SUPERHYDROPHOBIC SURFACE MODIFICATION ON THE FABRIC FACE MASK MATERIALS

THANIGESH A/L THIYAGU

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SUPERHYDROPHOBIC SURFACE MODIFICATION ON THE FABRIC FACE MASK MATERIALS

by

THANIGESH A/L THIYAGU

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THANIGESH THIYAGU

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LIST OF SYMBOLS

Symbol	Description	Unit
C	Concentration (in weight percentage)	wt %
θ	Water contact angle	°
V	Percent volume per volume	% v/v
v	microliter	μL
v	milliliter	mL

LIST OF ABBREVIATIONS

CAH	Contact Angle Hysteresis
CVD	Chemical Vapor Deposition
COVID – 19	Coronavirus Disease 2019
$CuSA_2$	Copper (II) Stearate
DTMS	Dodecyltrimethoxysilane
EDX	Energy X – Ray Dispersive spectroscopy
HDTMS	Hexadecyltrimethylsilane
OTES	n – octyltriethoxysilane
PDMS	Polydimethylsiloxane
PET	Polyethylene Terephthalate
PPE	Personal Protective Equipment
SDG	Sustainable Development Goal
SEM	Scanning Electron Microscopy
Si	Silicon
SiO_2	Silicon Dioxide
TEOS	Tetraethylorthosilicate
TiO_2	Titanium Dioxide
WCA	Water Contact Angle
WHO	World Health Organization
ZnO	Zinc Oxide

MODIFIKASI PERMUKAAN SUPERHIDROFOBIK PADA BAHAN FABRIK TOPENG MUKA

ABSTRAK

Pembuangan topeng pembedahan and alat respirator N95 telah menyebabkan pencemaran alam sekitar. Maka permintaan terhadap pemakaian topeng muka fabrik yang boleh dibasuh, yang boleh digunakan kembali, yang mesra alam dan berlestari meningkat namun limitasi yang terdapat pada topeng muka fabrik disebabkan oleh sifatnya yang berhidrofilik meningkatkan peluang penularan wabak virus. Untuk mengatasi kemelut tersebut, permukaan kain fabrik yang digunakan untuk pembuatan topeng muka harus diubahsuai. Permukaan superhidrofobik dengan sifat pembersihan diri telah dicadangkan untuk menyelesaikan masalah ini. Tujuan penyelidikan ini adalah untuk menyiasat teknik mudah pembuatan salutan superhidrofobik pada bahan fabrik seperti kapas, mikrofiber dan mikrofiber campuran. Teknik salut celup telah digunakan untuk proses penyalutan larutan formulasi pada permukaan bahan fabrik. Kesan kepekatan HDTMS terhadap sifat hidrofobik pada permukaan kain fabrik telah disiasat. Apabila kepekatan HDTMS meningkat, sifat hidrofobik pada permukaan kain fabrik juga meningkat. Semua permukaan kain fabrik jenis kapas disalut dengan kepekatan HDTMS 5 % (v/v), 10 % (v/v) dan 20 % (v/v) menunjukkan sudut kenalan air lebih daripada 150° dan dapat mencapai fenomena superhidrofobik. Pengubahsuaian melalui teknik salut celup dua lapisan larutan HTDMS pada permukaan mikrofiber dan mikrofiber campuran dilakukan untuk meningkatkan sifat hidrofobik. Semua permukaan mikrofiber yang disaluti dua lapisan larutan HDTMS mempunyai sudut kenalan air lebih daripada 150° sementara permukaan mikrofiber campuran mencapai sudut kenalan air berhampiran dengan julat superhidrofobik.

Permukaan kain fabrik jenis kapas dan mikrofiber yang diubah suai menonjolkan sifat hindar air yang sangat baik dan sudut kenalan air yang melebihi 150 °. Kekuatan mekanikal dan kestabilan salutan superhidrofobik pada permukaan kain fabrik telah disiasat. Kain fabrik jenis kapas dan mikrofiber juag telah menunjukkan sifat permbersihan diri and sifat tahan bahan cemar yang baik.

SUPERHYDROPHOBIC SURFACE MODIFICATION ON THE FABRIC FACE MASK MATERIALS

ABSTRACT

Disposable surgical masks and N95 respirators have resulted in environmental pollution. Subsequently resulted in increased demand in wearing fabric face mask which are washable, reusable, environmentally friendly and sustainable however, the limitation on fabric-based face mask is the chances for transmission of virus is higher due to the hydrophilic nature. To tackle the problem, the surface of the fabric is modified. Superhydrophobic surface with self – cleaning is proposed to tackle the issue. The present work investigates facile techniques for superhydrophobic coating on cotton, microfiber, and microfiber blend materials. Dip coating was employed to coat the coating solution on the fabric surface. The effect of concentration of HDTMS on the hydrophobicity of different fabrics were investigated. As the concentration of HDTMS increases, the hydrophobicity of the surface also increased. All cotton fabric surface coated with 5 % (v/v), 10 % (v/v) and 20 % (v/v) HDTMS concentration exhibited water contact angle (WCA) more than 150° and able to reach superhydrophobic state. Modification through bilayer coating solution on the microfiber and microfiber blend surface was done to enhance superhydrophobicity. All bilayer coated microfiber surface has WCA of more than 150° while microfiber blend surface reached WCA near to the superhydrophobic range. Modified cotton and microfiber surface displayed excellent water repellence and WCA of more than 150°. The mechanical strength and stability of coating over time of the superhydrophobic cotton and microfiber surfaces have been evaluated. Modified cotton and microfiber displayed excellent self – cleaning and stain – resistant properties.

CHAPTER 1

INTRODUCTION

In this chapter, the detailed introduction on the background study of project is delivered which includes the surge in coronavirus cases globally and in Malaysia. The importance and limitation of current disposable surgical mask in pandemic scenario were discussed. Further highlighted about the environmental pollution caused by improper disposal of surgical face mask which shifted the demand to fabric – based face mask. The problem statement and research objectives will be addressed in detail accordingly. A summarized paragraph will be presented to explain the scopes of the project and thesis organization thoroughly.

1.1 Background study

Coronavirus disease 2019 (COVID – 19) has spread to more than 200 countries and regions (Andersen et al., 2020; Wang et al., 2020).The number of reported cases, as compiled by the World Health Organization (WHO) by 28th July 2021, has exceeded 195,266,156 worldwide with 4,180,161 deaths. Coronavirus is highly contagious. The transmission can spread via direct or indirect contact through respiratory droplets produced by sneezing, coughing, and talking. Respiratory droplets refer to the saliva and nasal mucus from the infected person.

With the continuous rise of the infectious COVID-19 cases globally, the public has been advised to wear face masks to prevent transmission of coronavirus. Face masks is an example of personal protective equipment (PPE) that was utilized predominantly against exposure of pathogens and contaminants in a hospital environment. Alarming COVID – 19 pandemic situations across the globe have increased the necessity to wear face masks in public places.

Most common type of face masks widely used are the disposable surgical masks and N95 respirators. Spunbond – meltbond – spunbond structures consisting of melt – blown microfibers is the most common structure combination used for the surgical masks (Wibisono *et al.*, 2020). Surgical masks are made of three layers where each layer plays different functions specifically. First layer enhances hydrophobicity by preventing fluid to penetrate. The second layer functions as filter which retains viruses in both directions. Third layer act as absorbent to absorb fluids from the wearer. Diagram of disposable surgical masks with three layers is shown in Figure 1.1.

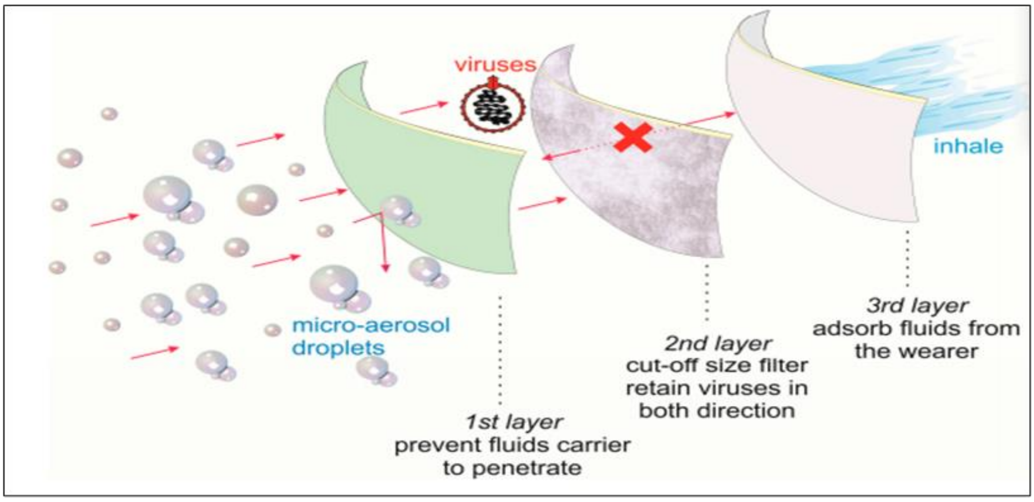

Figure 1.1 Diagram of surgical mask restricting transmission of viruses by three – layer level protection (Wibisono *et al*., 2020).

Meanwhile N95 respirators also consists of three main layers which are the inner layer, middle layer, and outer layer. The filter layer (middle layer) has a filtration efficiency of 95 % and produced from fabrics such as cotton, polypropylene, nylon, polyester, and other polymer fibers (Feng *et al.*, 2020).

Sudden increase in demand for face masks have led to shortages in supply chain. China is a major producer of face masks with almost 50 % contribution in global face masks consumption (Wu *et al.*, 2020). Countries having the capacity of producing face masks are also suffering from a shortage of face masks. These countries include developed nations like the European Union, United states of America, Japan, and the United Kingdom. Figure 1.2 below depicts the total face mask usage in Asian regions Subsequently, many medical frontliners would need to wear or reuse the unsafe masks for a longer period. WHO targeted that PPE supplies will increase by 40 % monthly with an estimated of 89 million medical masks produced to deal effectively with COVID – 19 scenario (Singh *et al.*, 2020).

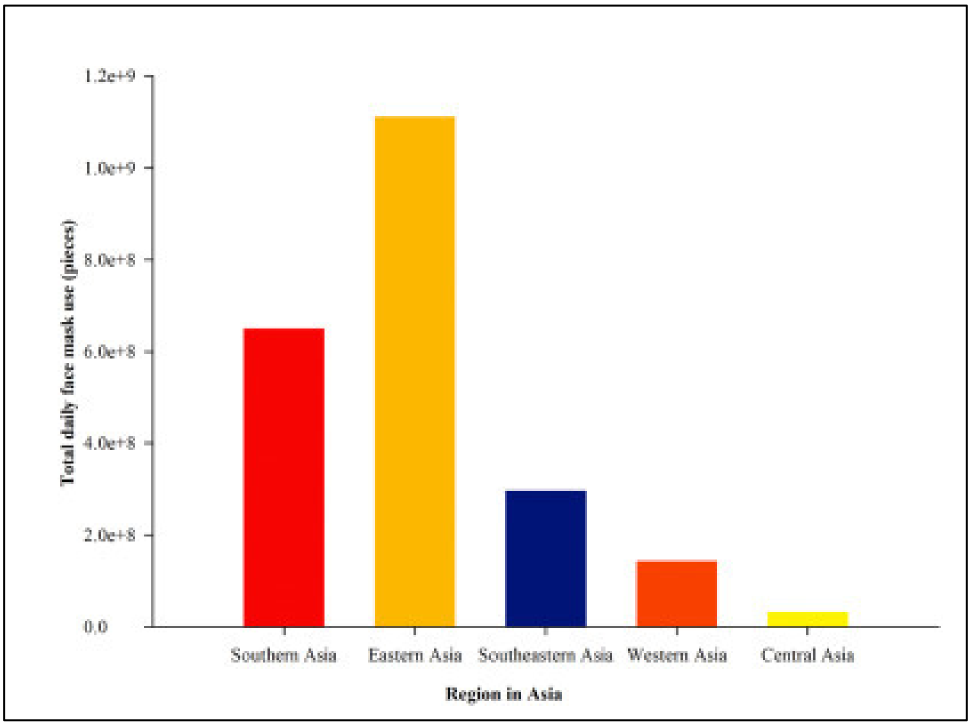

Figure 1.2 Total daily face mask usage in Asian regions (Sangkham, 2020).

Since disposable surgical mask and N95 respirators are mostly of single time use and need to be disposed, this has led to additional accumulation of solid waste due to mass utilization added by incline in number of infected cases daily. For instance, medical waste generated in Wuhan, China was nearly 240 tonnes daily during the pandemics (Singh *et al.*, 2020). In Malaysia, it has been reported that the total daily face mask use was estimated to be 7,049,901 (Sangkham, 2020). Agamuthu *et al.* (2020) reported that the incline of clinical waste in Penang, Malaysia was 27 %. Table 1.1 shows generation of clinical waste during COVID – 19 pandemics in various countries.

Table 1.1 Generation of clinical waste by countries/cities during COVID - 19 pandemic.

Cities/Country	Number of clinical wastes (including face masks) produced (kg/bed/day)	Percentage of incline in clinical waste (including face masks) produced (%)	References
Taiwan	0.9 – 2.7	-	(Agamuthu *et al.*, 2020)
Jordan	3.95	1000	(Abu-Qdais *et al.*, 2020)
Wuhan, China	0.6 – 2.5	213	(Yu *et al.*, 2020)
Bandung, Indonesia	2.2	17.1	(Agamuthu *et al.*, 2020)
Penang, Malaysia	0.4 – 1.0	27	(Agamuthu *et al.*, 2020)
Thailand	2.9	-	(Agamuthu *et al.*, 2020)
Mexico	2.0 – 2.2	-	(Agamuthu *et al.*, 2020)

Due to improper disposal management of masks, discarded masks become floating marine debris contributing detrimental effects to the environment. Medical masks made from plastics such as polypropylene has longer degradation rate and releases potentially hazardous substances (Fadare *et al.*, 2020). The surgical mask of single use once disposed will degrade into smaller pieces of particles of size 5 mm which are called as microplastics (Schnurr *et al.*, 2018). The degraded plastic particles will retain in oceans and can potentially be food source for marine animals. If similar situation continues, this will build up in the food chain in future years hence resulting in harmful health effects in humans. In addition, the improper disposal of surgical masks could also lead to virus outbreak as the respiration droplets containing viruses and contaminants can still retain and grow on the surfaces (Reid *et al.*, 2019). Improper handling of contaminated masks can lead to transmission of viral pathogens to waste collectors as well.

Figure 1.3 Images of improper disposal of surgical face masks into the environment (Fadare, 2020).

1.2 Problem statement

Wearing face masks especially during COVID – 19 pandemic situations have become a norm to prevent the spread of the virus chain. Due to shortages of disposable surgical face mask, the public has opted for an alternative to wear face masks made from fabric materials such cotton, microfiber, and microfiber blend (Konda *et al.*, 2020). In addition, the fabric materials are readily available household materials, cheap, soft, and breathable nature. However, hydrophilic nature of the fabrics has resulted the face mask to be wetted easily. Respiratory droplets may contain viruses and contaminants which can increase possibilities of virus transmission. For instance, cotton fabric is made up of cellulose fibers and has high number of hydroxyl (-OH) groups which makes it naturally be hydrophilic (Chauhan *et al.*, 2019). Moreover, it can also absorb liquids through capillary actions (Sharabaty *et al.*, 2008). (Ballerini *et al.*, 2012) discussed that the rough and porous surface structure of cotton fabric accelerates water spreading in between the fiber – matrix interface region. The usage of cotton fabric as protective layer in face mask eventually absorbs and collects more respiratory droplets.

High wettability increases the affinity of water droplets and other liquids to stain on the fabric surface. When this mask is worn for a longer period, the chances of contact between virus and wearer is increased. Besides, a used fabric mask becomes wet, contaminated with respiratory droplets, and dirty, hence it is not advisable to reuse the mask more than once (Parlin *et al.*, 2020). Even though the fabric face mask can be rewashed, however this can deteriorate the durability of the masks. Washing of fabrics under harsh conditions could reduce the mechanical strength of the fabric (Liu *et al.*, 2016).

To overcome the current problem, superhydrophobic surface modification on fabric-based face masks has been identified as an effective potential technique. Superhydrophobic surface provides self – cleaning properties as an added advantage (Manoharan *et al.*, 2019). However, most fabrication techniques of superhydrophobic surface coating have limitations which are time – consuming, utilization of high – cost materials, complicated instruments usage, rigorous processing conditions and low mechanical durability (Chauhan *et al.*, 2019; Pan *et al.*, 2019). Hence, implementing facile and effective techniques to construct superhydrophobic coating is necessary to be time and cost efficient. As stated above, fabric face masks are made of varies fabric materials which need to be surface modified to enhance the surface hydrophobicity.

1.3 Objectives

This specific research study comprises of the following objectives:

1) To investigate on facile techniques for superhydrophobic coatings.

2) To formulate coating composition on facile technique.

3) To evaluate and validate the superhydrophobicity modification on cotton, microfiber, and microfiber blend fabric face mask materials.

1.4 Scope of study

The focus of project is on the investigation of facile superhydrophobic coatings fabrication method. Dip coating and ultrasound irradiation techniques were investigated. The two fabrication techniques were screened to identify the potential technique. For validation, the superhydrophobic surface modification was done on varies mask materials such cotton, microfiber, and microfiber blend face mask materials. Characterization study by SEM and EDX were done to study the formation surface roughness and elemental composition present in the modified fabrics. Stability of coating, self – cleaning, stain resistance and durability properties of the modified masks were also investigated to evaluate the effectiveness of surface modification.

1.5 Thesis organization

A relatively simple and conventional organization of thesis has been applied in the presentation of project paper. Basically, the project report consists of five chapters which includes the introduction on overall project, literature review on related research, materials and research methodology, discussion on the results obtained and conclusions to summarize the entire project.

Chapter 1 covers the introductory part of the thesis. The research background of transmission of coronavirus and the importance of wearing face masks. Limitation of disposable and N95 respirators which causes environmental pollution was discussed.

Chapter 2 presents a literature review on the introduction to superhydrophobic surfaces, wetting phenomena, requirement for superhydrophobic surface modification. Advantages and limitations of fabric face mask was discussed. Techniques of fabricating superhydrophobic coating on fabric materials were also discussed.

Chapter 3 explains the materials and methodology of the entire project

Chapter 4 describes the observations, results, and discussion of the project outcomes. The screening of dip coating and ultrasound irradiation techniques were employed. Potential fabrication techniques were employed by measuring WCA. The potential technique and coating formulation was utilized for surface modification of microfiber and microfiber blend fabric surfaces. Surface morphology and elemental composition analysis was done by Scanning Electron Microscope (SEM) and energy dispersive X – ray spectroscopy (EDX) on the modified and pristine surfaces. Self – cleaning properties, stain resistance, stability of coating over time and durability were evaluated on modified and pristine fabrics.

Chapter 5 draws a conclusion of the study. Some recommendations are suggested for future research work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to superhydrophobic surfaces

When the surfaces have a water contact angle (WCA) between 150 ° and 180 °, the surface is superhydrophobic. In contrary, superhydrophobic surface refers to surface with rolling angle lower than 10 °. The contact angle refers to the angle formed between the intersection of solid – liquid and solid – gas interface. Superhydrophobic surfaces is commonly related to 'Lotus effect' phenomena. In 1997, Barthlott and Neinhuis documented the lotus effect phenomenon of superhydrophobic interface (Drexler *et al.*, 2008). Lotus effect phenomena tend to have a low water tilt angle on surfaces with low contact angle hysteresis (CAH), low adhesion, water repellence and antifouling properties. Lotus leaves, for example, consist naturally of hierarchical rough structure and hydrophobic wax coating. The rough nature of Lotus leaf hails from the microstructures formed by papillose epidermal cells while wax coating present due to epicuticular waxes. The water contact angle of lotus leaves is 164 ° thus exhibiting superhydrophobicity (Bhushan *et al*., 2009). In nature, wings of butterfly, spider silks, legs of water striders possess superhydrophobic surface phenomena naturally.

2.2 Wetting phenomena

Wettability is an important parameter that is determined by the static WCA. Wetting can be divided into main types which are super wetting and anti – wetting. Parvate *et al.* (2020) investigated that super wetting happens when the liquid completely wets the surface while anti – wetting occurs when liquid forms a sphere on the surface. When liquid is in contact on a solid surface, it either forms a spherical shape or completely wets the surface. By measuring the WCA of the liquid droplet, the surface hydrophobicity can be measured. WCA can be defined as the angle between a tangential to the liquid surface and the tangential to the solid surface at the line of meeting three phases. Figure 2.1 below shows the WCA of a water droplet on a solid surface.

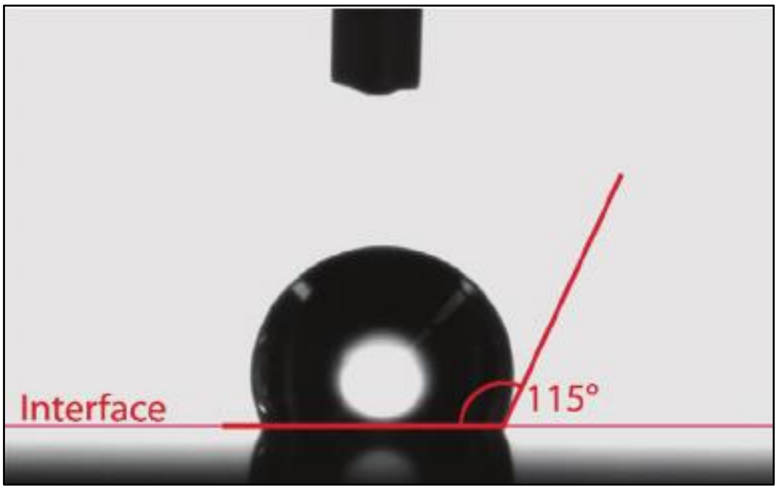

Figure 2.1 WCA of a water droplet on a solid surface (Schelcher *et al.*, 2011).

According to the WCA measurement, hydrophobicity may be classified into four distinct regimes. The WCA is found in the range of 0 ° < θ < 10 °, 10 ° < θ < 90 °, 90 ° < θ < 150 ° and 150 ° < θ < 180 ° where it can be categorized as superhydrophilic, hydrophilic, hydrophobic and superhydrophobic (Das *et al.*, 2018). Figure 2.2 below illustrates the four different regimes that characterizes the hydrophobicity of a surface.

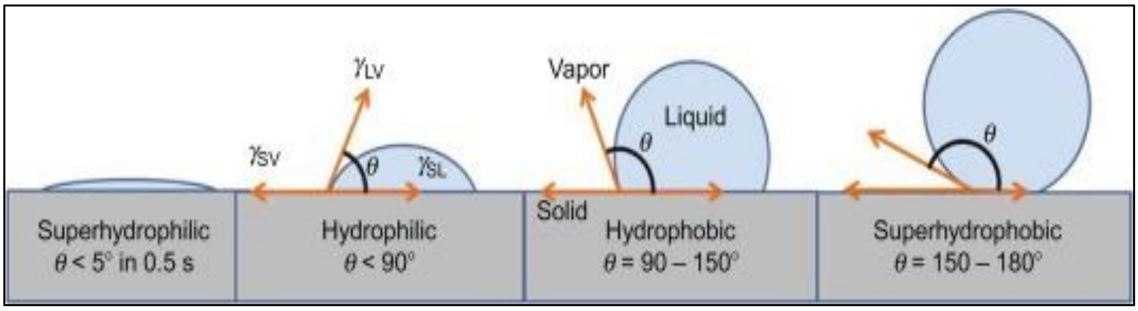

Figure 2.2 Illustration on formation of WCA on solids surfaces of different hydrophobicity (Chieng *et al.*, 2018).

2.3 Requirements for superhydrophobic surface modification

Fabrication of superhydrophobic surface is identified to be a promising and active research area. For a superhydrophobic surface, there are two vital requirements which are the surface should have low surface energy and rough. The two factors should exist simultaneously to achieve the state of hydrophobicity on the surface and high wettability.

2.3.1 Surface energy

Reduction of surface energy is one of the techniques to fabricate superhydrophobic surfaces. Techniques of lowering surface energy can be done either by modifying the surface chemistry of existing surface by producing micro/nanoscale structures or utilizing chemical film that is adhered to the surface. Common type of low surface energy materials utilized can be divided into two categories which are inorganic materials such as zinc oxide, titanium oxide, clay, and silica while organic materials include graphene, carbon nanotube, fluorocarbon, polymer, and bio – based materials.

2.3.2 Surface roughness

Surface roughening creates a heterogeneous (uneven) surface. In a rough surface, air bubbles are usually trapped in the upper and lower parts of the surface. Hence, when droplets of liquid are displaced onto the surface, it does not meet all the points in surfaces. The tendency for water to penetrate is low thus creating lower contact area with the surface and reduction the friction in between. The liquid droplet will slip off the surface easily. Figure 2.3 below depicts the trapped air bubbles between the liquid – solid interface when roughness in fabricated on the surface.

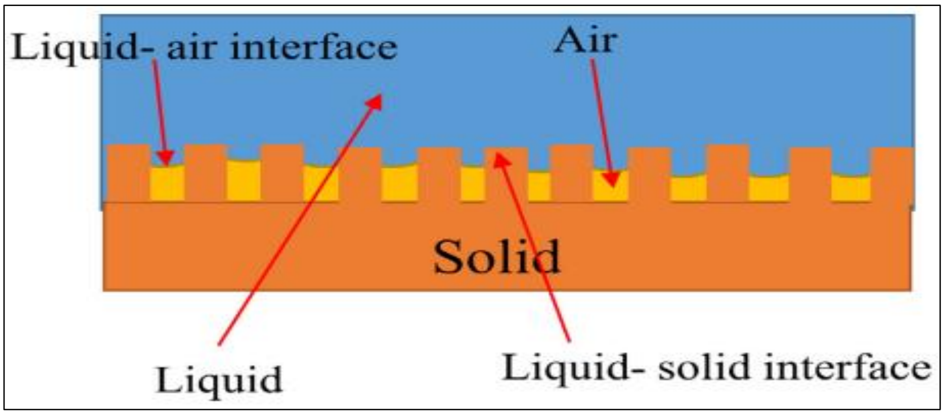

Figure 2.3 Trapped air bubbles in between liquid – solid surface(Manoharan, 2019) (Manoharan *et al.*, 2019).

The morphology and surface free energy are the two main factors that determine the hydrophobicity of the surface. A vital role is played by the surface roughness and surface free energy in the wetting properties of the surface. To achieve a superhydrophobicity condition, combination of low surface energy and surface roughness should be present simultaneously on the fabricated surface.

Young. (1805) introduced the first model in which the surface wettability was expressed in WCA of a water droplet.

$$\cos\theta = \frac{\gamma_{sv} - \gamma_{sl}}{\gamma_{lv}} \tag{2.1}$$

Based on equation 2.1, γ_{sv}, γ_{sl} and γ_{lv} refer to the interfacial surface tension while abbreviations S, L and V as solid, liquid and gas, respectively. The Young's equation is based on an ideal flat surface which shown in Figure 2.4 (a). Then, Wenzel proposed an equation where the surface roughness and surface energy associated with the contact angle (Agarwal, 2015). The Wenzel regime is described where the liquid is completely penetrates the gaps between roughness features that shown in Figure 2.4 (b). However, Wenzel model is only practical and workable for homogeneous surfaces.

$$\cos \theta_w = r \cos \theta \quad (2.2)$$

Based on equation 2.3, θw and r refer to the Wenzel's contact angle affected by surface roughness and roughness parameter, respectively. On the other hand, (Cassie, 1944) provided another model for the heterogeneous surfaces. In the Cassie-Baxter model, the liquid is assumed to be contacted with the solid only at the roughness tips and where the water droplet is assumed to stay on top of a layer of air which shown in Figure 2.4 (c).

$$\cos \theta_{CB} = f_1 \cos \theta_1 + f_2 \cos \theta_2 \quad (2.3)$$

According to equation 2.3, f_1 and f_2 are the surface fraction of constituent 1 and 2 while the θ_1 and θ_2 are the contact angle of constituent 1 and 2. When one of the constituents is air, the equation can be further simplified (Cassie, 1944).

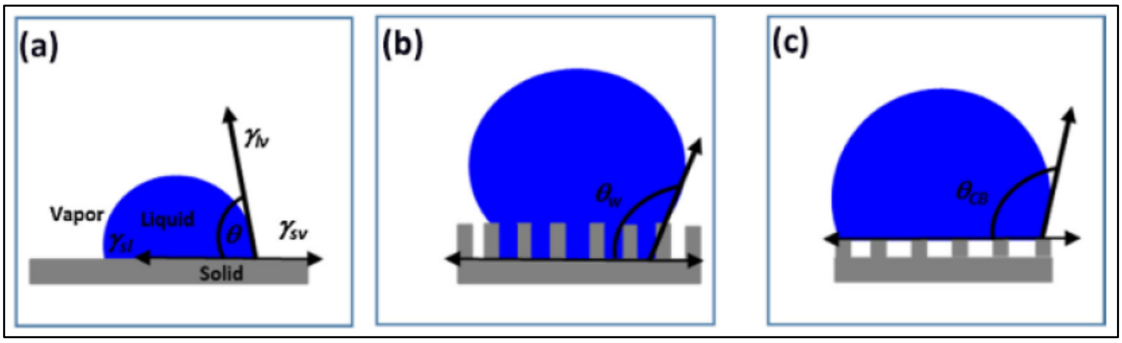

Figure 2.4 Three different wetting models (a) Young's model, (b) Wenzel model and (c) Cassie – Baxter model (Fihri *et al.*, 2017).

2.4 Rolling angle and contact angle hysteresis

Rolling angle is the lowest horizontal angle to the in which the droplet began to move on the surface and rolls on it. The angle should be less than 10 ° for superhydrophobicity state (Parvate *et al.*, 2020). Superhydrophobic surfaces must have very low CAH to acquire self-cleaning effects and low drag. The contact angle at the front of the droplet is denoted as a forward angle based on the figure below, while the contact angle at the back of the droplet is denoted as a forward angle in Figure 2.5. The difference between the advancing and receding contact angle results in CAH. The reasons for CAH to occur is heterogeneity and roughness of surfaces. When the surfaces have low CAH, the energy dissipation is reduced when droplets flow along a solid surface and brings along contaminants with them, providing self – cleaning ability which is known as lotus effect.

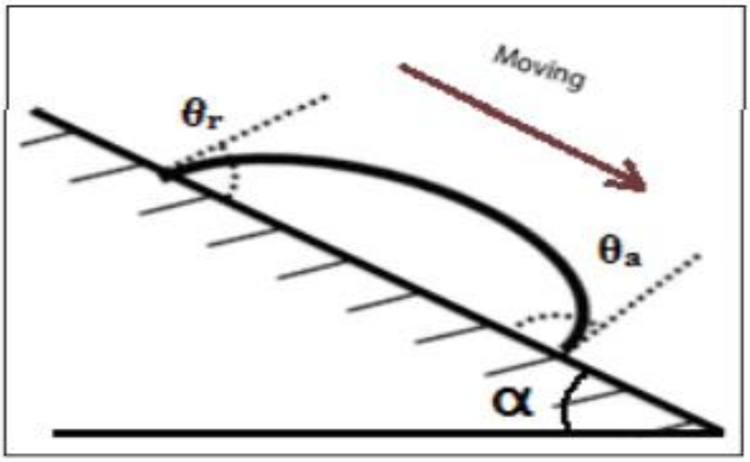

Figure 2.5 Advancing, θa and receding angle θr on a moving liquid droplet (Manoharan et al., 2019)

2.5 Fabric face mask

A global shortage, deteriorating environmental effects and poor solid waste management of N95 respirators and surgical masks have shifted the demand to reusable fabric-based face masks. Cotton, polyester, silk, and nylon are the common materials used to design as face coverings due to wide availability. There are several noted significances of using this reusable fabric masks. First and foremost, the lack of knowledge on disposal of surgical masks among the public. Surgical masks are generally incinerated however it will be difficult for public to follow the disposal method. Moreover, members of the public can wear the certified reusable fabric mask due to the shortage of surgical grade face masks which are highly needed by the medical frontliners whom require higher self-protection when dealing with infected patients. The certified fabric mask can be an effective alternate which also helps to prevent transmission of respiratory droplets. Fabric mask is washable and recyclable. This also helps to reduce the cost of buying masks. In addition, being environmentally friendly and sustainable are added advantages.

However, the current fabric mask still has some limitations. One of the limitations is the fabric mask lacks superhydrophobic properties caused by the respiratory droplets which contains the virus to remain on the surface. Secondly, filtration efficiency as a function of different aerosol sizes ranging from ∼10 nm to ∼10 μm scale sizes (Lin *et al.*, 2020). Hence, the bacteria and virus filtration efficiency need to be improved. Thirdly, the mask cannot be used repeatedly since the absorbed viruses might be active on the surfaces.

Transmission of coronavirus can hardly spread by itself and requires respiratory droplets as virus carrier. Fabric mask has the highest penetration of particles at 95 %

followed by disposable surgical face masks at 44 % and N95 respirators at 0.01 – 0.1 % (Wibisono *et al*., 2020). The risk of contamination is highest for fabric mask as it might originate from self – contamination because of repeated usage.

2.6 Techniques of fabricating superhydrophobic surface on fabric materials

Superhydrophobic surface modification on fabric substrates are achieved by introducing two main factors which are coating should have micro/nanostructures which increase surface roughness and low surface energy materials for instance hydrocarbons or fluorocarbon compounds. The common techniques for creating rough coatings for fabric – based materials can be classified into two main parts which are wet chemical and dry physical methods which is illustrated in Figure 2.6.

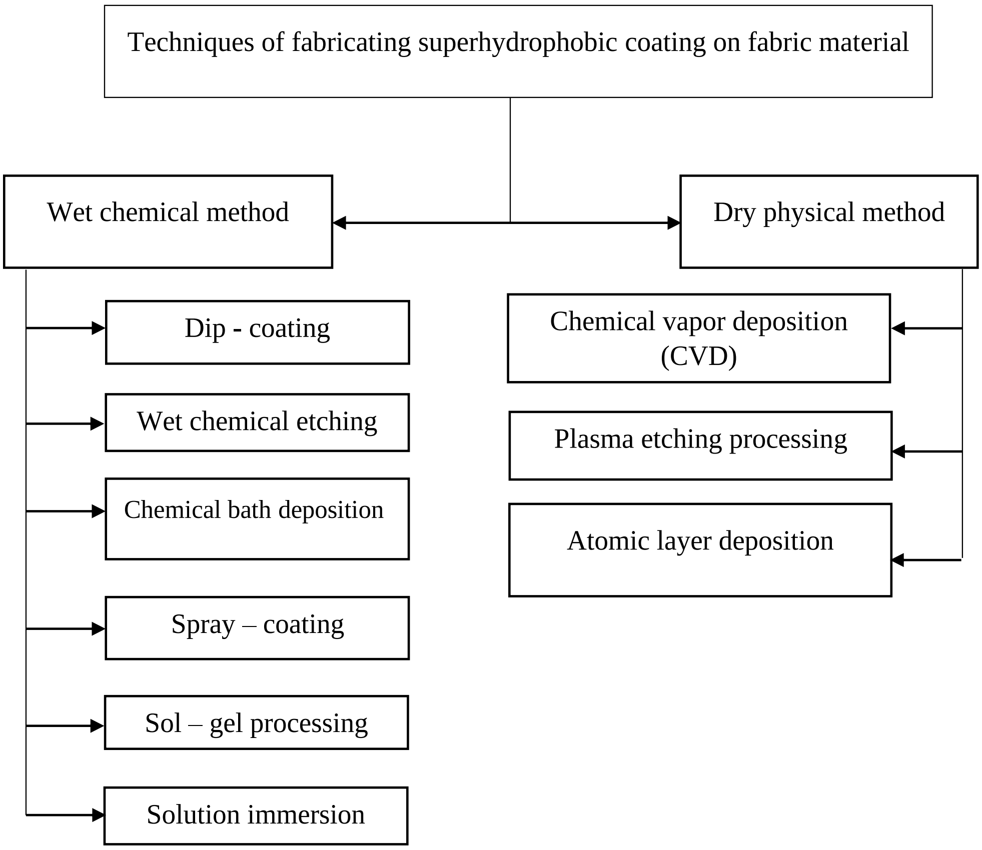

Figure 2.6 Techniques of fabricating superhydrophobic coating on fabric material.

2.6.1 Wet chemical methods

Dip coating is a method to coat the surface of fiber with hydrophobic nanoparticles layer for instance titanium oxide (TiO_2), silicon dioxide (SiO_2) and zinc oxide (ZnO). During the process, the fiber is dipped into the material solution for a particular period. Later the immersed fiber is taken out slowly to enable the coating layer to bind to the surface. The coated fiber is then allowed to dry to ambience temperature for a specific time. Wang *et al.* (2015) studied dip coating technique on cotton fabric where the fabric was dipped into solution containing SiO_2 nanoparticle and dodecyltrimethoxysilane (DTMS). Immobilization of SiO_2 nanoparticles on the fabric surface with the help of self-polymerization of dopamine. The WCA was found to be 153 ° for the superhydrophobic fabric surface. High oil – water separation efficiency, chemically stable to acid/alkali solution and durable. To enable a thicker coating layer to be adhered, fabric is dipped for several times.

On the other hand, Pan *et al.* (2019) experimented dip coating technique on coating fabric but incorporated modification through in situ growth of copper stearate ($CuSA_2$) followed by coating in polydimethylsiloxane (PDMS) solution. Presence of $CuSA_2$ constructed nanostructures while PDMS provided the adhesion layer to the fabric. The prepared fabric had about WCA of 158 ° as shown in Figure 2.7. Furthermore, the superhydrophobic coated fabric exhibited good stability towards strong acid and alkali solutions. Excellent mechanical abrasion, oil – water separation efficiency (greater than 96 %) and anti – fouling properties were obtained in the modified fabric. No complicated procedures were done, and the materials utilized are of low cost and environmentally friendly.

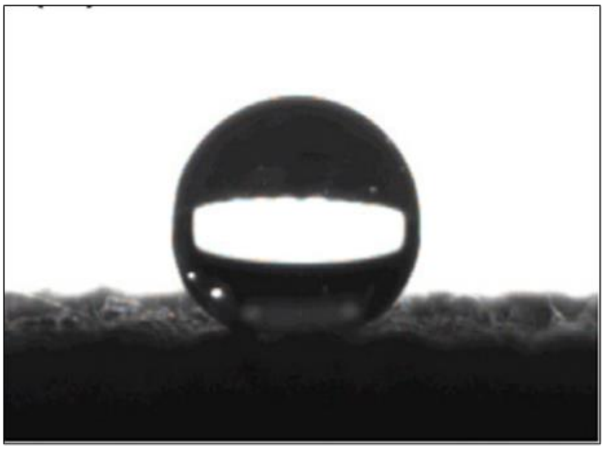

Figure 2.7 SEM images of static WCA on the coated cotton fabric (Pan *et al*., 2019).

Chemical etching is a technique of fabricating surface roughness via nanostructures growth. Xue *et al.* (2014) investigated chemical etching technique by coating PDMS on polyethylene terephthalate (PET) textile fabric surface. PDMS altered the surface roughness together lowered surface energy of the fabric. The chemically etched textile recorded a WCA of more than 150°. Etching does not only reduce the area of contact in between the etched PET textile and water droplets but also enhances the superhydrophobic properties. The surface of chemically etched fabric had superhydrophobic surface, which was slippery, excellent durability, and washing resistance. Synthesis of chemical etching on PET textiles were shown in Figure 2.8.

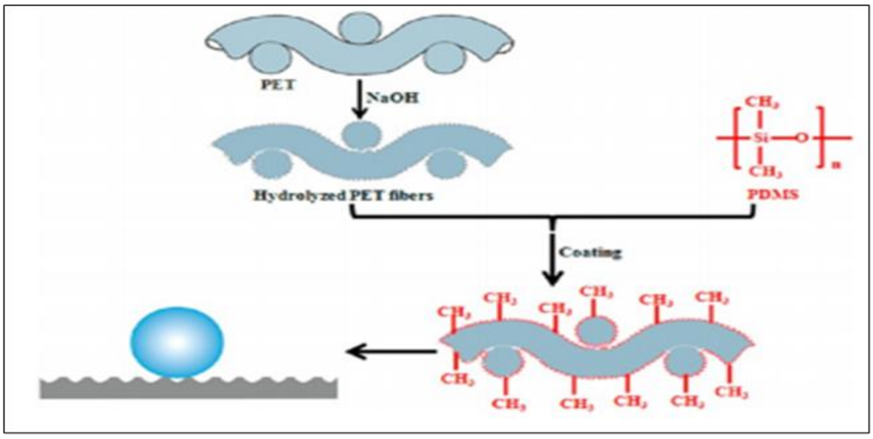

Figure 2.8 Fabrication of PET textiles by chemical etching (Xue *et al*., 2014).

TiO_2 nanoparticles were utilized by Huang *et al.* (2015) as superhydrophobic coatings on cotton fabric by in-situ growth via a chemical bath deposition process. The cotton was immersed in the reaction mixture for several hours at temperature of 80 °C. Then a self-assembling process of fluorodecyltriethoxysilane (PTES, F17) was carried out to construct a superhydrophobic TiO_2 coated fabric as shown in Figure 2.9. The prepared fabric recorded a high WCA of 160 °. The prepared fabric was promising as it showed excellent efficiency in oil – water separation, self – cleaning performance and anti – ultraviolet (UV) radiation.

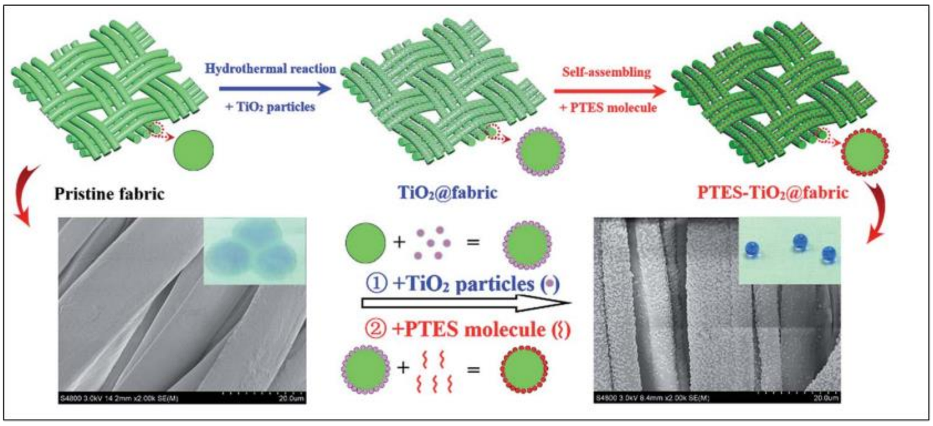

Figure 2.9 Illustration of fabrication of cotton fabric by chemical bath deposition (Huang *et al*., 2015).

Sol – gel is one of the promising facile techniques which synthesizes gels and nanoparticles. Gurav *et al.* (2015) investigated an easier approach of implementing a single step sol – gel method. Silica particles were modified with methyl groups using methyltrichlorosilane as a modifying agent to fabricate superhydrophobic coatings on. The WCA of water droplet on the coated surface was 153°. The synthesized superhydrophobic coating displayed excellence in self – cleaning and water repellency test.

Immersion techniques is one the cheapest yet effective way to fabricate superhydrophobic coatings. Chauhan *et al.* (2019) performed a modification on cotton fabric by immersing the received fabric in Hexadecyltrimethoxysilane (HDTMS) solution for five hours. HDTMS was used as the low surface energy material in the research. After modification, the WCA was found to be 157° as shown in Figure 2.10 and rolling angle less than 10°. In addition, the superhydrophobic cotton fabric exhibited better mechanical durability, self – cleaning, resistance to stains, chemically stable and thermally stable.

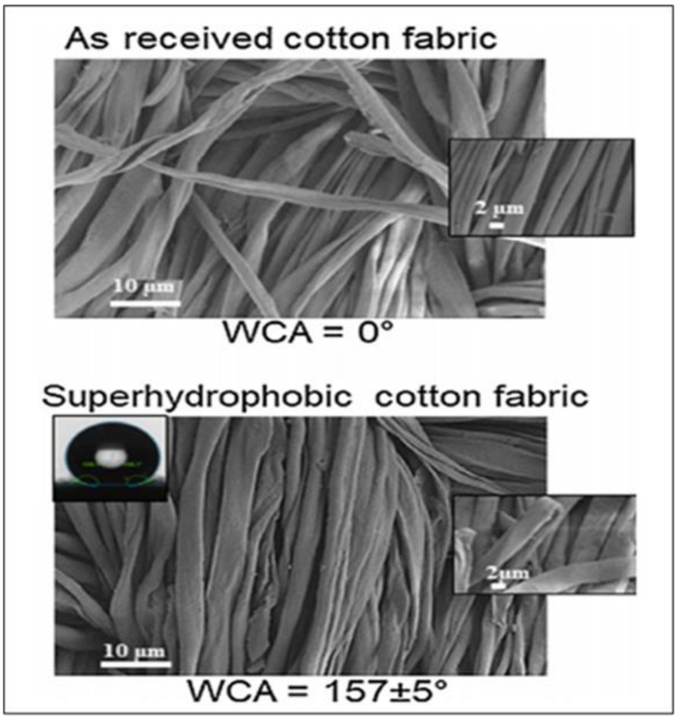

Figure 2.10 SEM images of received and superhydrophobic cotton fabric.

In spray coating, the prepared coating material in form of liquid is filled into a gun and sprayed over the fabric with aid of pressurized air. Ma *et al.* (2015) developed a facile method by spraying polyacrylate emulsion on leather surface. Then, followed by spraying hydrophobic silica dispersed in ethanol solution. The adhesion action between polymer emulsion and silica nanoparticles constructed micro/nano – structures with low surface energy. Figure 2.11 below shows the illustration of spray coating of polyacrylate emulsion as coatings on leather The maximum WCA in the prepared leather coatings was 170.3° with four layers of coatings sprayed as shown in Figure 2.11**.** The fabricated leather displayed better self – cleaning performance and water repellency. Yazdanshenas *et al.* (2013) studied on fabrication of superhydrophobic coating on cotton fabric by silica nanoparticles synthesis via n - octyltriethoxysilane (OTES) and tetraethylorthosilicate (TEOS) where the WCA measured was 152.8 °. The modified fabric displayed excellent durability though repeatedly washed.

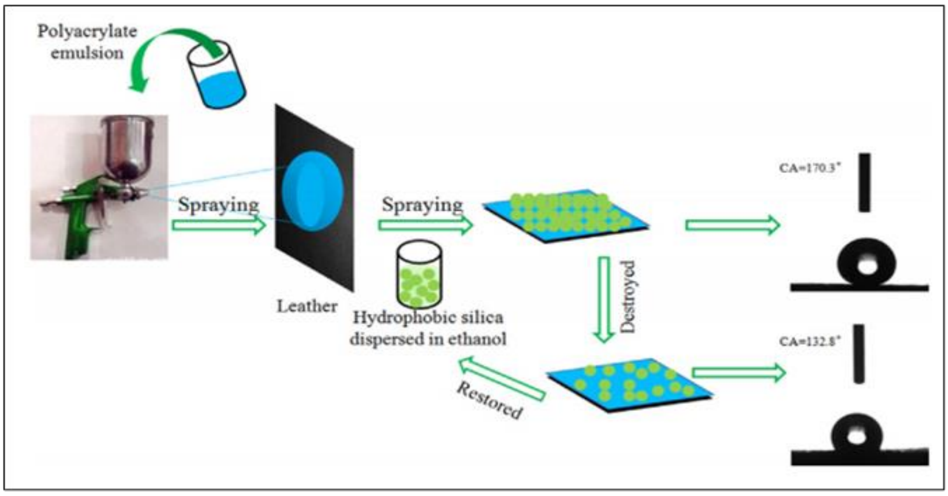

Figure 2.11 Illustration of spray coating on leather (Ma *et al*., 2015).

2.6.2 Dry physical methods

Chemical vapor deposition (CVD) is a dry process that enables chemical and physical fine structure tuning to be deposited on a substrate in the form of non-volatile film through gaseous reactant reaction. Zhou *et al.* (2013) studied modification of cotton fabric by combination of polyaniline and fluorinated alkyl silane. The prepared fabric obtained a WCA of 156°. It showed high durability and chemical stability towards strong acid and alkali solutions. High efficiency in oil – water separation which is higher than 97.8 %. In result, the method consumes lesser time.

Kwon *et al.* (2014) employed oxygen plasma-based nano structuring method with a subsequent coating with a low-surface-energy material to produce a single-faced superhydrophobic lyocell fabric through oxygen plasma etching and maintaining its inherent high moisture absorbing bulk property. Recorded WCA was more than 160°. The achieved asymmetric wetting properties on a fabric layer would be significant and relevant for applications that require water repellency and self-cleaning properties.

Xiao *et al.* (2015) investigated superhydrophobic surface modification on wool fabrics by depositing aluminum oxide (Al_2O_3). The deposition was carried out by exposing the fabric to water and trimethylaluminum at temperature of 80 °. The Al_2O_3 nanoparticles increased the surface roughness and lowering the surface energy of the coated fabric. The WCA of the prepared fabric was 160 ° compared to the original fabric which had WCA of 130°. Furthermore, the superhydrophobic coated fabric exhibited better durability.

Although several fabrication techniques on fabric materials are available but simple and facile methods are of high in interest. Together, vital properties such self – cleaning, wettability and durability are important to be present in the fabricated face mask. Hence, dip coating and ultrasound irradiation techniques were opted as potential methods for preparation of formulation and screening on fabric face masks. In addition, both the techniques are of low cost, does not require complex procedures, specific equipment of high cost, easy deposition and environmentally friendly. Table 2.1 below summarizes available superhydrophobic fabrication techniques on fabric materials.

Table 2.1 Summary of superhydrophobic fabrication techniques on fabric materials

Technique	Type of fabric	Type of roughness formation	WCA (°)	References
Dip coating	Cotton	Nanoparticle coating	153	(Wang *et al*., 2015)
Dip coating	Cotton	Nanoparticle coating	158	(Pan *et al*., 2019)
Wet chemical etching	PET	Nanoparticles growth via etching	>150	(Xue *et al*., 2014)
Chemical bath deposition	Cotton	Deposition of nanoparticle film	160	(Huang *et al*., 2015)
Sol – gel method	Cotton	Synthesis of sol – gel and nanoparticles	153	(Gurav *et al*., 2015)
Solution immersion	Cotton	Nanostructures coatings via immersion	150	(Chauhan *et al*., 2019)
Ultrasound irradiation	Cotton	Nanoparticle growth via ultrasound	152	(Yazdanshenas *et al*., 2013)
Spray coating	Leather	Nanostructures by spraying	170.3	(Ma *et al*., 2015)
Chemical vapor deposition	Cotton	Nanostructure growth via polymerization	156	(Zhou *et al*., 2013)
Plasma etching	Lyocell	Nanostructures growth by etching	>160	(Kwon *et al*., 2014)
Atomic layer deposition	Wool	Deposition of nanoparticle layer	160	(Xiao *et al*., 2015)