Evaluation of contact angle of water proof coated fabric made from melt-blown polyester non-woven and acrylic polymeric materials

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Abstract. Water proof fabrics are hydrophobic in nature. Contact angle measurement is a characterization technique used to gauge how well the water proof fabrics designed and developed is useful in preventing seepage of water through fabric surface. The water-proof coated fabric was developed using polyester non-woven fabric made by melt-blown process as base material. Binder made using acrylic polymer was used for preparation of the water-proof coated fabric using layering technique. Different layer configurations like single and double layers was used to develop the non-woven coated fabric. The developed fabrics was subjected to morphological studies using scanning electron microscope. The water proof coated fabrics was also analyzed for its static contact angle measured using rame-hart instrument. The contact angle was measured for both single layer and double layer water proof fabrics at different positions. The contact angle range was reported from 96°-58° for single layer water proof coated fabrics. However, for double layer water proof coated fabrics the contact angle reported was in the range of 82°-68°. The contact angle changes for different angle of measurements and orientations. The results show that contact angle is not only a function of hydrophobic character of polyester non-woven fabric base material but also depends on the process parameters like number of layers, the thickness of coating, coating technique and the moisture levels that the acrylic polymer binder coating material absorbs the liquid from its surface. The forces acting on the surface of the coating material mainly influences the contact angle of the coated surface. The water repellency increases with the increase in static contact angle.

1. Introduction
Polyester non-woven fabrics are usually made through melt blown process. The fiber itself is characterised by inbuilt hydrophobic qualities. However, when used and coated with an acrylic polymer binder which can take up 1-2 % moisture, the nature of the hydrophobicity can change. The fabric which is highly crystalline will get masked with a layer of acrylic polymer binder and shows good resistance to chemicals and water. These properties find many applications due to its water-proof characteristics. In this study, Non-woven melt blown polyester fabric is coated with acrylic polymer...
binder to develop a water proof fabric. The developed water-proof fabric is observed for its morphological changes on its surface and its contact angle is measured by sessile drop test using goniometer. The characteristics of water-proof fabric is analyzed and discussed in this research work. The water-proof fabric initially behaves like a hydrophobic surface, however when contact angle is measured at different orientations and positions of the sample and over a period of time the contact angle changes to hydrophilic values. This clearly indicates the influence of coating parameters and coating process and surface roughness directly affect the hydrophobic characteristics of the sample.

2. Literature survey
Lin et al. (2018) reported the hydrophobicity of composite insulators are dependent on characteristic features of water droplets like the shape of the water droplets, the mean eccentricity values and other values which were studied using spray test method [1]. Huhtamäki et al. (2019), carried out research on the wetting surface characterization by measuring receding contact angle and advancing contact angle by varying the volume of the water droplets. They concluded that the tests take about 15-20 minutes to complete the measurement of one pair of water droplets [2]. Wolf et al. (2009), has reported his findings on modelling and simulation using lattice-Boltzmann equations and has found that there exists a interaction between the solid walls and particles [3]. Prajinto et al. (2016), In their experiments on stainless steel 304 metal surface using Nano fluid drops mixed with zirconia nanoparticle was able to measure the contact angle. The contact angle was measured with demineralized water on different surfaces with varying surface roughness values [4]. Mohammad Jafari and Jongwon Jung, has demonstrated the measurement of static and dynamic contact angles using highly smooth and glass micromodel for chemically homogenous conditions. They infer that the interface water- carbon dioxide bubbles present on the flat surface effects the contact angle [5]. Gao (2009), Reported the findings of measuring contact angle for fabric surfaces made from cotton and polyester fibers. The fabric surface was treated with silica sol developed by hydrolysis and condensation of tetraethoxysilane. Alkaline conditions was used for depositing the silica on the fabric surface. The coated fabrics were again treated with HDTMS (hydrolysed hexadecyltrimethoxysilane) to impart hydrophobic characteristics to the fabric. They attributed the super hydrophobic characteristics of the fabric was due to presence of HDTMS [6]. Wang et al. (2019), used coating solutions which were made from alkysilanes composed of different chain lengths. They inferred from the superhydrophobic characteristics was more in coated surfaces having higher surface roughness and longer chain lengths [7]. Li, et.al (2016), reviewed the behaviour of fabric surfaces and reasoned out the causes for superhydrophobic characteristics. Their conclusion was based on the study conducted on the morphological characteristics of coated surfaces and their anti-wetting properties. Creating such wettability features on fabric surface finds numerous applications ranging from anti-bacterial and flame retardant finishes [8]. Birzu et al. (2017), carried research on the wetting characteristics of wool textiles which were treated with zinc oxide and titanium dioxide. The wool textile was pre-treated with oxygen plasma before depositing zinc oxide and titanium dioxide on the surfaces of samples. Contact angle was studied using Cassie-Baxter model and the reported contact on wool textiles showed reduced values than the pure samples [9]. Volkov et al. (2003), has studied the effect of hydrophilic characteristics of fibers and polymer binders in non-woven. They have inferred that the increase in fiber hydrophilicity is due to the increase in the capillary size in the fabric samples [10]. Ghimire et al. (2017), found that the air-plasma treatment was more effective to impart surface wettability and absorbency values. Low temperature plasma is found to be successful in creating hydrophilic functional groups on fabric surfaces. The contact angle reduced in treated samples and absorbency values increased [11]. Musadeeq et al. (2017), carried out contact angle and tilting angle measurements of samples made from Nano-filament polyester fabrics. Hydrophobic finish was applied on these samples prior to measurement of their contact angle and tilting angle. They concluded 3 % finish samples showed the best results [12]. Masloompour, et al. (2007), studied the horizontal wicking characteristics using electrical resistance technique on untreated and finished cotton fabrics. They conclude the finish chemicals directly affect the surface characteristics and water droplet penetration.
rate. They also attribute the existence of numerous solid-vapour regions with different surface tensions on finished fabrics [13]. Salukhe (2018), studied using shape analysis method for analysing the contact angle on surfaces coated with CuS (Copper sulphide) thin films [14]. Zimmermann et.al (2009), used a new technique to study the wetting characteristics of super hydrophobic surfaces. They study the shedding angle which is defined as the ability of the surface to repel droplets of water when impacted on the fabric surface. This water shedding angle study is a new addition to the existing method of characterisation of wetting characteristics of surfaces [15]. Kusuktham (2010), used vinyltriethoxysilane as the surface modification agent for polyester fabrics. The emulsion was applied using padder and cured at 150° C for a duration of 15 minutes. The coated fabrics showed increased contact angle values as compared to uncoated ones. The deposition of silicone particles helped to enhance the surface roughness values and resulted in increased contact angles [16].

2.1. Concept of super hydrophobicity

Contact angle is the angle formed when a fabric surface comes in contact with the liquid forming fabric-liquid interface (Figure 1). The tangent drawn to the droplet profile at the contact point (as shown in Figure 1) gives the measure of the contact angle. Based on these measures the surfaces are classified as Hydrophilic, Hydrophobic and super hydrophobic type. Super hydrophobicity is a phenomenon where the contact angle of a surface exceeds 150°. Super hydrophobicity in fabric surfaces means that a drop of water when sprinkled on the surface will exhibit a very low roll-off angle. This behavior is due to the occurrence minimum contact angle hysteresis and reduced contact area between fabric surface and liquid. In nature we find many examples which mimic the different contact angles. Some include like the lotus leaf effect where the water repellency is due to the low adhesion and super hydrophobicity phenomenon (>160°)(Figure 2). Few of the other plants that show super hydrophobicity are Lady’s Mantle and Water ferns. The contact angle hysteresis is well described by Cassie–Baxter and Wilhelm Barthlott. They found the lotus leaves and leaves of Indian canna possess tubular-platelet type waxy crystals. It is the waxy projections on the leafs surface which make the surface super hydrophobic. They also found the presence of Nano or micro topography on surface of substrate coupled with low surface energy contributed to super hydrophobic characters. The surface of textile fabrics are highly hydrophilic due to the presence of polar (OH) groups. The surface is also varied by adapting the process of weaving, knitting and non-woven to vary the fabric structures and fabric surfaces. In order to make the fabric surface hydrophobic many techniques are also employed for modifying the surface like etching, plasma treatment, deposition techniques, etc. Coating of the fabric surface with Nano-materials, Phase change materials and Acrylic polymer binders will also render the surface hydrophobic [2][7][8][9].

2.2. Theory of contact angle

The 3 phase contact line or the triple point is a well-known term describing the line where solid, liquid and vapour phases of materials coincide with each other. If a contact angle is reported less than 90° means the surface is assisting the spread of the liquid over a large area. The opposite ensures the
surface to be super hydrophobic. In case of complete wetting, the contact angle is reported to be 0° (Figure 1). Thomas Young, working on the contact angle concept in 1805 describes the occurrence of contact angle of an ideal solid surface is due to the existence of mechanical equilibrium and action of surface tension normally referred to as interfacial tension. He describes the contact angle as the cosine function of the surface tension existing under the surface interactions \[\gamma_{lv} \cos \theta = \gamma_{sv} - \gamma_{sl}\] (1).

\(\gamma_{sl}, \gamma_{lv}, \gamma_{sv}\), and is the solid-liquid, liquid-vapor, and solid-vapor, surface tension, \(\theta\) represents the contact angle. This equation (1) is known as Young’s equation.

3. Materials
In this research work, melt blown polyester non-woven fabric is used as the base material to develop water-proof fabric. Polyester by its nature is a synthetic fiber having its characteristic hydrophobic property. Acrylic polymer binder on the other hand is used as a coating fluid to develop the polyester melt blown non-woven fabric. The polyester non-woven fabric has a weight of 46 grams per square meter with randomly laid web configuration. The fabric has a thickness of 0.4 mm. The acrylic polymer binder has a characteristic greyish color, thixotropic fluid properties, having the nature of solidifying after application over a period of time and gets hardened with excellent tensile strength values. The mixed density is reported at 1.75 g/cc with a curing time of 30 minutes at 30º C. The acrylic polymer binder is reported to take up 1.2% water after application and would in all probability reduce the hydrophobic characteristic of polyester base fabric used in this work. Table 1 shows the technical specification of acrylic polymer binder used in this work. Figure 3, 4, 5 shows the raw-materials used in development of water-proof coated fabric [11].

| Description                  | Specifications                                      |
|------------------------------|-----------------------------------------------------|
| Appearance                   | Greyish color                                       |
| Fluid Type                   | Fluid with thixotropic properties                   |
| Strength(Tensile)            | 1.2 $\text{N/mm}^2$ (at 1.0 mm thick) (ASTM D 882)  |
| Density                      | 1.75$\text{g/cc}$ (Consistency at brushable level)  |
| Time for curing              | At 30º C – 30 minutes                                |
| Concrete - Adhesion bonding  | 2.0 $\text{N/mm}^2$                                 |
| Absorption of water          | 1.2% (ASTM D 570)                                   |
| Toxicity                     | Found to be Non-Toxic                               |
| Elongation % - Flexibility   | 40                                                  |
4. Methodology
Polyester non-woven fabric which is by nature a hydrophobic fiber is first laid in sheets on a flat table. Binder made from acrylic polymer is then padded through roller coating process. The coated fabric is then dried, followed by curing process, finally resulting in formation of the water proof fabric. The flow chart explaining the methodology followed is shown in Figure 6. The developed fabrics were subject to surface morphology analysis and measurement of contact angle of the samples [10].

4.1. Application of acrylic polymer binder on melt blown polyester fabrics
The samples for this study were prepared using the raw materials described in previous sections and coating using manual roller coating process. The polyester melt-blown non-woven fabric was padded with polymer acrylic binder followed by drying and curing. The base layers were prepared in 2 configurations namely the single layer and the double layer ones. Care was taken to ensure there was no wrinkle formation on the layers. The coating thickness was controlled by ensuring even number of strokes given after every padding cycle. The coated fabric were dried in lab conditions and then taken
for curing upto 48 hrs in an oven. A thick flexible sheet was developed as shown in Figure 7. Table 2 gives the details and coating parameter that was measured after the development of water-proof fabric [12][14][16].

![Figure 7. Developed water-proof coated fabric](image)

### 4.2. Thickness and weight measurement methods for coated and uncoated samples

ASTM D1777 (American Society for Testing and Materials) standard is used to measure the thickness of coated and uncoated fabrics. The instrument consists of presser foot which has a diameter of 9.5 mm and weighing 143 gms. The thickness is sensed by the digital set-up in the apparatus and then processed to display the results according to the English or metric system. The method is very simple and quite accurate.

ASTM D3776 standard is used to measure the thickness of coated and uncoated fabrics. Sample sizes of 10” x 10” is cut and then their weight is measured on an electronic balance. The weight of the samples is converted to SI units and results are expressed in Grams per square meter.

| Sr No | Description of sample          | Sample weight (GSM) | % Coated | Thickness (mm) | % Change in thickness |
|-------|--------------------------------|---------------------|----------|----------------|-----------------------|
| 1     | Uncoated -Non-woven fabric     | 46                  | ---      | 0.4            | --                    |
| 2     | Coated fabric – Single layer   | 1163                | 96       | 0.72           | 44                    |
| 3     | Coated fabric – Double layer   | 1102                | 92       | 1.15           | 65                    |

### 4.3. Evaluation of hydrophobicity of the surface by measuring static contact angle

Contact angle of the coated fabric surface is measured using the sessile drop method. The surface and liquid form an interface and results in contact angle. The measurement of this angle is carried out by using a goniometer. This instrument measures a 2-Dimensional angle resulting due to surface interaction of coated fabric and water droplets. The procedure involves the measurement of contact angle after a single drop of liquid is dropped on the coated surface. This static contact angle method is carried out using ramehart instrument (Figure 8 & 9) as per ASTM D7490-13 standards which is the test standard for measuring the contact angle and surface tension of solid coating substrates and pigments [17].
The Instrument is connected to a camera and an image capturing LabVIEW software. The contact angle is measured by moving the camera at different orientations in a circle around the liquid drop. The instrument gives both the left and right angles and the average of left and right is reported. The instrument uses small strips (5 x 1 cm) of samples which are prepared and placed on a microscope slide for observation. After placing the glass slide on the stage, a 5-μL (Micro liter) drop of liquid is pushed using a syringe on the fabric surface. This process is continued at different intervals, orientation and direction of the camera.

4.4. Scanning electron microscope (SEM)
Scanning Electron micrographs as shown in Figure 10, 11 and 12 was captured at an electron acceleration voltage of 5.0Kv(Kilo voltage). The resolution and magnification of the images was set at 500 X(Times) with the view filed images being taken at 415 μm. However, the distance between the objective lens and the sample was adjusted to capture the best possible micrograph of the sample surface. A scale bar of 100 μm was used for all the three figures. The polyester non-woven fabric and coated fabrics was scanned to analyse its surface features using TES SCAN VEGA – 3 SEM Instrument (Make of the Instrument). The instrument is incorporated with a facility to emit electrons by using a tungsten filament. Different modes are available which can be used to capture images under different resolutions and magnifications. The sample were mounted and its surface features was captured under different magnifications and resolutions [13][15].

5. Results and Discussion
5.1. Morphological changes of specimens
Referring to Scanning electron images (SEM) (Figure 10, 11, 12), the SEM image of polyester non-woven fabric shows presence of air pockets which are randomly distributed. In the single layer surface image on can observe the presence of rough surface with uneven marks of fibers. This surface features increase the contact angle. However, in the double layered scanning electron micrograph, the reported contact angle is relatively lower by 10°. The double layer coated surface appears to be more uniform as compared to other images. Thus, the present coating technique and method will also influence the degree of hydrophobicity (Figure 13) [4].
The samples, both single layer and double layer water-proof fabrics show hydrophobic properties. However, they are not indicating any superhydrophobic characters. The measurement of contact angle at different orientations and directions of the recording camera is shown in Figure 13. The main reason for this variation with respect to time may be due to the type of fabric surface created by the coating method followed and the presence of acrylic polymer binder itself. The fabric surface created using single layer non-woven material showed more uniform surface roughness relative to double layer fabric surface (see SEM Images) resulted in higher contact angle. There is no evidence of hydrophilicity of the developed fabric surface in both the fabric samples. There is also a drop in contact angle when measured after some time. Figure 13 also shows the drop in the contact angle after measuring at different time period.

6. Conclusion
In this study the water proof coated fabrics demonstrate the hydrophobic characteristics initially even though the measured contact angle cannot be classified as a fabric substrate with superhydrophobic characteristics. The developed fabric surface due to the coating parameters, coating method followed has resulted in surface roughness of varying degrees of contact angle as seen in scanning electron
micrographs. In conclusion the fabric surface and the coating material properties highly influence the contact angle of a water-proof fabric.

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