ISSN (Print) : 2337-9030 ISSN (Online) : 2685-2608
PILLAR OF PHYSICS
Vol. 17 (2), 2024, page. 147-155
DOI : 10.24036/13811171074

EFFECT OF VARIATIONS OF COTTON FABRIC DYEING ON CHITOSAN-SIO2 COMPOSITE SOLUTION ON HYDROPHOBIC PROPERTIES FOR ANTI-VIRUS MASK

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ABSTRACT

Coronavirus Disease 2019 (Covid-19) has attracted worldwide attention since December 2019 and was declared a pandemic by the World Health Organization (WHO) on March 11. Covid-19 is believed to have originated in Wuhan, China, and has spread to more than 200 countries, every day the number of Covid-19 cases in the world continues to increase. One of the efforts to overcome Covid-19 is wearing a mask. This study aims to manufacture hydrophobic masks from cotton fabric that have antivirus properties from chitosan-SiO2 composite materials. This type of research is a laboratory experiment, Chitosan-SiO_2 is used in the size of nanoparticles using a High Energy Milling (HEM) tool. Next, analyze the SiO_2 content using X-Ray Fluorescence (XRF) and Infra Red Spectrophotometer (FTIR) to calculate the degree of deacetylation of chitosan. To analyze the crystal structure and crystal size using X-Ray Diffraction (XRD) and to determine the morphological structure of the Chitosan-SiO_2 composite layer. The results of this study are hydrophobic masks derived from cotton fabric are anti-virus. After washing the cotton fabric layer sample, contact angle melting occurs. The best variation of dyeing against contact angles and antivirus tests characterized by surface temperature imagery patterns is 4 times dyeing with a contact angle of 136.138° reaching ultrahydrophobic angles and surface temperatures of 41.6°C.

Keywords: Chitosan– SiO2, Hydrophobic, Cotton Fabric, Antivirus

I. INTRODUCTION

Coronavirus Disease 2019 (Covid-19) has attracted world wide attention since December 2019 and was declared a pandemic by the World Health Organization (WHO) on March 11. Covid-19, which is believed to have originated in Wuhan, China, has now spread to more than 200 countries, after being declared a pandemic every day the number of Covid-19 cases around the world continues to increase. The Covid-19 response effort from the Covid-19 Task Force for Behavior Change focuses on increasing 3M compliance, namely wearing masks, maintaining distance, and washing hands. cannot be used repeatedly. According [1], medical mask waste found in the Cilincing and Marunda Rivers was 432±68 per day in March 2020 and 552±102 per day in April 2020. Disposable medical masks are formed from several polymers such as polypropylene, polyacrylonitrile, polystyrene, polycarbonate, and polyethylene that can degrade into microplastic pollutants [2]. Therefore, innovation is needed in the form of making cloth masks that can prevent the spread of Covid-19 and can be reused to mediate an environmental crisis due to disposable mask pollutants.

Submitted: .October 18, 2022 Accepted: October 21, 2022 Published: . October 31, 2024

Cotton fabric has the characteristics of being comfortable to use so that it can be applied as a protective face mask that functions to filter particles, viruses, and bacteria in the nose and mouth. Cloth masks have the advantage of being washable and reusable. However, cloth masks are easily moist, causing bacteria to breed, therefore masks must be kept from being moist from droplets or water vapor when we breathe or other factors, so masks must be hydrophobic (waterproof), to determine the hydrophobic nature of the fabric, a test is carried out hydrophobic [3]. The shape of the surface that is dripped with hydrophobic water can be seen in Figure 1.

Fig 1. Water on the surface of lotus leaves [4]

Based on Figure 1, there are hydrophobic properties in lotus leaves which have surface characteristics such as a waxy layer forming nano-sized protrusions [5]. The hydrophobic surface can be determined from the contact angle (WCA), the angle between the air droplet and the surface of the object. If the contact angle on the surface is >90o, then the surface is called a hydrophobic surface [6]. Factors that affect hydrophobic properties are the chemical composition contained on a surface and the influence of the roughness factor [7].

The chemical composition of a surface material will affect its hydrophobic properties because this water-repellent property is not symmetrical or polar, therefore the surface of a superhydrophobic surface must be nonpolar. While the roughness factor will affect the water-repellent properties, where when water interacts with the surface of the material it will cause action and reaction forces between the two. By using the principle of the law of equilibrium when a surface is getting rougher or the surface of the water that touches it will cause an increase in the hydrophobicity of the surface of the material. The superhydrophobic nature uses low surface energy so that it will be able to reduce the wettability of the solid surface which will produce a surface with hydrophobic properties [8]. According to [9], the contact angle (θ) is the angle that will be formed from a tangent to a liquid contained in the contact line and the line through which the bottom of the liquid drops. Many natural materials have hydrophobic properties, for example, rice husks.

Rice husk (rice husk/rice hull) is the outermost part of the rice grain and has the highest silica content compared to other rice by-products [10]. The silica content of rice husk ash is 94-96% and if the silica content is below it is most likely due to husk samples that have been contaminated with other substances with low silica content [11]. The natural ingredient used as an antibacterial is chitosan. Chitosan is obtained from the deacetylation of chitin consisting of glucosamine *(2-amino-2-deoxy-D-glucose, 75-85%) and N-acetylglucosamine (2-acetamido-2-deoxy-D-glucose, 15-25%)* which are polycationic and carry a positive charge in the pH range below 6.5. This cationic nature causes chitosan to interact with negative materials, including the microbial outer cell membrane. The positive charge of chitosan when interacting with the negative charge on the microbial cell membrane can cause damage to the components inside the microbial cell so that it can inhibit the growth of the microbe. The structure of chitosan can be seen in Figure 2.

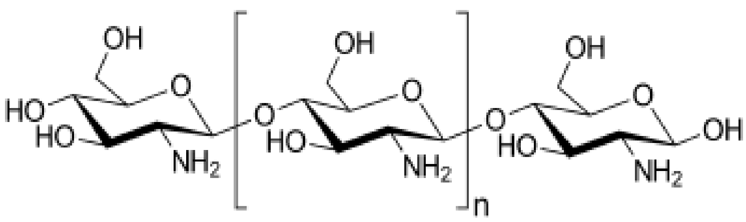

Fig 2. Chitosan Structure [12]

Based on Figure 2, the structure of chitosan is irregular, its shape is crystalline or semicrystalline. In addition, it can also be in the form of a white amorphous solid with a fixed crystal structure from the initial form of pure chitin. Chitin has high biological and mechanical properties including renewable, biodegradable, and biofunctional [13]. Based on previous research on making hydrophobic masks and having antivirus properties using TiO_2-SiO_2 coating. The coating obtained very good results on its hydrophobic properties, morphology, and antivirus activity. The contact angle resulting from TiO_2-SiO_2 coating was 110.4º with a surface temperature of polyester fabric which is coated at about 40ºC. So the polyester fabric of this layer can be used as an antivirus mask [14].

II. METHOD

This research is a type of laboratory experimental research. This study examines the effect of variations in the dyeing of cotton fabric in a Chitosan-SiO_2 composite solution on the hydrophobic properties of antivirus masks. The tools used in this research are a measuring cup, beaker, Erlenmeyer, oven, magnetic stirrer, measuring pipette, dropper, digital scale, 100 mesh sieve, furnace, DSLR camera, and full scale. While the characterization tools used is XRD, XRF, SEM, thermal camera, and UV-C lamp. The materials used in this study were rice husk ash, cotton fabric, crab chitosan, sodium hydroxide (NaOH), chloride solution (HCl), equades, xylene, and polyethylene.

The stage of extracting silica from rice husks is carried out by cleaning the rice husks first so that they rice husks are free from impurities that can affect the silica yield. The rice husks were then dried and heated at a high temperature of about 700ºC for 5 hours to form rice husk ash. Furthermore, rice husk ash was added to a 4% NaOH solution and placed on a magnetic stirrer. Stirring was carried out continuously at a temperature of 800ºC for 1 hour. Then the resulting solution was filtered with NaSiO3 to separate the precipitate. The NaSiO3 solution was added little by little to 37% HCl until a precipitate formed. The resulting precipitate was in the form of a white gel to obtain gel maturity, the gel was left for 48 hours. After that, the gel was washed until the washing water was white and then dried in the oven to dry.

The synthesis step of the Chitosan-SiO_2 composite was carried out using the sol-gel method. First, weigh 1.5 grams of polyethylene and 90 ml of xylene solvent used into a 250 ml beaker. polyethylene and xylene were stirred using *a magnetic stirrer* at a temperature of 200ºC with a rotational speed of 1000 rpm for 25 minutes. Next, enter the silica that has been weighed as much as 1.5 grams into the polyethylene and xylene solution for 15 minutes. Then add 1.5 grams of chitosan into a solution of polyethylene, xylene, and silica that has been mixed and stirred using a*n a magnetic stirrer* for 30 minutes at 800 rpm.

The coating stage is carried out on masks made from cotton fabric which are prepared by sterilizing the cotton fabric first. Cotton fabric measuring 2 x 3 cm before a coating is sterilized using an ultrasonic cleaner. The cotton fabric is soaked using alcohol in a 100 ml beaker for 5 minutes in an ultrasonic cleaner. Furthermore, the cotton fabric is removed and dried using an oven for 5 minutes at 80ºC so that the cotton fabric is ready for coating. Coating the mask is done by dipping the mask in the Chitosan-SiO_2 composite solution for approximately 5 minutes and then removing it using tweezers and placing it in a closed room and waiting for it to dry. Drying time is required for 5 minutes in the oven at 90ºC. For this composite solution, a coating is carried out on the outer part of the mask only and will then be coated in the middle and the uncoated mask. Then characterize using SEM to see the morphological structure of the coated mask and measure the contact angle. After the sample was dry, the cotton fabric was tested for hydrophobicity by testing the contact angle. The sample was placed in a dark room that was only lit by a flashlight, then water was dripped using scale injection on the surface of the fabric and an image was taken using a camera. The contact angle measurement was carried out using Image J software.

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Testing the SEM results using Image J software. Characterized samples are then analyzed using Image J to obtain the particle size of the sample. The grain size of the resulting particles is in nanometers (nm). Testing of antivirus properties on cotton fabrics was carried out to determine the antivirus properties of various dyeing cotton fabrics with Chitosan – SiO_2 composite solution, irradiating the layer using UV-C light for 20 minutes. Furthermore, the sample saw the pattern of the surface temperature image generated by using a thermal camera. Then measure the contact angle washing of cotton fabric samples was carried out to see the hydrophobic properties of cotton fabrics with variations in dyeing in the Chitosan – SiO_2 composite solution and whether there was contact angle melting or not. The samples were washed by soaking the layered cotton fabric in warm water for 15 minutes. oven, then the contact angle analysis was carried out using Image J software.

The data analysis stage on chitosan and silica that have been characterized using XRD to determine the crystal size of Chitosan – SiO_2 in each dyeing variation can be analyzed using the Debye-Scherer equation, namely:

$$D = \frac{0,94\,\lambda}{\beta \cos\theta} \tag{1}$$

Information:

D = Crystal size (nm)
λ = X-ray wavelength (nm)
β = FWHM value in units (o)
θ = Bragg diffraction angle

III. RESULTS AND DISCUSSION

This study used variations of dyeing 1 time, 2 times, 3 times, and 4 times to see the effect of contact angle and antivirus properties on cotton fabrics. X-ray diffraction pattern produced from Chitosan-SiO2 composite layer using 2 x 3 cm cotton fabric by variations dyeing found at the 2θ peak and intensity. Sample characterization using XRD to determine the crystal size. The results of XRD data from each dyeing variation can be seen in Figure 3.

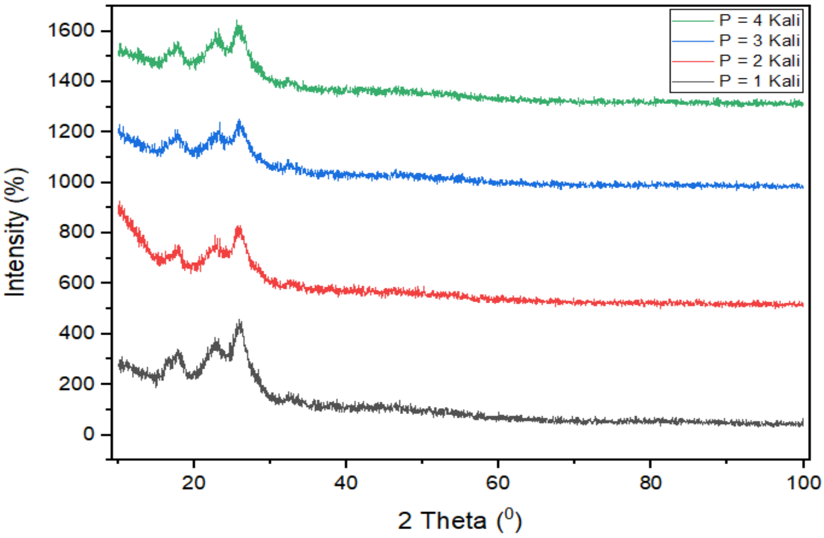
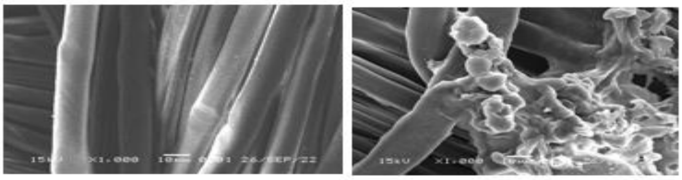


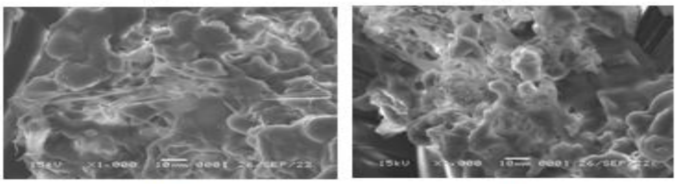
Fig 3. Comparison of XRD results by dyeing variation

Based on Figure 3, it can be seen several peaks from each dyeing variation. The types of Chitosan-SiO2 composite crystals formed are orthorhombic and hexagonal. The size of the crystals produced in the dyeing variations is 44.80 nm, 43.28 nm, 46.28 nm, and 45.28 nm. Additionally, the change in dyeing has an impact on peak width, with each variation in immersion resulting in a distinct peak width. XRD crystal size obtained through the Scherrer equation. For the results of the crystal size is already in the form of nano where the material range is said to be nano if it meets <100 nm, so it can be said to be a nanocomposite. Particle size is not identical to crystal size. The crystal is part of the grain, while the grain is part of the particle so that the crystal size < grain size < particle size. However, in nanometer-scale crystals in single-dominant crystalline materials, the crystal size is the same as the grain size and almost the same as the particle size [15].

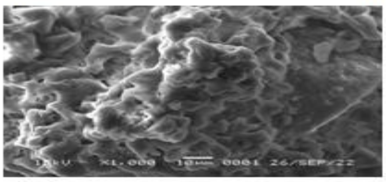
Cotton fabric coating with Chitosan-SiO_2 composite can be characterized by Scanning Electron Microscopy (SEM). The difference between a cotton fabric that has been coated in a composite with a dye variation and a regular cotton fabric. The Chitosan-SiO_2 composite that sticks strongly to the cotton fabric indicates that the dyeing process was successful, as shown in Figure 4.



(a) (b)



(c) (d)



(e)

Fig 4. The results of SEM characterization with a magnification of 1,000 times for (a) plain cotton fabrics, (b) 1 time dyed cotton fabrics, (c) 2 times dyed cotton fabrics, (d) 3 times dyed cotton fabrics, and (e) 4 times dyed cotton fabrics

Based on Figure 4, it can be seen the difference in morphology of ordinary cotton fabrics with cotton fabrics coated with Chitosan-SiO_2 composite. The results of SEM characterization can measure the particle size using the Image J application. The particle sizes produced by dyeing variations are 84 nm, 77 nm, 62 nm, and 32 nm. The surface of the cotton fabric is wide, densely packed, and lump-free when it is not coated. The cotton fabric in the composite solution became more flimsy, wide in shape, lumpy, and bumpy after one dyeing. The cotton fabric was dyeied in the composite solution twice, and the second time it was more dense, broad, lumpy, and bumpy. The larger the lumps become when cotton fabric is dyeied three times as deeply in the morphological composite solution from the surface, but there are still grain protrusions where the distribution of the particles is uneven in different parts of the sample, giving the appearance of lumps of particles piling up unevenly. And when 4 times of dyeing the cotton fabric turned tighter and there were still grain protrusions.

Particle size affects the roughness of the surface morphology, the smaller the particle size formed causes the hydrophobicity to be higher and the surface energy to decrease. This decreased surface energy is due to the small interaction between water and the surface which causes high interaction of water with air, so that the water will tend to maintain its spherical shape, as a result the contact angle between the water and the surface formed is getting bigger [16].

This contact angle measurement is carried out to determine the hydrophobic nature of the cotton fabric. A surface can be said to be hydrophobic if it has a large contact angle of 90^o. And antivirus testing of coated cotton fabric masks can be observed temperature image patterns using a thermal camera and UV-C light. The image pattern of the surface temperature of the coated cotton fabric and the magnitude of the contact angle resulting from each variation of dyeing cotton fabric can be seen in Figure 5.

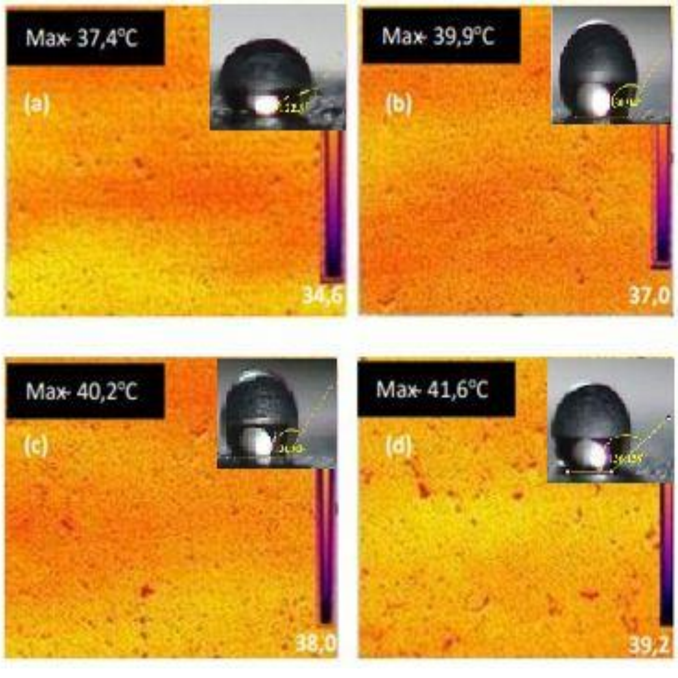


Fig 5. Contact angle and surface temperature image pattern of coated cotton fabric (a) 1 time (b) 2 times (c) 3 times (d) 4 times dyeing

Based on Figure 5 the dyeing that has the largest contact angle is 4 times dyeing, when testing the contact angle, the attractive force that occurs between the distilled water and the coating will cause the adhesion force to be small. Adhesion is the attractive force between dissimilar particles. A hydrophobic surface has a large contact angle value with water so that the contact between layers with a small liquid is also called low-adhesion.

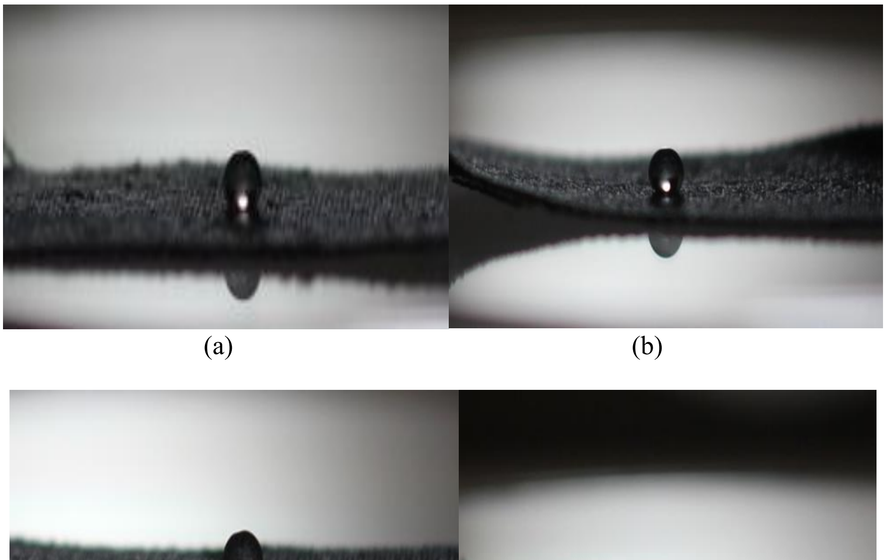
The contact angle technique used is the static drop method or Sessile. It is carried out with successive drops of liquid until convexity at the contact angle is reached. This convexity is known as the forward contact angle.

After testing the forward contact angle, the contact angle will be obtained by combining the contact angles as a droplet equal to the volume of the liquid is successively withdrawn from the droplet. It is important to understand that forward and backward from a corner are usually not the same. There is usually a high degree of experimental hysteresis resulting from sample pre-hydration, surface roughness, chemical heterogeneity, evaporation and/or molecular motion. Thus, the contact angle allows one to measure the degree of hysteresis attached to the sample surface.

According to [17],research, the hydrophobicity stability of hydrophobic silica composite membranes utilizing cotton cloth as a buffer layer is affected by the amount of stability. According to the study's findings, the membrane generated with a distance of 2 times has the largest contact angle, which is 141,62^{o}, and the angle of 5 times has the maximum stability with a gradient value of decreasing the contact angle of 1,673.

Research conducted by [14], this involves covering anti-virus masking materials with a TiO2-SiO2 composite using the dip coating method. Due to only a 10-minute exposure to UV-C radiation, the coating on the kain used in the antivirus penguin test reached a temperature of at least 40 degrees Celsius. This is a good result. Due to the receptor binding motif from the protein spike SARS-CoV-2 undergoing conformational melting at a temperature of 40^{o}C, kain hasil coating has the most potential to be used as an antivirus masker as a result of this. And the surface temperature image pattern increases with each dyeing variation, so the greater the number of dyeings, the greater the surface temperature. This shows that cotton fabric with Chitosan-SiO_2 coating can be used as an antivirus mask because it produces a surface temperature exceeding 40^{o}C with a UV-C light irradiation time for 20 minutes, at that temperature, it will inhibit the DNA growth of the SARS Cov-19 virus [18].

Measurement of contact angle on cotton fabric that has been coated with Chitosan-SiO2 composite solution after washing is obtained by soaking cotton fabric that has been coated with alcohol soaked for 15 minutes. Testing of contact angle after washing on variations of dyeing cotton fabric coated with Chitosan-SiO2. This measurement is carried out to determine whether the fabric is still hydrophobic or not after washing. The results of the contact angle after washing the coated cotton fabric can be seen in Figure 6.



(a) (b)

(c) (d)

Fig 6. Result of contact angle after washing (a) 1 time (b) 2 times (c) 3 times (d) 4 times dyeing

Figure 6 above shows an image of the contact angle produced after washing with each dye variation which is then processed using Image-J software. It can be said that the variation of dyeing can affect the contact angle magnification, because the more solution that is repeatedly coated on the fabric, the rougher the surface of the fabric, so the resulting contact angle will be greater. The comparison of the contact angle values before and after washing is shown in Table 1.

Table 1. Contact Angle Value Before And After Washing

Variation Dyeing	Contact Angle Before Washing	Contact Angle After Washing
1 time	122.4º	120.6º
2 times	130.914º	129.971º
3 times	134.563º	134,412º
4 times	136.138º	135º

Table 1 above can be made as a comparison graph of the contact angle for each variation of dyeing. The comparison can be seen in Figure 7.

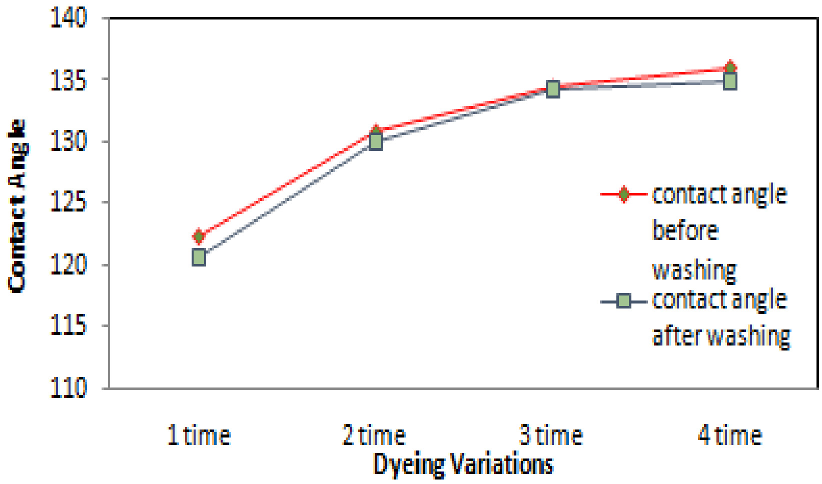


Fig 7. Graph of comparison of contact angles before and after washing

Figure 7 shows that the resulting contact angle after washing has decreased but is still ultra-hydrophobic. This can be seen from the contact angle with variations of 1 time of dyeing where the contact angle before washing is around 122.4º and after washing the contact angle in other dyeing variations also slightly decreases to 120.6º.

In the variation of dyeing 2 times, 3 times, and 4 times dyeing after washing the contact angle decreased, respectively, namely 129.971º, 134,412º, and 135º so that the washing factor by soaking cotton fabric into the alcohol causes the contact angle to decrease but is still ultrahydrophobic.If a layer has a contact angle between 120° and 150°, it is considered to be ultrahydrophobic. The Wenzel model is the appropriate wetting model for the surface layer after washing because the hydrophobic air droplets on the surface will be impacted by the surface's roughness.

IV. CONCLUSION

The Chitosan-SiO_2 composite coating process on dye variations on cotton fabrics showed very good results from the aspect of morphology, hydrophobicity, and antivirus activity. The surface morphology of cotton fabrics which were characterized using SEM showed that the composites bonded strongly to the fabric fibers. The contact angle resulting from variations in the dyeing of cotton fabric that has been coated with Chitosan-SiO2 composite increased the contact angle of each variation. Meanwhile, after washing the cotton fabric experienced a slight decrease. The higher the dyeing, the higher the resulting contact angle. The highest contact angle is obtained at four times of large dyeing and is ultra hydrophobic. The surface temperature of the composite coated fabric which was characterized by a thermal camera and UV-C irradiation showed good results, the surface temperature exceeded 40^{o}C. Therefore, the coated fabric with variations of four times dyeing of the composite solution has great potential to be applied as an antivirus because the receptor binding of the viral motif will begin to close.

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