NATURAL PIGMENT BASED ON ORANGE FRUIT FOR SUPERHYDROPHOBIC DYEING IN COTTON KNITTED TEXTILE APPLICATION

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***Abstract*— The study of pigment based on orange fruits for superhydrophobic dyeing in cotton knitted textile applications. The synthetic pigment is replaced by orange fruit pigment to apply into cotton knitted textile applications in this study. The main objective of this study formulates and synthesis the orange fruit pigment for superhydrophobic dyeing and evaluate the mechanical and physical test for superhydrophobic dyeing in cotton knitted textile applications. The orange fruit pigment was extracted and used to dye the cotton knitted fabric in different concentration of orange fruit pigment which is 20%, 40%, 60% and 80%. Then, the cotton knitted fabric is sprayed by the superhydrophobic solution. The component of dyeing was involved pigment, solvent, binder, additive, and hardness. All the dyeing components have been synthesis to apply superhydrophobic dyeing in cotton knitted textile applications. A few physical and mechanical tests are conducted on the sample. In the water droplets test (ASTM D5946-04), the 60% of orange fruit pigment had the excellent superhydrophobic properties which are 151˚ of water droplet surface contact angle. For the colourfastness to crocking test (ASTM D5053), all the fabric had the same dry crocking result which is reading of 5. In wet crocking result, the 80% of orange fruit pigment that applied to the fabric without superhydrophobic coating had a highest degree of colour transferred which is reading of 3.5. For the bursting strength test (ASTM D3786), the 80 % of orange fruit has the highest bursting strength which is 42.97 kPa for coated fabric and 41.78 kPa for fabric without superhydrophobic coating. For the abrasion resistance test (ASTM D4966-98), the 0% of orange fruit pigment that applied to the fabric without superhydrophobic coating has the highest percentage of weight loss which is 21.05% in 2500 times of rubbing. The combination of the concentration of 60% orange pigment and solution can be applied on cotton fabric due to its good mechanical and physical effect on the cotton knitted fabric surfaces and produced the optimum for the ability of superhydrophobic characteristic.**

Keywords- Pigment, Orange fruit, Superhydrophobic, Cotton knitted fabric, Dyeing

I. INTRODUCTION

In this research, we study the organic pigment based on the fruits to replace synthetic pigment. The synthetic pigments have contains chromium, mercury, sodium chloride, toluene, copper, lead, benzene and others [1]. These chemical substances can be toxic and have fatal effect in the human body. Thus, the orange fruits are chosen and used as pigment because the orange fruit don't have toxic substances and easy to get in our country. Nowadays, cotton is widely used in textile product , however it has low water resistance characteristics. Means that it can absorb water easily, and this case always annoyed us. This is because, our clothes that made from cotton was getting wet and dirty easily since the cotton has high water absorbent characteristics. A stain is a physical or chemical interaction of two difference materials and it is hardly removed from cotton fabric by using washing powder.

Hence, orange fruit pigment was investigated because it have water-resistant and self-cleaning characteristics that can help to prevent it happens [2]. Orange fruit pigment can increase environmental awareness because of less toxic, non-pollutant, less health hazard, rare colour idea and no allergic reactions. This study is to formulate and synthesis the orange fruit pigment for supehydrophobic dyeing, and evaluate the mechanical and physical test for superhydrophobic dyeing in cotton knitted textile applications.

II. LITERATURE REVIEW

Usually, dyeing is completed in special solution that contains dyes and particular chemical material. The dyeing process in textiles is the colour would be transferred to a textile material or finished textile to add permanent and long-lasting color. It can be done by machine or by hand. Dyeing can be in water or some other carrier also, but it must be able to penetrate and colour the textile materials in the process. The textile is completely saturated by the colored and dye, when the dye and the textile had come into contact. There are two main factors in dyeing which is time controlled and temperature [3]. The dyes and chemical aids such as surfactants, acids, alkali/bases, electrolytes, carriers, leveling agents, promoting agents, chelating agents, emulsifying oils, softening agents and others are applied to the textile to get a uniform depth of colour with the colour fastness properties suitable for the end use of the fabric [4]. This dyeing process includes the dispersion of the dye into the liquid phase, then adsorption onto the outer surface of the fibers, and finally dispersion and adsorption on the inner surface of the fibers [5].

The main purpose of superhydrophobic dyeing that applied in the textile field is to repel water, and the nanoscopic surface layer of superhydrophobic dyeing was made the fabric has water resistance characteristics. Superhydrophobic dyeing can fully rebind the droplets when superhydrophobic dyeing was hit by droplets [6]. However, superhydrophobic dyeing had cleaning capabilities which are helped or protected the fabric from dirty water or chemical liquid that can spoil the fabric and this case can be referred to Figure 1. This kind of dyeing can be used in the textile regions such as sports shoes, T-shirts, bags, pants, and others [7].

Figure 1: Shoes is treated and untreated by superhydrophobic dyeing [7]

Superhydrophobic textile surfaces are referred to surfaces with excellent water repellency with a water contact angle more than 140° and low contact angle hysteresis of less than 10°. This situation can be explained from Figure 2. The most well-known example was the development of superhydrophobic self-cleaning materials that mimic lotus leaves [8].

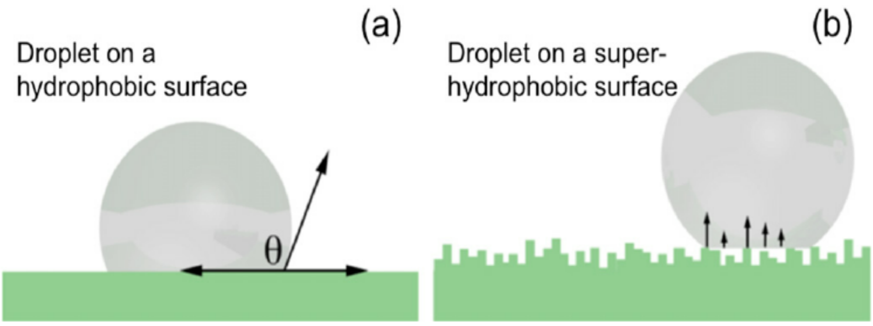


Figure 2: Syperhydrophobic surface [10]

At such surfaces, dirt and soils are loosely attached, and a rolling water drop can easily be attached the loosely bonded substances, removing them from the surface, giving self-cleaning effects. Due to this, the phenomenon of self-cleaning resulting from a superhydrophobic textile surface that does not become wet is called the lotus effect. This surface characteristic is applicable in industries for oil repellence, anti-corrosion, anti-fogging/frosting, anti-bioadhesion, and water-oil separation [9,10]. Because of this, there had been active research for the past several decades on various methods and materials that propose superhydrophobic and ultra-oil repellence that controlled wettability for water, oil, and non-polar liquids through the chemical makeup of solid surfaces and designing geometrical surface structure [11]. Superhydrophobic textiles can grant not only excellent water repellences and oil resistance, but also activated self-cleaning performance, and thus they can be used as high protective clothing textiles and functional outdoor clothing material [12]. Apart from this, it can be reduced the number of launderings thanks to the self-cleaning performance. When the number of launderings is reduced, the performance of the highly functional textiles can be maintained for long times and can lead to the development of environment-friendly materials that can reduce the use of resources and energy needed for laundry [13].

III. METHODOLOGY

A. *Preparation of Orange Fruit Pigments*

In this study, it was decided to focus on the preparation of the orange fruit pigment as a natural superhydrophobic dyeing. The method of preparing the pigments is by a straightforward preparation method. Orange fruit pigments are extracted from the fruit by using suitable extraction method to get the colour pigment known as carotenoid. Then, the orange fruit pigment is used into superhydrophobic dyeing to apply at the cotton knitted fabric surfaces.The water extraction method is used to extract the orange fruit pigments. The pigment has been prepared from the extract of the mature orange fruit that store in room temperature. In order to obtain the raw pigment of orange fruit (caroternoid), the extract fruit juice must not contain any additives.

The orange fruit was peeled to remove the fruit skins and obtain the pulps. The pulps are cut into the pieces and blend it to get the juice by using blender machine. To get rid the seeds and grains, the cloth filter is used to filter orange fruit juice. After that, filter tunnel and filter paper was used to filter orange juice again for extract the orange fruit pigments, and the pure orange fruit pigment is getting. In order to prepare the pigments for the surface dyeing, the extraction of orange fruit pigments are divided into four sets of different concentrations which are 20 %, 40 %, 60 % and 80 % of concentration which can be referred in Figure 3. The distilled water with an average room temperature between 27°C to 35°C has been added to four sets of pigments. The distilled water is added to the pigments and make a comparison between each set of pigment concentrations. The ratio of distilled water has been added is shown in Table 1.

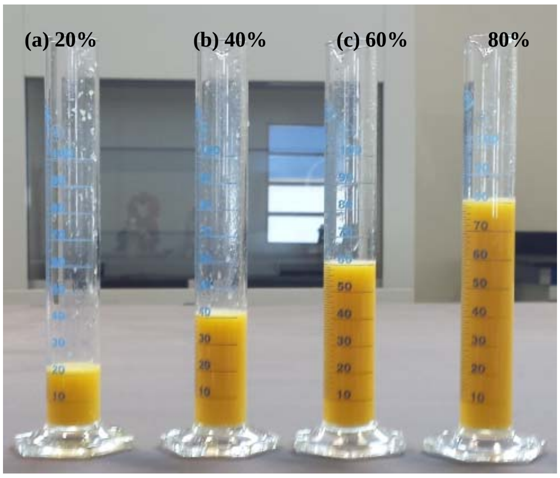


Figure 3: Different concentration of orange fruit pigment (a) 20%, (b) 40%, (c) 60% and (d) 80%

Table 1: The amount of distilled water has been added in this study

Orange fruit concentration, %	Amount of distilled water added, (*ml*)
20	100
40	100
60	100
80	100

B. *Preparation of Superhydrophobic Coating*

The solution for superhydrophobic coating is prepared. To complete the preparation of superhydrophobic coating, the solution of components for the coating has been prepared. The solvent and hardener are used to prepare the mixture of the dyeing. The ratio of solvent to hardener was used in this study which is 50 *ml*: 5 *ml*. The solvent, hardener and additive are mixed and stirred together in a beaker by using a magnetic stirrer to ensure both solutions have well mix. The amount of solvent, hardener and additive, for four different concentration of orange pigment is displayed by Table 2.

Table 2: The amount of solution contains of orange fruit concentration, solvent, hardener and additive.

Samples	Orange fruit concentration, (%)	Solvent, (*ml*)	Hardener, (*ml*)	Additive, (*ml*)
A	20	50	5	5
B	40	50	5	5
C	60	50	5	5
D	80	50	5	5

C. *Application of Orange Fruit Pigment on Cotton Knitted Fabric Surfaces.*

First, the white cotton knitted fabric was prepared. The cotton knitted fabric was cut into small pieces and measure the weight of cotton fabric around 8 gram by using the weighing machine. The cotton knitted fabric that has been cut was pre-soaked in the warm water with fixatives agent which is vinegar and soak the cotton knitted fabric around 45 minutes at 90-degree Celsius. After that, the warm water is poured out into the sink, then, strain and wring the pre-soaked cotton knitted fabric. Then, the orange fruit pigment and pre-soaked fabric are put together into stainless steel beaker and stir about it. Next, the stainless steel beaker was closed with a cover. The stainless steel beaker that contains orange fruit pigment liquid and pre-soaked cotton knitted fabric is placed into dyeing machine to rotate and stir automatically around 45 minutes at 90 degree Celcius. The wet cotton knitted fabric was left dried around 1 hour before to apply superhydrophobic dyeing mixture on top of the pigment dyeing. Lastly, the different concentration of orange fruit pigment has been succeeded to apply on the cotton knitted surface as in Figure 4.

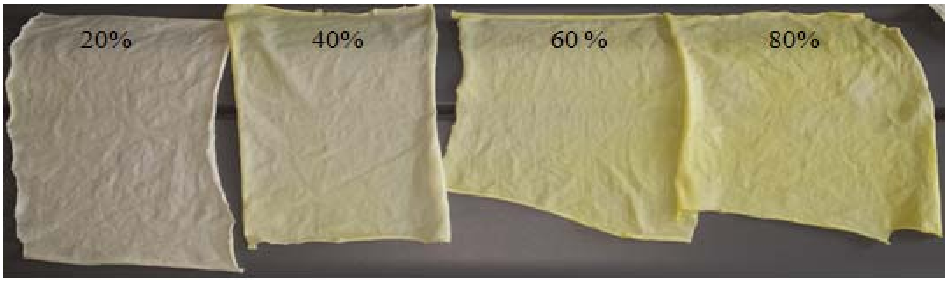


Figure 4: From left to right are 20%, 40%, 60% and 80% of orange fruit pigment that applied to the cotton knitted fabric.

D. *Method of Coating*

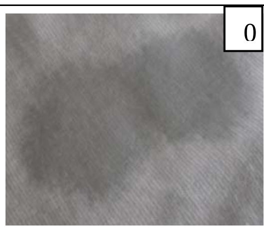
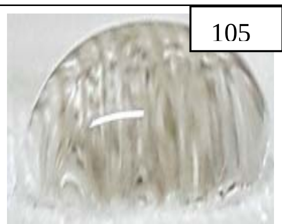
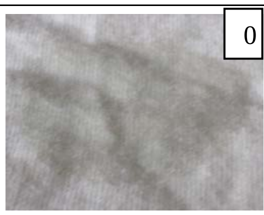
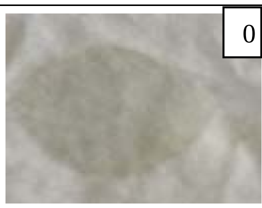
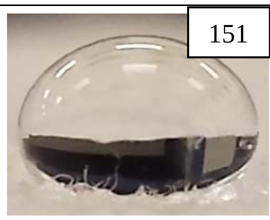
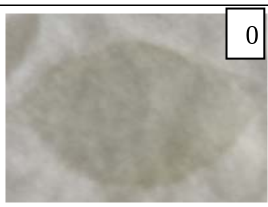
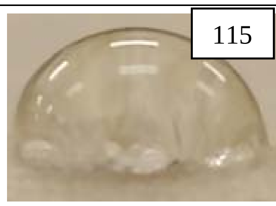
The superhydrophobic coating solution was applied to the surface of the fabric that has been dyed with orange fruit pigment by using spraying gun technique. A spray gun was used in the process to apply the mixture of superhydrophobic coating onto the surface of the fabric. This is an extremely fast method of coating and one of the most economic, effectively and efficiency used when large volumes of coating are used opposed to small quantities, which are typical repaint maintenance operations. The requirement of the standard before carried out the coating, the environment used for determining the drying time of air dry coatings is a temperature of 23±2°C and relative humidity of 50±5% under the diffuse daylight. The superhydrophobic coating was poured into the spray gun and coated with 45 degree angle by swing the gun back and forth across the surface. The coating was left to completely dry for a day before conducting any test on the samples.

IV. RESULTS AND DISCUSSIONS

A. *Water Droplet Test*

Water droplet test (ASTM 5946-04) was conducted to measure water contact angle (WCA) for superhydrophobic surface. Water droplets were placed onto the fabric surface with certain technique and the picture was taken by using DSLR camera.Contact angles between water drops and surfaces can be measured directly from the angle formed at the contact between the liquid and the flat surface. The superhydrophobic coating should be in between 140º to 175º and it can be considered as superhydrophobic when the water contact angle is more than the desired value. The fabric without superhydrophobic coated is not be considered as superhydrophobic. This is because the water droplet surface angle is 0º from 0 % to 80 %. This case is indicated that the water is totally absorbed by the cotton fabric that without using superhydrophobic coating when the water is dropped to the surface fabric. Besides that, the Table 3 also showed that the actual angle of contact obtained for superhydrophobic coating is between range 105º and 151º.

Table 3: Water droplet test of cotton knitted fabric without superhydrophobic coating and cotton knitted fabric with superhydrophobic coating

Concentration of orange fruit pigment %	The water droplet surface contact angle (˚)	
0		
20		
40		
60		
80		

The concentration of 0 %, 20%, 40% and 80 % of orange fruit pigment cannot be regarded as superhydrophobic properties since the angle of water droplets contact didn't reach the range of superhydrophobic properties required and the water droplet surface contact angle of 0 %, 20% and 80 % are 105˚ and 115˚ respectively. The 40% and 60 % of orange fruit pigment can be regarded as superhydrophobic properties while the water contact angle in this 2 different concentration of orange fruit pigment is 146 º and 151º respectively. In addition, the fabric which has superhydrophobic properties or no has superhydrophobic properties can be observed and evaluated based on the appearance of the water droplet. For example, the appearance of the water droplet in 0 % is hemisphere shape but the appearance of the water droplet in 60 % had sphere perfectly. This happens can be said that the fabric which has superhydrophobic properties, the shape of water droplet has been more sphere when it compared with the fabric that didn't has superhydrophobic properties [13].

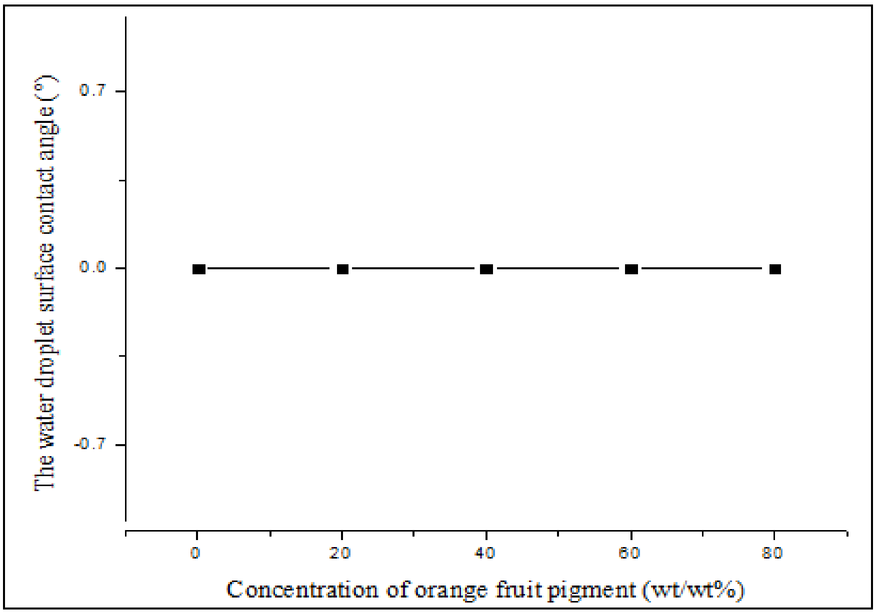


Figure 5: The water droplet surface contact angle (˚) for the fabric without the superhydrophobic coating

In Figure 5, the angle of water contact was kept at 0º despite the concentration of orange fruit pigment has been a rose. This evidence is proved that the increased orange fruit pigment concentration didn't give or improve the superhydrophobic characteristic of the fabric if the fabric has no coating by the superhydrophobic solution.Superhydrophobic surfaces possessing high advancing water contact angle (WCA) and lower water contact angle hysteresis, have recently attracted significant attention because their water-repellent and self-cleaning properties and their potential for practical applications ranging from biotechnology to self-cleaning commodity materials. The superhydrophobic coating gives unique characteristics to the coated surface which is when water dropped onto the surface, it shattered into particles and the rolled off from the surface [13].

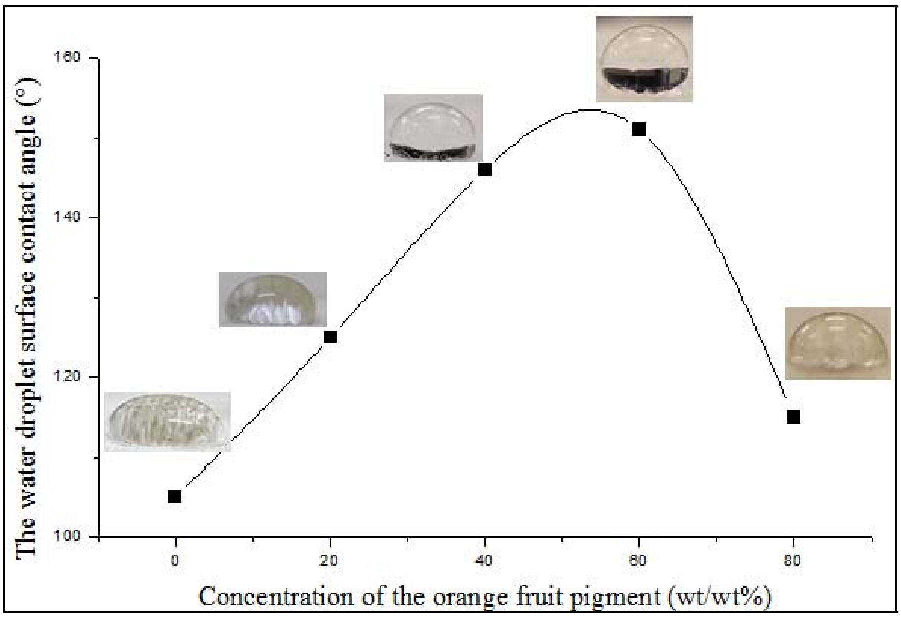


Figure 6: The water droplet surface contact angle (˚) for the fabric with the superhydrophobic coating

It was indicated that the water droplet surface contact angle (˚) has been increased from 105˚ to 151˚ during the concentration of the orange fruit pigment increased also from 0 % to 60 % as shown in Figure 6. After that, the water droplet surface contact angle is started to decrease at 80 % of orange fruit pigment which is 115˚. This structure is not stable due to higher percentages of orange fruit pigment that generate hydrophilic residue on the coating. Thus, the water droplet surface contact angle has been dropped at the 80 % orange fruit pigment. The result was portrayed that 60 % of orange fruit pigment is an optimal concentration to produce the superhydrophobic properties by synthesis with superhydrophic coating.

B. *Colourfastness to Crocking Test*

The colourfastness to crocking test (ASTM D5053) has been carried out to determine the amount of colour transfer from the surface of the colour cotton knitted fabric to other surfaces by crocking. The staining is determined by using the grey scale which showed the colour transfer. The scale has a rating of 1 to 5 whereby 1 is having the high degree of colour transfer and 5 has no colour transfer.

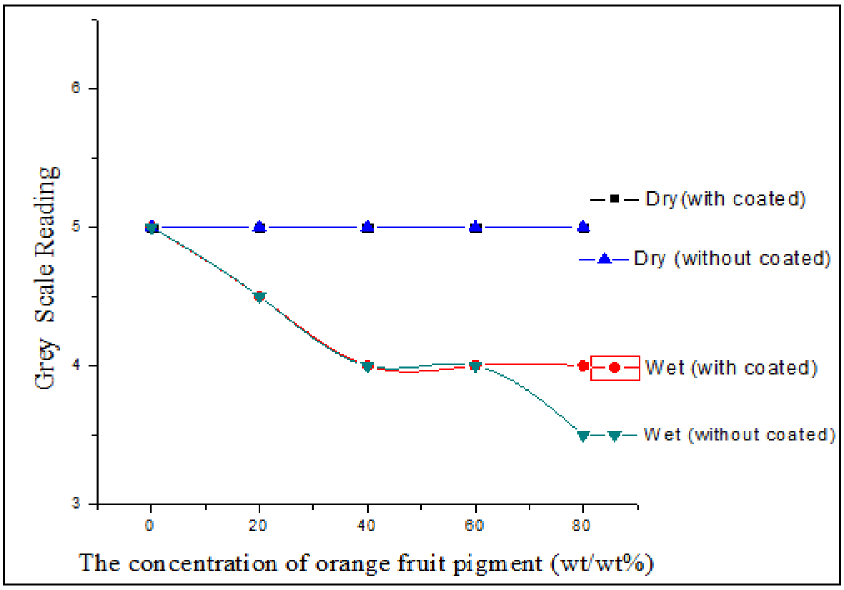


Figure 7: The grey scale reading of dry crocking and wet crocking cloth in between superyhydrophobic coating and without superhydrophobic coating

According to the Figure 7, the greyscale reading of the dry crocking test with the superhydrophobic coating or dry crocking test without the superhydrophobic coating are maintained at 5 when the concentration of orange fruit pigment is increased from 0 % to 80 %. Other than that, the greyscale reading of the wet crocking test with the superhydrophobic coating and without the superhydrophobic coating are decreased slowly from 5 to 4 when the concentration of orange fruit pigment has been increased from 0 % to 40 %. For the wet crocking test with the superhydrophobic coating is maintained at reading of 4 in between 40 % to 80 %. From 40 % to 60 % , the fabric without superhydrophobic coating is maintained constant, after that, it was decreased from 4 to 3.5 when the concentration of orange fruit pigment has been reached to 80 %. Throughout this experiment, the dry crocking result is showed better rubbing fastness than wet crocking result of the both type fabrics because it maintain at reading of 5. This is because in wet crocking, unfixed dyes was dissolved in water then it is transferred to the test fabric, Thus resulting in poor wet crocking fastness and the grey scale reading of wet crocking test also lowered than dry crocking test [15].

C. *Bursting Strength Test*

Bursting strength test (ASTM D3786) was conducted to measure the strength in which the material is stressed in all directions at the same time. The cotton knitted fabric samples is preparing by cutting the fabrics into circular shape following the rubber diaphragm's size of fabrics. Based on the Figure 8, the bursting strength of the coated fabric or the fabric without coated has been increased during the concentration of orange fruit pigment is increased too. The range of coated fabric bursting strength is laid in between 37.26 kPa to 42.97 kPa when the fabric without coated was given a range value of 36.77 kPa to 41.78 kPa. In the 80 % of orange fruit, the bursting strength of the fabric coated and the fabric without coating has the highest bursting value which is 42.97 kPa and 41.78 kPa respectively while the 0 % concentration of orange fruit has the lowest bursting strength which is 37.26 kPa for coated fabric and 36.77 kPa for fabric without superhydrophobic coating. The bursting strength of the fabric coated is always higher than the fabric without coated in all different of the orange fruit concentration. This may due to the fiber tensile characteristics, the fiber with higher elongation increases the fabric bursting strength. This can be explained that the coated fabric has higher elasticity than fabric without coating since the bursting strength of coated fabric is higher than fabric without coating [16].

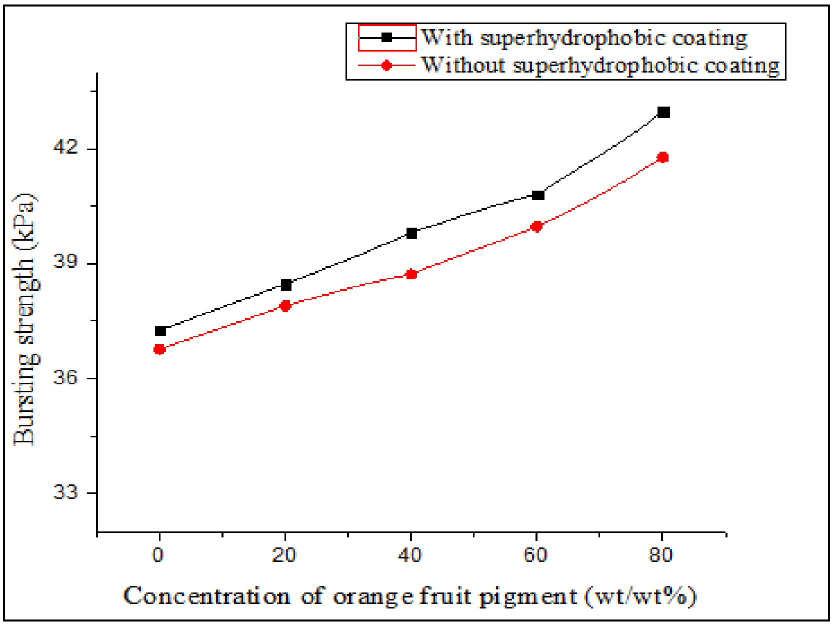


Figure 8: Bursting strength (kPa) of cotton knitted fabric in between superhydrophobic coating and without superhydrophobic coating

D. *Abrasion Resistance Test*

Abrasion resistance test (ASTM D4966-98) was conducted to measure of how well fabric allows the passage of air through it. The abrasion resistance is the capability of a fabric to oppose surface wear caused by flat rubbing contact with another material. The measurement of the resistance to abrasion of textile and other materials are very complicated. This is because, the resistance to abrasion was easily affected by many factors, such as the inherent mechanical properties of the fibers; the structure of the yarns; the construction of the fabrics; the dimensions of the fibers; and the type, kind, and amount of finishing material added to the yarns, fibers, or fabric.

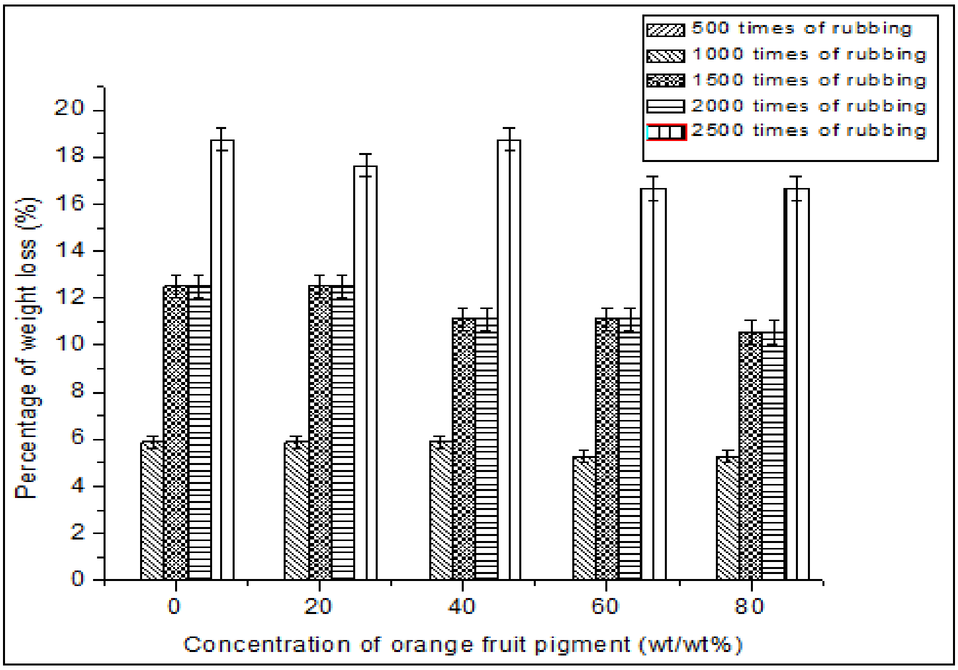


Figure 9: Abrasion test of cotton fabric with superhydrophobic coating in different number times of rubbing

There are 5 different times of rubbing is carried out in all different concentration of orange fruit pigment which is 500 times of rubbing, 1000 times of rubbing, 1500 times of rubbing, 2000 times of rubbing and 2500 times of rubbing. The Figure 9 is illustrated that the coated fabric didn't have any weight loss in 500 times of rubbing which is 0 percent of weight loss. The percentage of the weight loss is increased in all different concentration of orange fruit pigment during the times of rubbing has been increased from 500 times to 2500 times. The percentage of weight loss is same in between 1500 times of rubbing to 2000 times of rubbing nevertheless the concentration of orange fruit pigment is different. This condition was showed by the Figure 9. In short, the largest percentage of weight loss was 2500 times of rubbing for 0%, 20%, 40%, 60% and 80% of orange fruit pigment were 18.75%, 17.65%, 18.75%, 16.67% and 16.67% respectively. In 2500 times of rubbing, all type of fabric has the highest percentage of weight loss in all the different of orange fruit pigment concentration. More and more fibres were coming out on the fabric surface and cut off from the surface due to number times of rubbing is increasing [10]. Thus, this condition was resulting the weight loss percentage of the fabric has been increased.

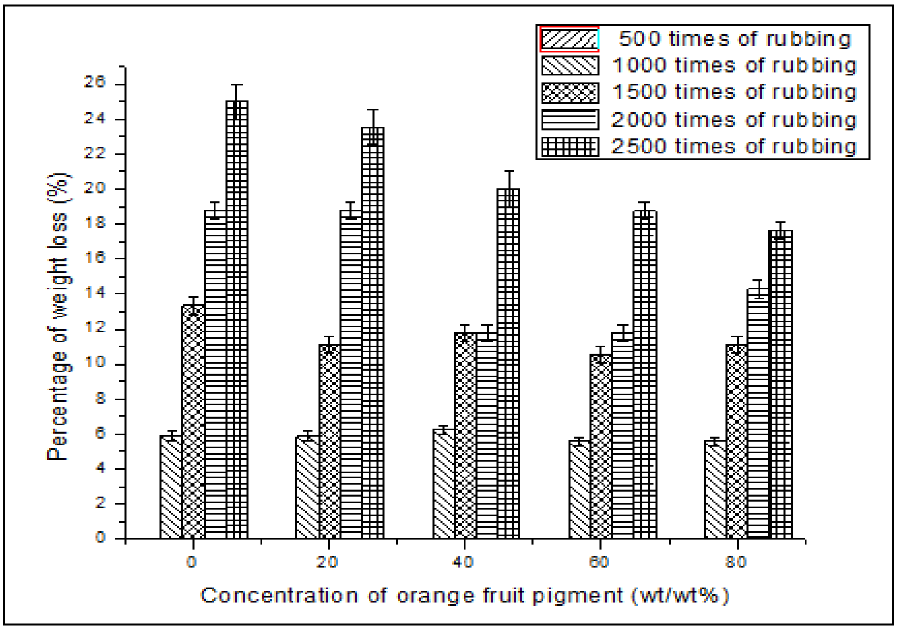


Figure 10: Abrasion test of fabric without superhydrophobic coating in different number times of rubbing

From the Figure 10, there is totally no any change in weight before and after which is 0 percent of weight loss in 500 times of rubbing. In 1000 times of rubbing, the percentage of weight loss is remain constant which is 5.88 % from 0% to 20 % of orange fruit then increase to 6.25 % of weight loss at the 40 % of orange fruit pigment. After that, the 6.25 % of weight loss has been decreased to 5.56 % and maintain constant in between 60 % to 80 % of orange fruit pigment. For 2500 times of rubbing, the fabric without coated has undergone a great loss of weight especially for 0 % of orange fruit pigment which is 21.05% of weight loss. This graph is expressed that the fabric without the superhydrophobic coating has the worst performance in mechanical properties such as fabric strength and durability since the weight loss of fabric without coating is higher than the fabric coated. The fabric surface has been replaced and covered by the superhydrophobic coating of the fabric to accept the direct damage from rub cycle. Hence, the abrasion resistance of the fabric coated is better than the fabric without superhydrophobic coating.

V. CONCLUSION

In conclusion, the objectives of this study have been achieved. The orange fruit pigment for superhydrophobic dyeing was formulated and synthesized. The mechanical and physical test for superhydrophobic dyeing in cotton knitted textile applications have been evaluated. In this study, the orange fruit pigment was chosen and used to dye the cotton fabric. The mechanical and physical tests were conducted to analyze the properties of the fabric with superhydrophobic coating and the fabric without the superhydrophobic coating based of the different concentration of orange fruit pigment. In the water droplets test, 60% of the fabric that coated with superhydrophobic solution have the most excellent superhydrophobic properties in all different concentration of orange fruit pigment. This is because the water droplet contact angle of 60 % is 151˚ which is highest degree of contact angle in all the different concentration of orange fruit pigment.

In the colourfastness to crocking test, the fabric with superhydrophobic coating and the fabric without the superhydrophobic coating have better dry crocking fastness than wet crocking fastness because it maintain at reading of 5. This is because in wet crocking, unfixed dyes were dissolved in water then it is transferred to the test fabric, hence resulting in poor wet crocking fastness and the grey scale reading of wet crocking test also lowered than dry crocking test.In the bursting strength, the bursting strength of the fabric coated is always higher than the fabric without coated in all different of the orange fruit concentration. This may due to the fiber tensile characteristics, the fiber with higher elongation increases the fabric bursting strength. This can be explained that the coated fabric has higher elasticity than fabric without coating since the bursting strength of coated fabric is higher than fabric without coating. In the abrasion resistance test, the fabric without coating was undergone a great loss of weight especially for 0 (wt/wt%) which is 21.05% of weight loss during the number times of rubbing has been reached to 2500. Lastly, the 60% of orange fruit pigment is suggested to use and applied on the cotton knitted fabric due to the 60% of orange fruit pigment can be produced the optimum of superhydrophobic characteristic.

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