and any possible micro-cracks present on the film during formation. Scanning electron microscope will be used to further understand the morphology of the thin films(Luo et al., 2018). UV-visible spectroscopy will be used to record the optical transmission through these films concerning the amount of silica nanoparticles in the films.

3.6 Evaluation of Surface Wettability

Surface wettability of the silica/PDMS nanocomposite thin films will be investigated using sessile drop method. DI water will be used to study the hydrophobicity of the surface. Pure PDMS films on glass substrate without silica nanoparticles will be used as control samples(Huhtamäki et al., 2018). To study the static water contact angle, 5 μL of water droplet will be placed at various positions of the thin film and the image of the water droplet will be recorded using an image recording device (Camera)(Kang et al., 2016).

The image will be then analyzed by using ImageJ software to obtain the contact angle made by the liquid droplet at the solid/liquid/vapor interface. The final static water contact angle will be expressed as average of water contact angles obtained from at least 10 different locations of the film. In order to study the dynamic water contact angle, we will use a movable stage where a 5 μL water droplet, similar to the static water contact angle will be applied on the thin film surface. The thin film will be first placed horizontally and at this position the image of the droplet will be recorded(Kang et al., 2016).

The stage will be then moved at an inclination angle increasing by 1° until the water droplet rolls off the surface or the surface completes ration of 180°. The image of the water droplet will be recorded at every angle. Using the ImageJ software, we will then determine the advancing and receding contact angles, contact angle hysteresis and siding angle. These values will be calculated for different amounts of silica nanoparticles in the silica/PDMS nanocomposite thin film and compared.

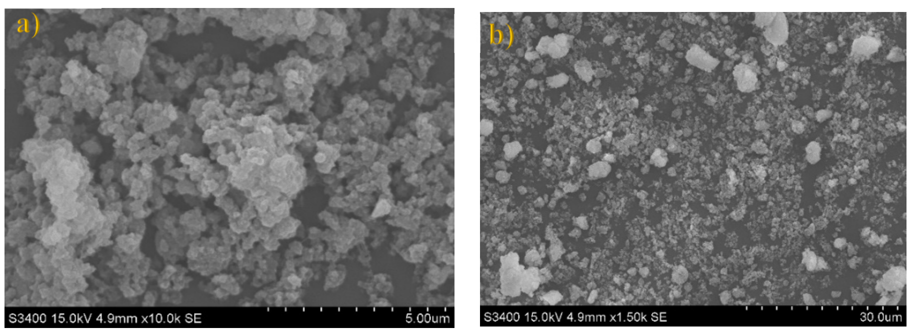
CHAPTER 4
RESULTS AND DISCUSSION

All experiment were performed by introducing Silica/PDMS into n-hexane solvent, and the substrate used was glass. Dip coating technique was used to coat the glass substrate with a desire of achieving super hydrophobicity. This section describes all the results obtained in various experiments.

4.1 Surface Morphology of SILICA/PDMS Coatings

Figure 4.1

SEM Micrographs of Plain PDMS Coating on Glass



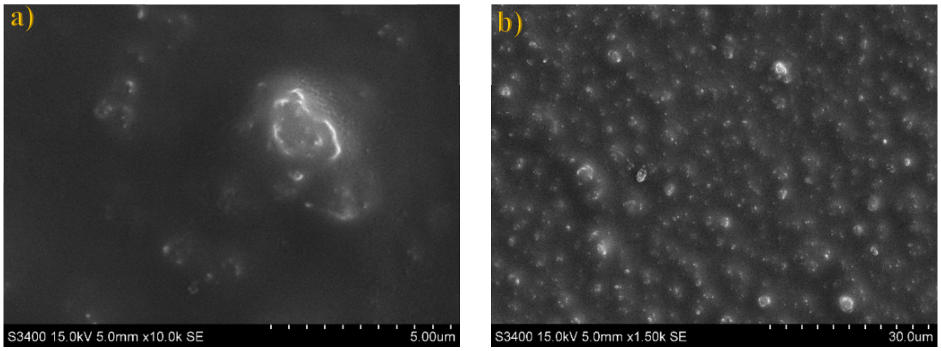
Note. The images presented in the figure 4.1 displays the SEM micrograph of plain PDMS thin film deposited on glass substrate.

In this study, surface morphology and grain size of the sample thin film were analysed using a Field Emission scanning electron microscopy (FESEM) using (JEOL-JSM-7600F). Figure 4.1 Shows the SEM micrograph of plain PDMS thin film deposited on glass substrate.

These images reveal the information about the surface morphology of the plain PDMS material. The image with magnification of x10.0k shows the roughness over the surface of the coating. The coating has the morphology with clusters which are distributed over a region as shown in SEM image with lower magnification i.e., x1.50k.

Figure 4.2

SEM Micrographs of 0.1% SILICA/PDMS Nano Composite Coating with KH-550:

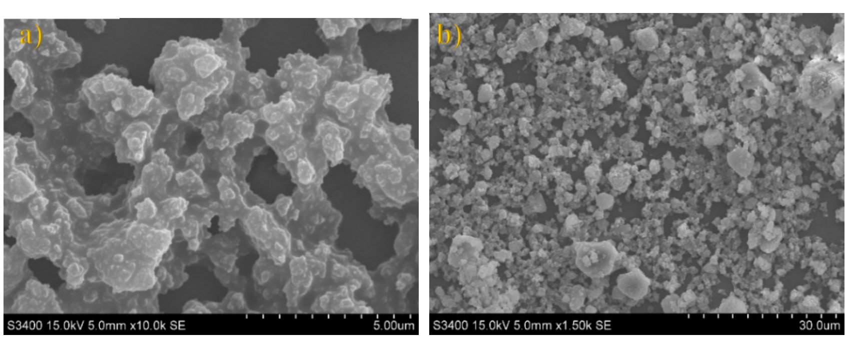


Note. The images presented in the figure 4.2 shows the SEM micrograph of Silica/PDMS Nanocomposites with 0.1% at various magnifications.

The SEM images of PDMS nanocomposite with 0.1% Silica are captured with different magnifications, i.e., x10.0k and x1.50k. The image with x1.50k magnification i.e., 30µm scaled image shows the surface morphology of the nanocomposite over a wide area. As the magnification increased to x10.0k, it shows the bulge on the surface due to the presence of lower content of silica. At the same time, it was also observed that silica nanoparticles are spread over the surface in agglomerated form, indicating poor dispersion of the silica nanoparticles in the PDMS matrix.

Figure 4.3

SEM Micrographs of 1% SILICA/PDMS Nano Composite Coating KH-550

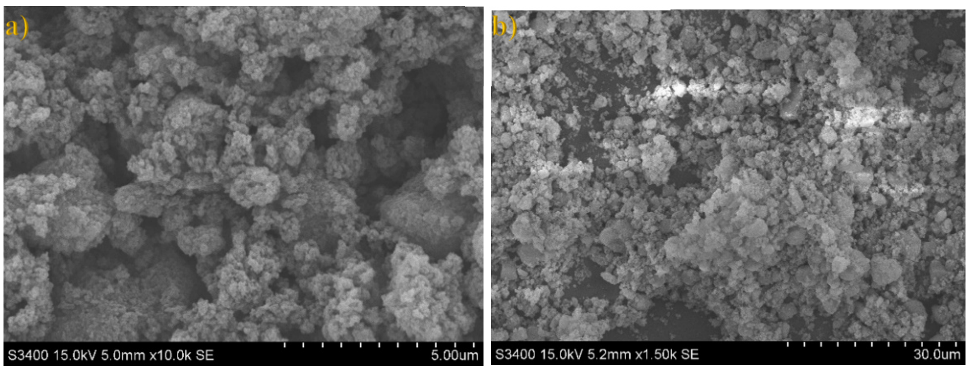


Note. The images presented in the figure 4.3 shows the SEM micrographs of 1% Silica/PDMS Nano composites at various magnifications.

Above are the SEM images of the PDMS nanocomposites with 1% Silica. In the image with the magnification of x10.0k, the distribution of Silica nano particles appears clearly. At x1.50k magnification, the surface showing higher surface roughness compared to that of the PDMS nanocomposite with 0.1% Silica.

Figure 4.4

SEM Micrographs of 5% SILICA/PDMS Nano Composite Coating KH-550



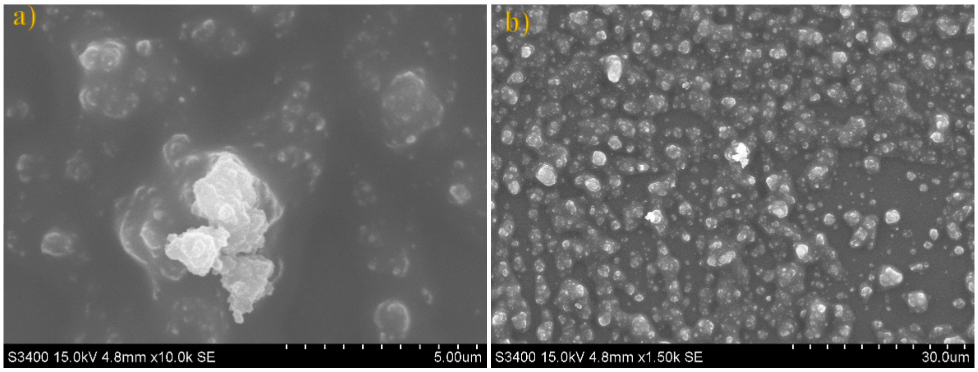
Note. The images presented in the figure 4.4 shows the SEM micrographs of 5% Silica/PDMS Nano composites at various magnifications.

Above are the SEM images of the PDMS nanocomposites with 5% filler Silica nanoparticles. As the content of the silica increases, the agglomeration of the silica nano particles on the surface of the PDMS increases. This agglomeration causes to increase the surface roughness of the composite which is shown in the image with the magnification of x10.0k.

From the comparison of the SEM images of the PDMS nanocomposite with the content of silica with varied compositions, the agglomeration of the silica nanoparticles increases as the content of silica nanoparticles changes from 0.1% to 5%. This causes the formation of bulges and big clusters on the surface of the PDMS coating.

Figure 4.5

SEM Analysis of 0.1% SILICA/PDMS Nano composite with KH-570

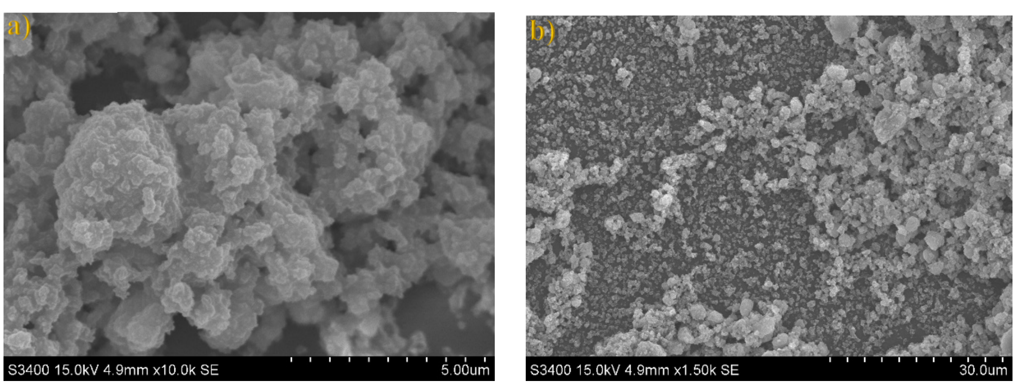


Note. The images presented in the figure 4.5 shows the SEM micrographs of 0.1% Silica/PDMS Nanocomposites at various magnifications.

The above SEM images gives the idea about the surface morphology of the PDMS nano composite with 0.1% silica that included KH570 as binding agent. The image at x10.0k magnification shows the bulge formed on the surface due to the lower content of the silica over PDMS material surface. The number of bulges can be seen over a broad area in the image with x1.50k.

Figure 4.6

SEM Analysis of 1% SILICA/PDMS Nano Composite with KH-570

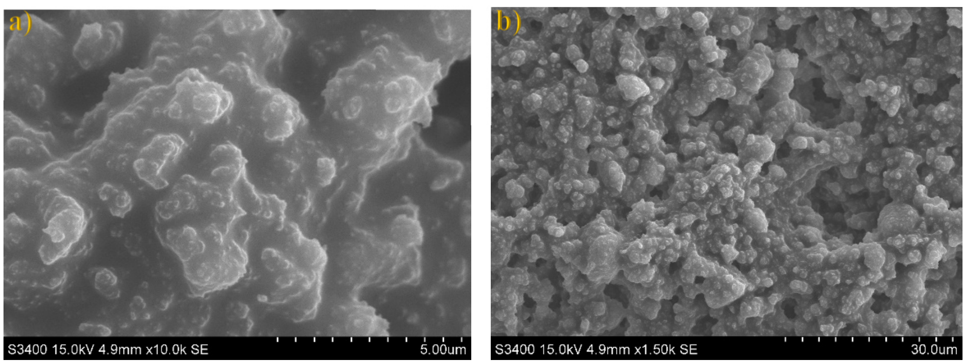


Note. The images presented in the figure 4.6 shows the SEM micrographs of 1% Silica/PDMS Nanocomposites at various magnifications.

Above SEM images explain the surface morphology of the PDMS nano composite with 1% silica that included KH570 as binding agent. In the image with x10.0k magnification, it is visible that the formation of the cluster is in large with small in size. The distribution of these clusters over the surface of the PDMS material is non-uniform, which can be seen in the image with x1.50k magnification.

Figure 4.7

SEM Analysis of 5% SILICA/PDMS Nano Composite with KH-570



Note. The images presented in the figure 4.7 shows the SEM micrographs of 5% Silica/PDMS Nanocomposites at various magnifications.

In the above SEM images, it is clear that the presence of 5% silica nanoparticles in the PDMS material with KH570, causes the formation of bulk clusters on the surface. The images with magnification of 1% and 5% shown the surface morphology of the PDMS nanocomposite.

As the content of silica increases in the PDMS material, due to the presence of the binding agent KH570, the bonds formation between silica and PDMS will be increased. This results in the surface morphology of the materials with the formation of bulk clusters. These silica clusters might be results in the improvement of the surface roughness of the thin film.

4.2 Water Contact Angle

Wettability is one of the most important phenomena used for the classification of surfaces based on the parameter water contact angle. WCA is the measuring parameter that is used for the classification of various surfaces based on the angle that produced with the liquid with respective surface

In this study we have coated the glass substrate with SILICA/PDMS nanocomposite prepared at various concentrations starting from 0.1% to 5% by wt. and we had varied the conditions of dip coater such as dip-time, dry-time, dip-speed and number. Of. cycles and investigated the wettability of the coated surface.

In the below section the Table 4.1 displays the standard coating that we have used for coating and in the followed sections we explain the results that we achieved through the following conditions. To check the uniformity and to evaluate the wettability of the surface we had measured the water contact angle at 9 different positions of the coated film and took the average of all positions.

Table 4.1

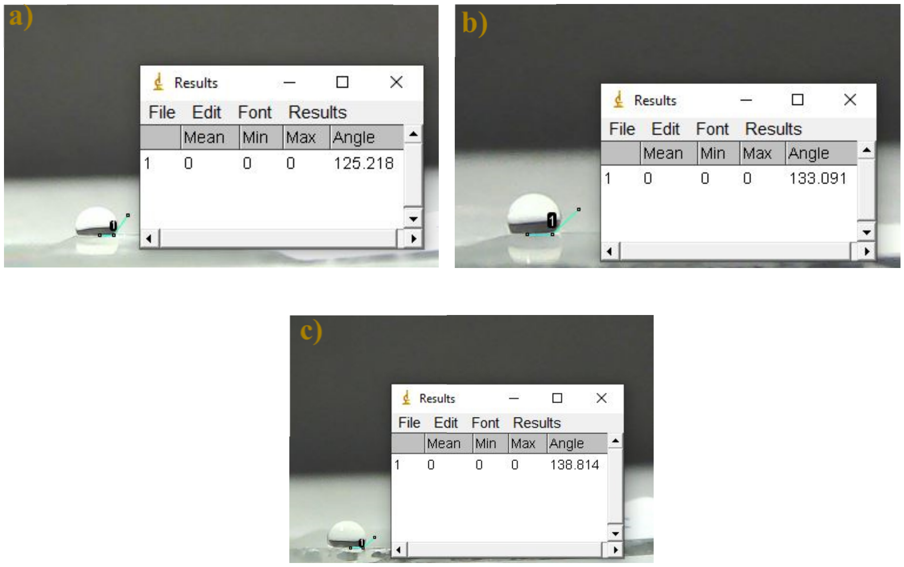
Conditions used for Coating Glass Substrate

Dip Parameter's	Condition 1	Condition 2	Condition 3
Dip Time	5 mins	10 mins	20 mins
Dry Time	10 mins	20 mins	30 mins
Dip Speed	1 mm/s	2 mm/s	3 mm/s
Number of cycles	1	2	3

Note: Table 4.1 presents the various dip-coating parameters and the conditions that has been used for coating the glass substrate.

Figure 4.8

Static Water Contact Angle with SILICA (KH-550) / PDMS of 0.1% Concentration by wt.



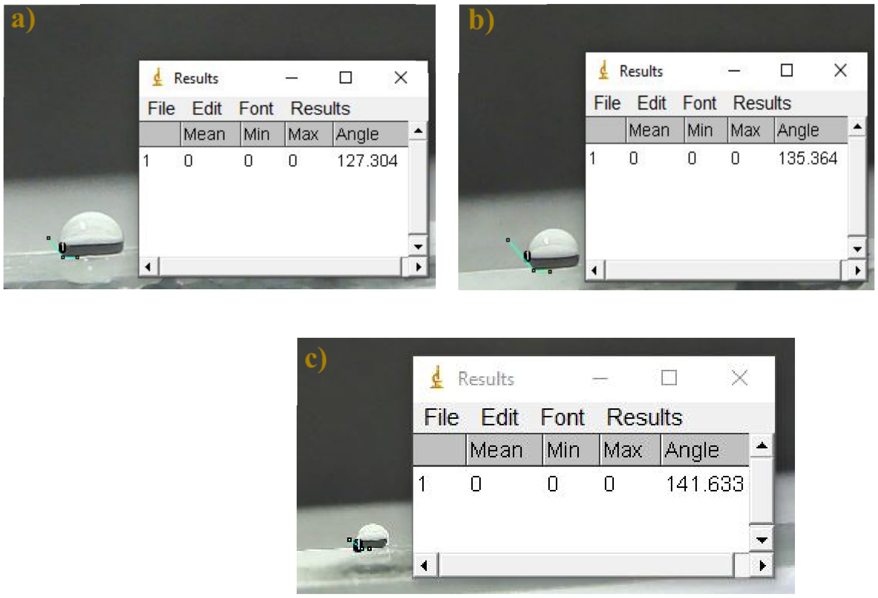
Note. The Images (a), (b), (c) displays the static contact angles of Silica/PDMS of 0.1% concentration by wt.

Static contact angle measurement was carried out by introducing a 5µl water droplet on a coated glass substrate and captured an image using advanced camera and dropped the captured image into ImageJ software and we achieved this contact angle of 125.2±3̊ when we coated the substrate using condition 1.

We introduced the sample into beaker with the speed of 1mm/s and we had repeated this cycle for 1 time, In the same way the contact angle displayed in figure-b i.e., contact angle of 133.1± 5̊ has been achieved when we coated the sample for 2 times with the coating conditions as mentioned in the table 4.1, and when we repeated this process for 3 times we achieved a contact angle of 138.8± 3̊.

Figure 4.9

Static Water Contact Angle of SILICA (KH-550) / PDMS of 0.5 % Concentration by wt.

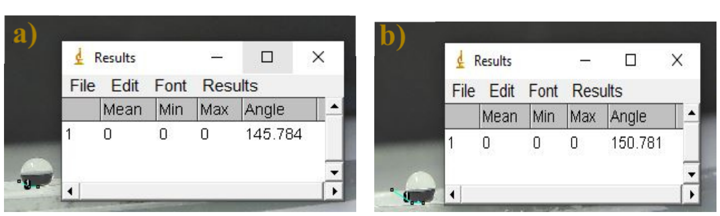


Note. The Images (a), (b), (c) displays the static contact angles of Silica/PDMS of 0.5% concentration by wt.

Throughout all the experiments we had followed the same coating conditions as mentioned in table 4.1 and we varied the concentration of SILICA/PDMS and observed the change in wettability of the developed surface if we compare the figures of 0.1% and 0.5% of SILICA/PDMS we observed the trend of increasing in the surface wettability as concentrations are varied.

Figure 4.10

Static Water Contact Angle of SILICA(KH-550) / PDMS of 1 % by wt.

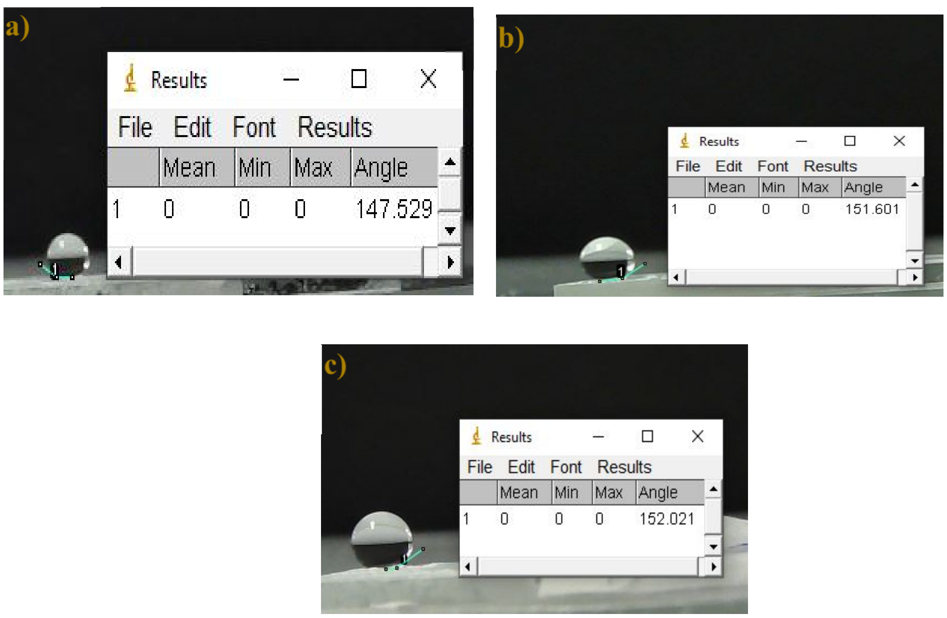


Note. The Images (a), (b) displays the static contact angles of SILICA/PDMS of 1% concentration by wt.

The results presented in the figure 4.10 are the static water contact angle of SILICA/PDMS composite mixture which are prepared with 1% by wt. and coated the prepared mixture on the substrate using condition.2 of Table 4.1 and achieved the contact angle of 145.8± 3° and coated using the condition.3 mentioned in Table 4.1 in and achieved the water contact angle of 150.8±2°.

Figure 4.11

Static Water Contact Angle of SILICA(KH-550) / PDMS of 2 % Concentration by wt.

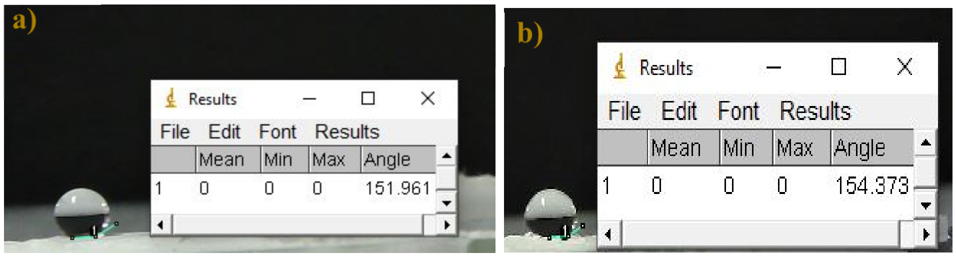


Note. The Images (a), (b), (c) displays the static contact angles of SILICA/PDMS of 2 % concentration by wt.

The results presented in figure 4.11 are the static water contact angle of SILICA/PDMS nanocomposite with 2% concentration and coated with conditions mentioned in Table 4.1 and achieved the water contact angles of 147.6±2°, 151.6±1°, 152.1± 3° and here in the figure 4.11 we had seen that samples coated with condition 2&3 of Table 4.1 has produced superhydrophobic surface.

Figure 4.12

Static Water Contact Angle of SILICA(KH-550) / PDMS of 5 % Concentration by wt.

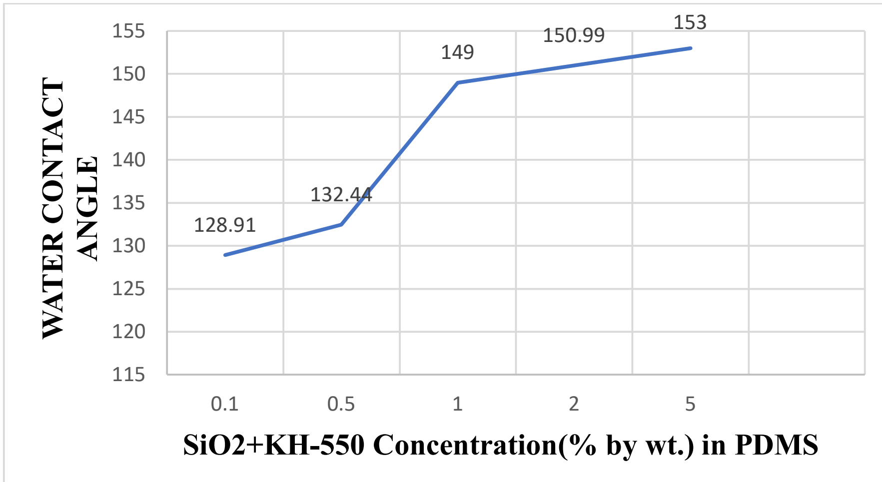


Note. The Images (a), (b) are the static contact angles of SILICA/PDMS of 5 % concentration by wt.

The results presented in figure 4.12 are the static water contact angle of SILICA/PDMS nanocomposite with 5% concentration and coated with conditions mentioned in Table 4.1 and achieved the water contact angles of 151.9±2°, 154.4±1° and here in the figure 4.12 we had seen that samples coated with condition 2&3 of Table 4.1 has produced superhydrophobic surface.

Figure 4.13

Static Water Contact Angle of SILICA(KH-550)/PDMS Nano Composite with Variation Silica Nanoparticle Concentrations.

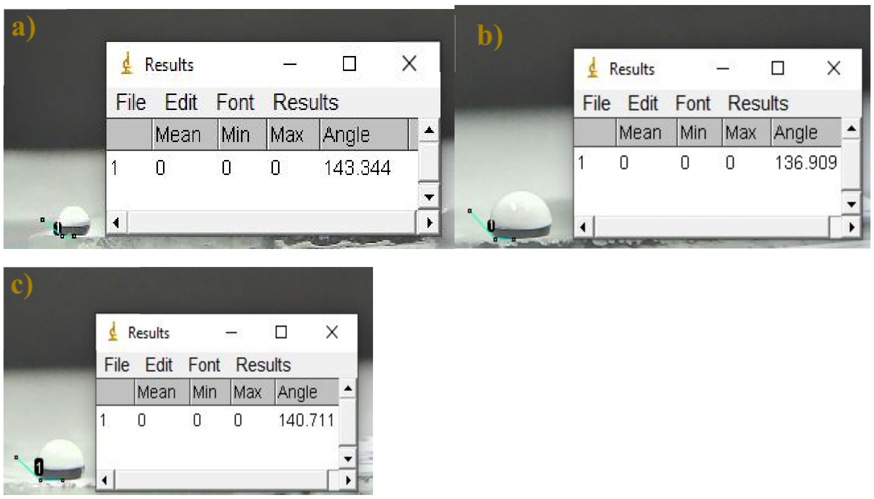


Note. The figure 4.13 shows the analysis of static water contact angle shown by SILICA/PDMS at various Concentrations.

The graph presented in the figure 4.13 is the analysis of static water contact angle SILICA/PDMS nano composite coated at various concentrations. The X-axis of the graph represents the concentrations of the mixture whereas the Y-axis represents the values of water contact angle, and the values marked on the graph are an average of water contact angle measured at 9-different positions of the opted sample. This evaluation of water contact angle helps to evaluate the uniformity of the coating and the graph we presented helps us to analyze the trend of water contact angle at various concentrations.

Figure 4.14

Static Water Contact Angle with SILICA(KH-570) / PDMS of 0.1% Concentration by wt.

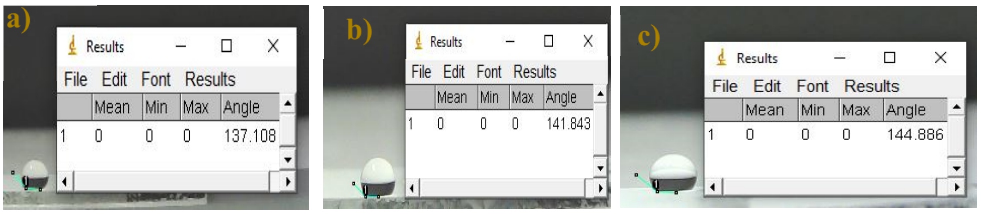


Note. The Images (a), (b), (c) displays the static contact angles of SILICA/PDMS of 0.1 % concentration by wt.

The results presented in the figure 4.14 are the static water contact angle of SILICA/PDMS composite mixture which are prepared with 0.1% by wt. and coated the prepared mixture on the substrate using condition 1,2,3 of Table 4.1 and achieved the contact angle of $136.9 \pm 2^\circ$,$140.7 \pm 3^\circ$ and got the contact angle of $143.3 \pm 2^\circ$ when coated using the condition.3 mentioned.

Figure 4.15

Static Water Contact Angle with SILICA (KH-570) / PDMS of 0.5 % Concentration by wt.

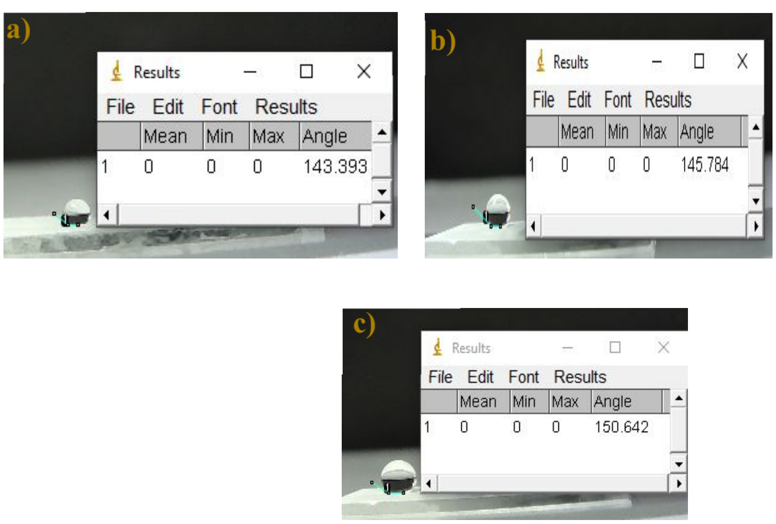


Note. The Images (a), (b), (c) displays the static contact angles of SILICA/PDMS of 0.5 % concentration by wt.

The results presented in the figure 4.15 are the static water contact angle of SILICA/PDMS composite mixture which are prepared with 0.5 % by wt. and coated the prepared mixture on the substrate using 3 conditions of Table 4.1 and achieved the contact angle of 137±3°,141.8 ± 2°, and 144.8± 2 °.

Figure 4.16

Static Water Contact Angle with SILICA(KH-570) / PDMS of 1 % Concentration by wt.

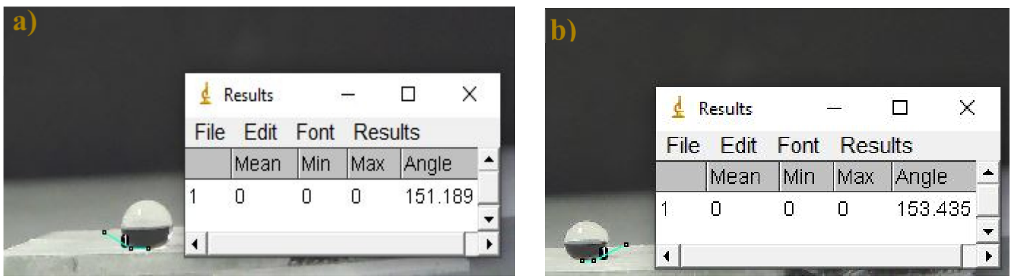


Note. The Images (a), (b), (c) displays the static contact angles of SILICA/PDMS of 1% concentration by wt.

The results presented in the figure 4.16 are the static water contact angle of SILICA/PDMS composite mixture which are prepared with 1% by wt. and coated the prepared mixture on the substrate and achieved the contact angle of 143.3±3°,145.7±3° ,150.6±2°.

Figure 4.17

Static Water Contact Angle with SILICA(KH-570) / PDMS of 2 % Concentration by wt.

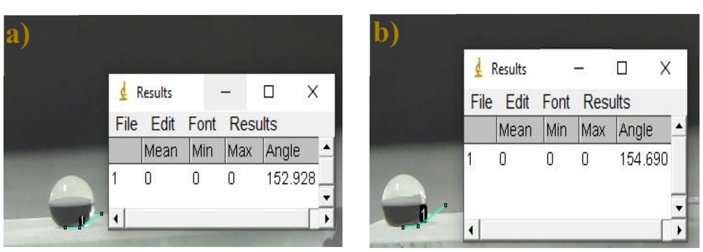


Note. The Images (a), (b) are the static contact angles of SILICA/PDMS of 2 % concentration by wt.

The results presented in figure 4.17 are the static water contact angle of SILICA/PDMS nanocomposite with 2% and achieved the water contact angles of 151.9±1°, 153.1± 2° and here in the figure 4.17 we had seen that samples coated with condition 2&3 of Table 4.1 has produced Super hydrophobic surface.

Figure 4.18

Static Water Contact Angle of SILICA (KH-570) / PDMS of 5 % Concentration by wt.

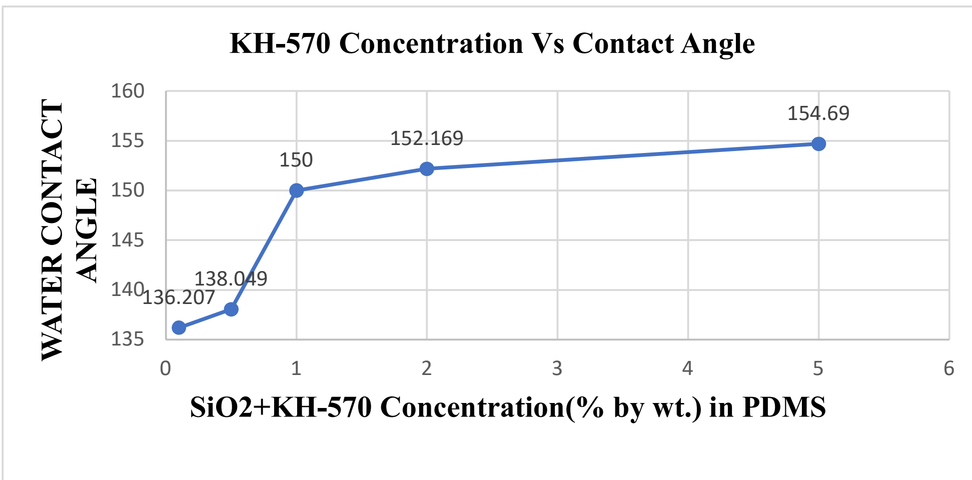


Note. The Images (a), (b) are the static contact angles of SILICA/PDMS of 5 % concentration by wt.

The results presented in figure 4.18 are the static water contact angle of SILICA/PDMS nano composite with 5% concentration and coated with conditions mentioned in Table 4.1 and achieved the water contact angles of 152.9±2°, 154.6±1° and here in the figure 4.18 we had seen that samples coated with condition 1&2 of Table 4.1 has produced super hydrophobic surface.

Figure 4.19

Static Water Contact Angle of SILICA(KH-570)/PDMS Nano composite with Variation of Silica Nanoparticle Concentrations.

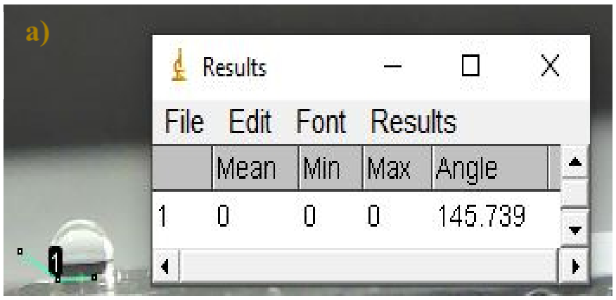


Note. The figure 4.19 shows the analysis of static water contact angle shown by SILICA/PDMS at various Concentrations.

The graph presented in the figure 4.19 is the analysis of static water contact angle SILICA/PDMS nano composite coated at various concentrations. The X-axis of the graph represents the concentrations of the mixture whereas the Y-axis represents the values of water contact angle, and the values marked on the graph are an average of water contact angle measured at 9-different positions of the opted sample. This evaluation of water contact angle helps to evaluate the uniformity of the coating and the graph we presented helps us to analyze the trend of water contact angle at various concentrations.

Figure 4.20

Contact Angle of PDMS:



Note. The Images (a), (b) are the static contact angles of SILICA/PDMS of 5 % concentration by wt.

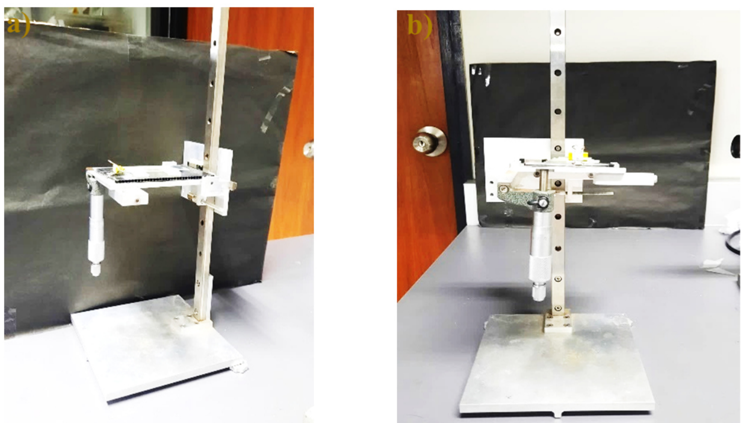
On Theoretical bases, we all know that PDMS is one of the major hydrophobic constituent materials, and it is one the most crucial material or compound which plays a key role in developing super hydrophobic surface. But through the figure 4.20 we can once again confirm and evaluate that PDMS is hydrophobic material and through this research study once again proves that how important is the role of PDMS in developing superhydrophobic surface.

4.3 Sliding Angle

It is a technique which measures the contact angle when the movement of particle takes place from its initial position. In order to measure sliding angle of the coated material we require some preliminary setup to measure sliding angle of the coated substrates, basic setup that required to measure sliding angle is presented and explained in the following section.

Figure 4.21

Basic Setup used for Measuring Sliding Angle



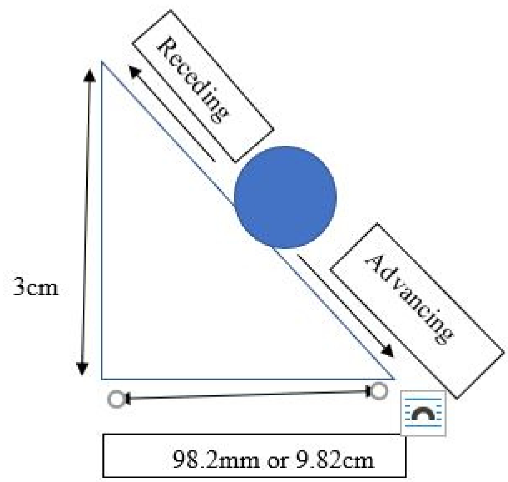
Note. The images presented in figure 4.21 are the images of basic setup used for measuring the sliding angle.

In order to measure sliding angle we used the preliminary setup that presented in figure 4.21(a&b) are the front view and side view of setup, This setup basically nothing but a vertical mount stand which provides the platform to place the sample and its underneath consists of screw, which helps to lift the platform and this setup is capable to lift its platform up to 3cm and this is the key setup to measure dynamic contact angle. Apart from this stand, in this setup we used an advanced USB Camera to capture and record the Videos and Images while measuring the sliding contact angle.

In the Sliding angle, we mainly measure 2 parameters such as advancing angle and receding angle.

Figure 4.22

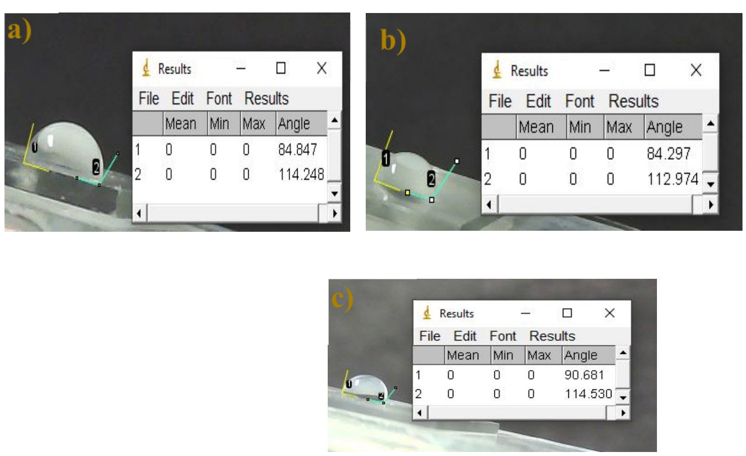
Basic outlook of Sliding Angle Measuring Setup



As shown in figure 4.22, (9.82cm is the horizontal length of the platform where sample is used to be placed and 3cm is the maximum height of the shaft which is used to lift the platform for providing the movement for water droplet present on sample).

Figure 4.23

Sliding Angle with SILICA (KH-550) / PDMS of 0.1% Concentration by wt.

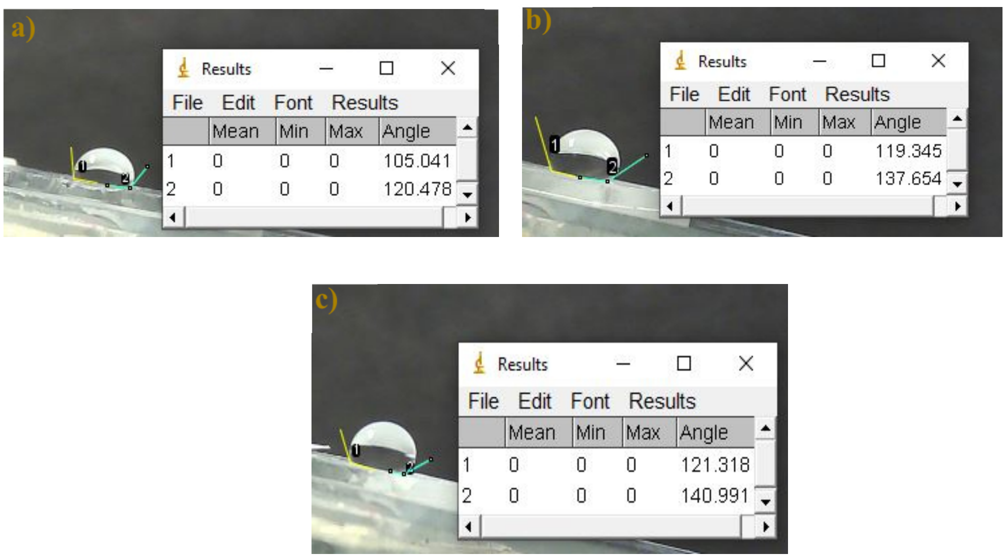


Note. The Images (a), (b), (c) displays the advancing and receding contact angles of SILICA/PDMS of 0.1% concentration by wt.

The advancing and receding contact angles presented in the figure 4.23(a), (b), (c) are the angles of 3 different samples coated with conditions presented in Table 4.1 and achieved the advancing angle of 113.9° and receding angle of 86.6° these are the average of 3 values,

Figure 4.24

Sliding Angle with SILICA(KH-550) / PDMS of 0.5% Concentration by wt.

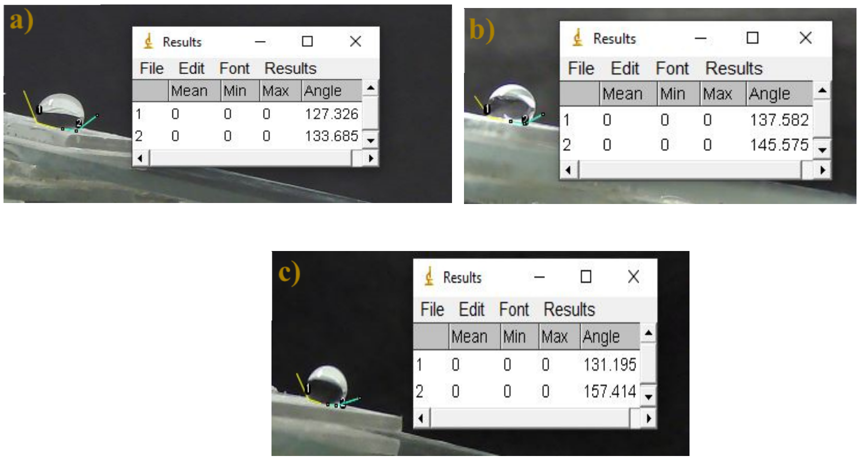


Note. The Images (a), (b), (c) displays the advancing and receding contact angles of SILICA/PDMS of 0.5% concentration by wt.

The advancing and receding contact angles presented in the figure 4.24 (a), (b), (c) are the angles of 3 different samples coated with conditions mentioned in Table 4.1 and achieved the advancing angle of 133.041° and receding angle of 115.234° these are the average of 3 values which presented in the figure, and in this case, there is no movement of water droplet.

Figure 4.25

Sliding Angle with SILICA (KH-550) / PDMS of 1% Concentration by wt.

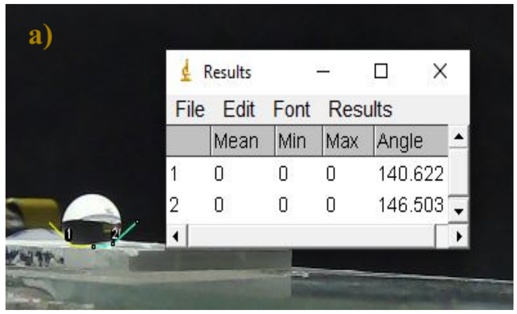


Note. The Images (a), (b), (c) displays the advancing and receding contact angles of SILICA/PDMS of 1 % concentration by wt.

The advancing and receding contact angles presented in the figure 4.25 (a), (b), (c) are the angles of 3 different samples coated with conditions mentioned in Table 4.1 and achieved the advancing angle of 145.558° and receding angle of 132.034° these are the average of 3 values which presented in the figure.

Figure 4.26

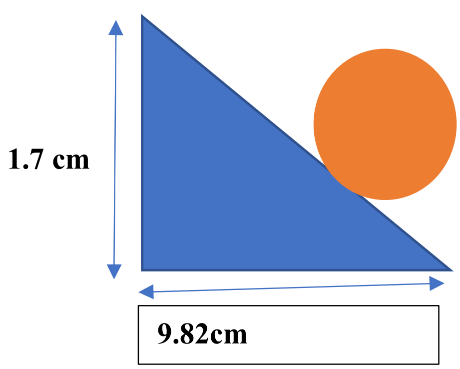
Sliding Angle with SILICA (KH-550) / PDMS of 2% Concentration by wt.



Note. The Image (a) of figure 4.26 displays the advancing and receding contact angles of SILICA/PDMS of 2% concentration by wt.

The advancing and receding contact angles presented in the figure 4.26 (a) are the results of sample coated with condition 1 of Table 4.1 and achieved the advancing angle of 146.503° and receding angle of 140.622°, and in this case we can mention it as best condition as it capable of producing movement in water droplet. This condition has produced the movement in water droplet at an angle of 9.82°.

In order to derive Sliding angle, we need to use "Tan Inverse" function of trigonometry, and in this process of deriving there are two important points to be noted i.e., the height at which the water droplet has moved, and this parameter varies from case to case and length of the horizontal platform where sample is used to place, in this case it is 9.82cm and it is fixed value.



$$\tan \theta = 1.7\text{cm} / 9.82\text{cm}$$

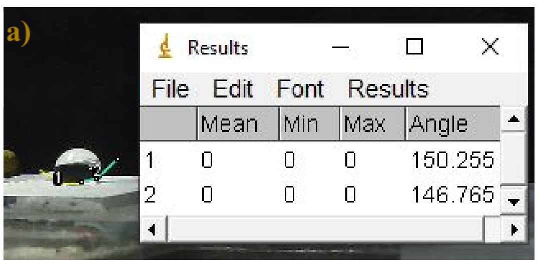
$$\Theta = \tan^{-1}(1.7/9.82)$$

$$\theta = 9.82^{\circ}$$

Therefore 9.82° is the sliding angle and through this it once again confirms that it is superhydrophobic.

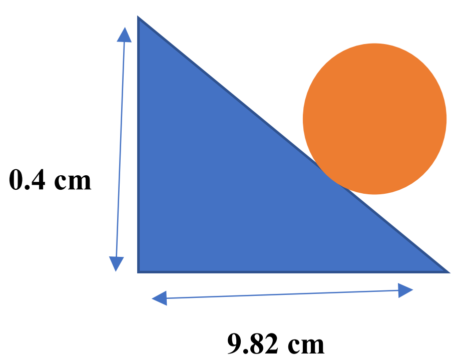
Figure 4.27

Sliding Angle with SILICA (KH-550) / PDMS of 5 % Concentration by wt.



Note. The Image (a) displays the advancing and receding contact angle of SILICA/PDMS of 5% concentration by wt.

The advancing and receding contact angles presented in the figure 4.27 (a) are the results of sample coated with condition 1 of Table 4.1 and achieved the advancing angle of 150.255° and receding angle of 146.765°, and in this case we can mention it as best condition as it capable of producing sliding angle. This condition has produced the movement in water droplet at an angle of 2.33°.



$$\tan\theta = 0.4cm / 9.82cm$$

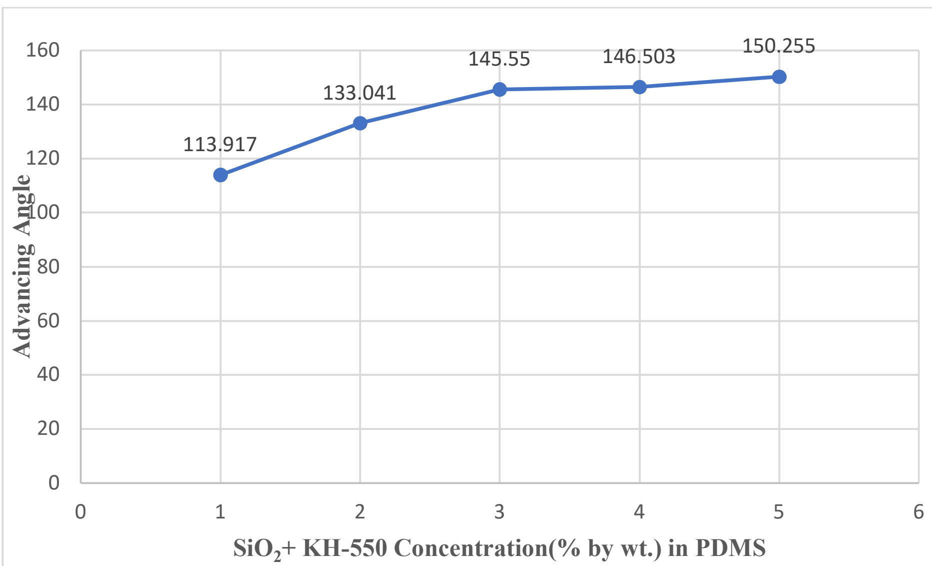
$$\Theta = \tan^{-1} (0.4/9.82)$$

$$\theta = 2.33^{\circ}$$

Therefore 2.33° is the sliding angle of figure 4.27 and through this it once again confirms that it is superhydrophobic surface.

Figure 4.28

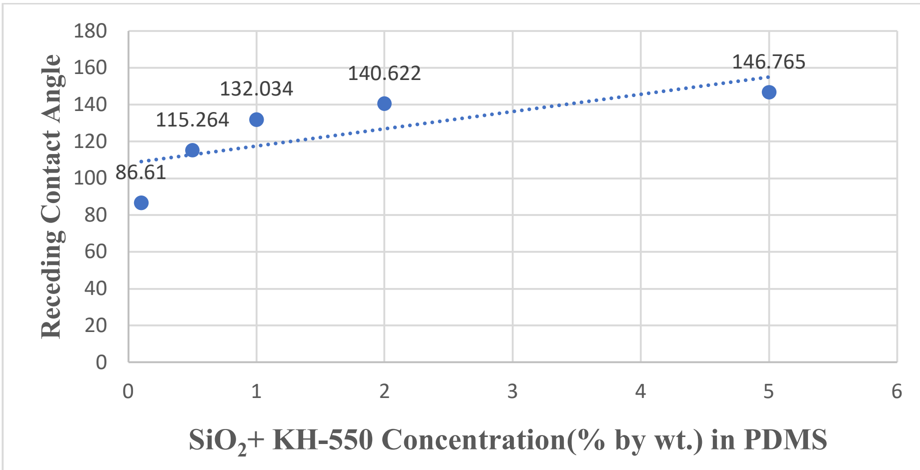
Variations of Advancing Contact Angles of SILICA (KH-550)/PDMS Concentrations



Note. Figure 4.28 presents the analysis of Advancing Contact Angle at various concentrations

Figure 4.29

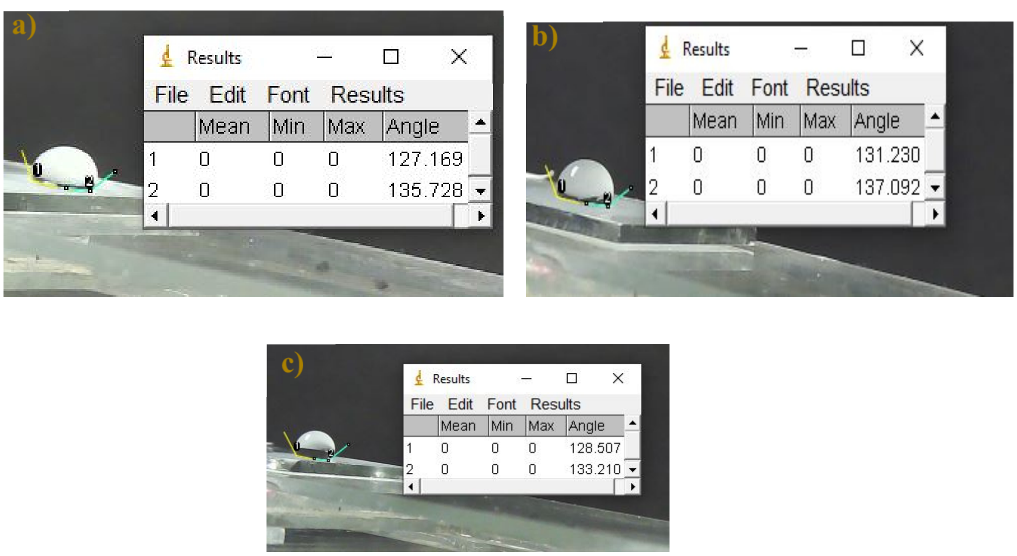
Variations of Receding Contact Angles of SILICA (KH-550)/PDMS at Various Concentrations.



Note. Figure 4.29 presents the analysis of Receding Contact Angle at various concentrations.

Figure 4.30

Sliding Angle with SILICA (KH-570) / PDMS of 0.1% Concentration by wt.

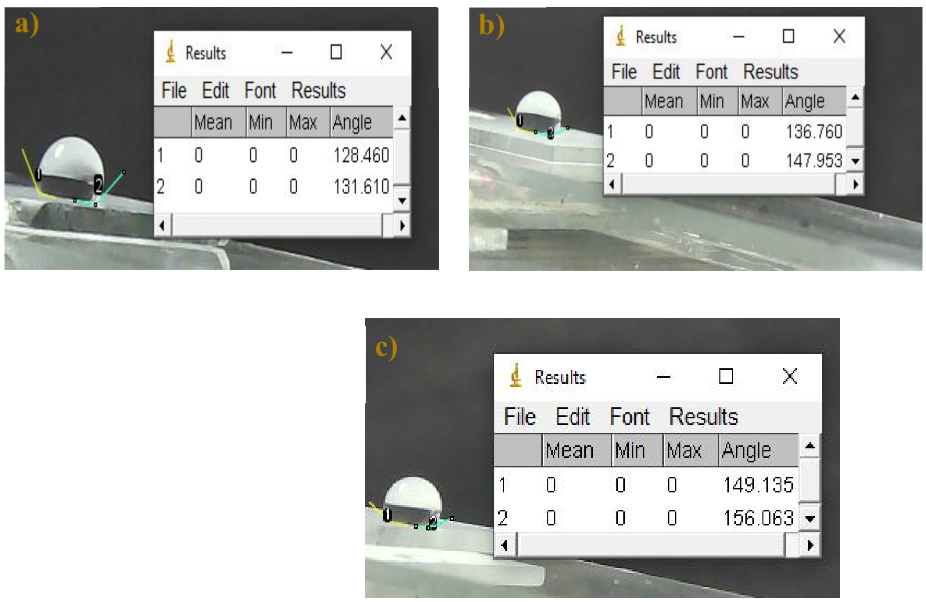


Note. The Images (a), (b), (c) displays the advancing and receding contact angles of SILICA (KH-570) /PDMS of 0.1% concentration by wt.

The advancing and receding contact angles presented in the figure 4.30 (a), (b), (c) are the angles of 3 different samples coated with conditions presented in Table 4.1 and achieved the advancing angle of 135.343° and receding angle of 128.968° these are the average of 3 values.

Figure 4.31

Sliding Angle with SILICA(KH-570) / PDMS of 0.5% Concentration by wt.

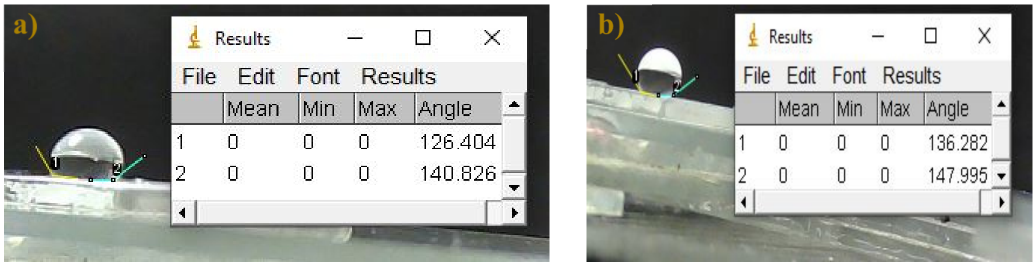


Note. The Images (a), (b), (c) displays the advancing and receding contact angles of SILICA(KH-570) /PDMS of 0.5 % concentration by wt.

The advancing and receding contact angles presented in the figure 4.31 (a), (b), (c) are the advancing and receding angles of 3 different samples coated obtained with conditions presented in Table 4.1 and achieved the advancing angle of 145.2° and receding angle of 138.1° these are the average of 3 values.

Figure 4.32

Sliding Angle with SILICA(KH-570) / PDMS of 1% Concentration by wt.

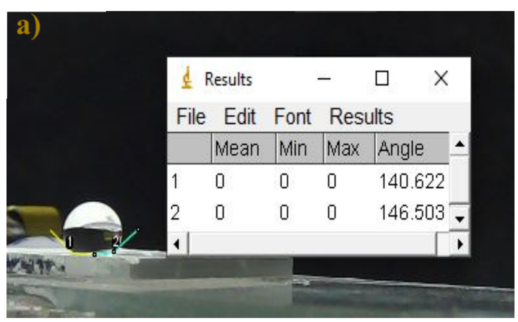


Note. The Images (a), (b) displays the advancing and receding contact angles of SILICA(KH-570) /PDMS of 1 % concentration by wt.

The advancing and receding contact angles presented in the figure 4.32 (a), (b) are the angles of 3 different samples coated with conditions presented in Table 4.1 and achieved the advancing angle of 144.4°and receding angle of 131.3° these are the average of 2 values.

Figure 4.33

Sliding Angle with SILICA (KH-570) / PDMS of 2% Concentration by wt.

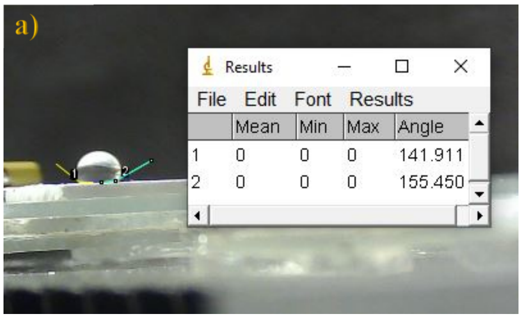


Note. The Images (a) displays the advancing and receding contact angles of SILICA(KH-570) /PDMS of 1 % concentration by wt.

The advancing and receding contact angles presented in the figure 4.33 (a) are the results of sample coated with condition 1 of Table 4.1 and achieved the advancing angle of 146.5° and receding angle of 140.6°, and in this case we can mention it as best condition as it capable of producing sliding angle. This condition has produced the movement in water droplet at an angle of 3.5°.

Figure 4.34

Sliding Angle with SILICA (KH-570) / PDMS of 5% Concentration by wt.

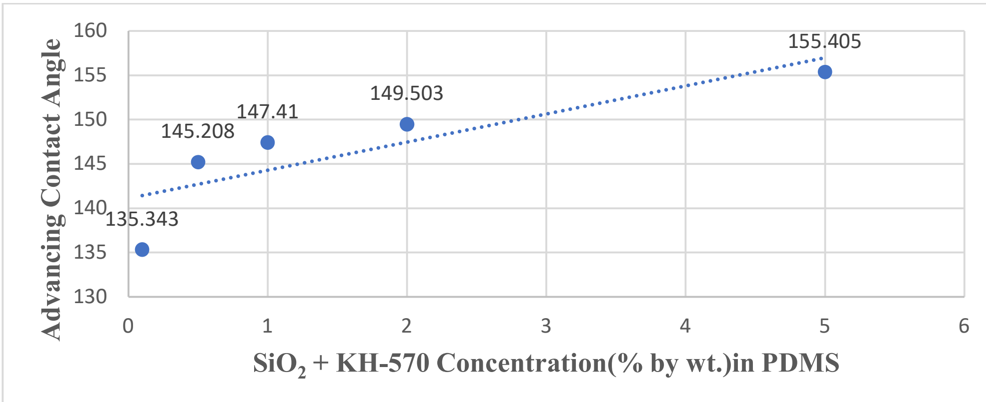


Note. The Images (a) presented in the figure 4.34 are the advancing and receding contact angles of SILICA (KH-570) /PDMS of 5 % concentration by wt.

The advancing and receding contact angles presented in the figure 4.34(a) are the results of sample coated with condition 3 of Table 4.1 and achieved the advancing angle of 155.4 ° and receding angle of 141.9°, and in this case we can mention it as best condition as it capable of producing sliding angle. This condition has produced the movement in water droplet at an angle of 2.9°.

Figure 4.35

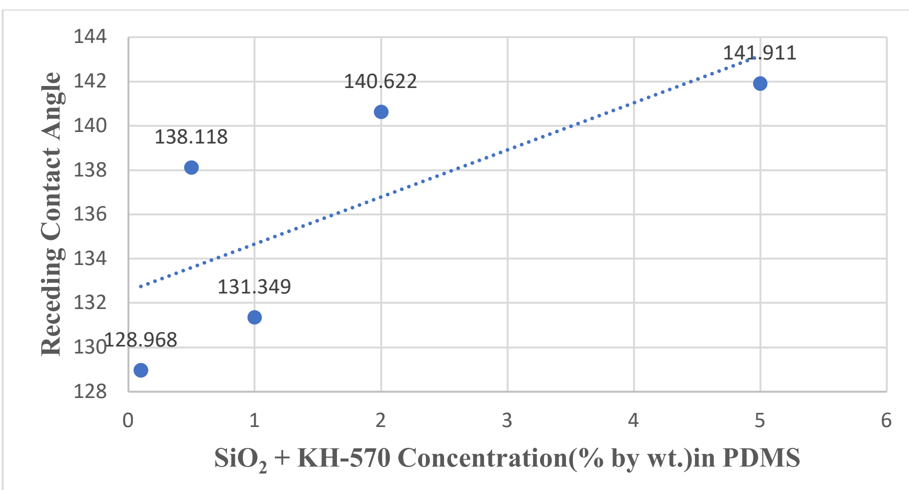
Variations of Advancing Contact Angles of SILICA (KH-570)/PDMS at Various Concentrations



Note. Figure 4.35 presents the analysis of advancing contact angle at various concentrations.

Figure 4.36

Variations of Receding Contact Angles of SILICA (KH-570)/PDMS at Various Concentrations.



Note. Figure 4.3 presents the analysis of Receding Angle at various concentrations.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

A superhydrophobic thin film coating with SILICA/PDMS was fabricated successfully. The SILICA/PDMS nanocomposite mixture was coated on glass substrate using dip-coating technique and the concentration of SILICA/PDMS nanocomposite mixture was varied from 0.1% to 5% by wt. Concentration, using KH550 and KH570 as binding agents. These thin films showed different variations in terms of their morphology which is evaluated using the Scanning Electron Microscope.

Coatings obtained at 2% and 5% concentration demonstrated superhydrophobic nature with water contact angle of 154.7°, while other coated thin films were found to be hydrophobic with water contact angle between 130° to 150°. These coatings have the scope for wide range of commercial applications such as self-cleaning surfaces, solar panels, marine coatings etc. This study demonstrated an inexpensive and simple superhydrophobic thin films that has the potential for future industrial applications.

In the future, further studies should be conducted to achieve better dispersion of the silica nanoparticles in the PDMS matrix. This can be done by investigating different types of binding agents to functionalize the silica nanoparticle surfaces. Additionally, effect of other types of nanoparticles apart from silica can also be investigated to improve the super hydrophobicity.

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VITA

First a fall, I am very glad that I Successfully came to AIT by overcoming the challenges and hurdles that I faced across my bachelor's journey. I can say master's at AIT is really a dream come true for me because as I came from a different education pattern it is very difficult to catch the international pattern in single attempt that too in one of the toughest fields and newly emerging technology "Nanotechnology".

It took me almost 1 year of time to understand the education pattern, In the process of understanding the education pattern, I had almost lost the focus on learning the important things and faced many challenges to meet the required CGPA Criteria. The Journey of beginning at the CGPA of 2.17 to bringing to CGPA of 2.83 is one of the biggest accomplishments that I have achieved in master's Journey. And I strongly believe that hard work and efforts that we put in our desired thing might pay you it's price little late but its pay's you more for sure and it mould's you as a perfect being which is most essential part of successful life.