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## Antifungal property of cotton fabric textile: Modification of cotton fiber functions by coating compounds of $\text{TiO}_2\text{-SiO}_2\text{/Chitosan}$

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### ABSTRACT

The antifungal activity from cotton fabric textile was studied by using  $\text{TiO}_2\text{-SiO}_2\text{/Chitosan}$ , with CTABr (Cetyl Trimethyl Ammonium Bromide) template as nano particle suspension disperser in the surface of cotton fabric textile. The difference of dip-spin coating time was correlated by the perfection of  $\text{TiO}_2\text{-SiO}_2\text{/Chitosan}$  coating and indicated by the gain of textile mass, stable to the washing and the ability of textile to inhibit the *Aspergillus niger* growth. Result showed that in the 24 h and 90 min of dip-spin coating, there was 14 mm of *A. niger* inhibition zone, compared with cotton fabric textile without treatment and was larger inhibition zone than cotton fabric textile treated by ketokonazol as a positive control. The chemical change occurred in the textile fiber after  $\text{TiO}_2\text{-SiO}_2\text{/Chitosan}$  coating was analyzed by FT-IR and SEM-EDX.

**Keywords:** antifungal, cotton, textile,  $\text{TiO}_2$ ,  $\text{SiO}_2$ , Chitosan

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### INTRODUCTION

Textile is primary need and must be provided in daily activity. The need of high quality textile is needed by consumer. In order to choose the quality of textile for its use, consumers have to know the basic knowledge about textile fiber to make it easier to identify the high quality textile for daily need [1]. Based on the type of fiber, textile can be classified as cotton, wool and silk [2]. Cotton is a popular textile product, made from cotton fiber with cellulose as primer component. Cotton has unique properties such as soft and light fiber, numb and easy in maintenance. Cotton fiber has porous so it absorbs sweat easily. This property gives the opportunity for microbe to grow in textile fiber [3].

The microbial growth is stimulated by the structure of fiber and environment [4,5]. The presence of microbes in the fiber can cause cross infection, color degradation and bad odor to textile [4]. The need of clean a hygiene textile is urgent due to the raise of infection caused by pathogenic microbe. Several techniques have been applied to push the growth of microorganism by using chemical compounds or physical interaction such as the polymerization of N-halamin monomer to textile fiber, the addition of N-halamin to *electro spinning*, enzyme immobilization (*eter crosslinked*), the placement of ammonium salt to fiber textile covalently [6].

The application of nanotechnology to small industries by the coating of nanomaterial compounds (metal oxide) is to modify the function of textile fiber. Nanotechnology can increase the quality of textile to be the multifunction textile product with complex properties (anti microbial, UV protection, and anti odor). The success of nanotechnology application in textile industry has several advantages such as durability, multifunction without degrade the basic properties such as process ability, flexibility and economic.

TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan nanoparticle is a semiconductor, inert, non-toxic and photo catalytic material [7]. It also has anti fungal property because in UV radiation, there is redox reaction in band gap (valence a conduction band) which causes the release of radical hydroxyl and *reactive oxygen species* (ROS) [8,9]. The radical of hydroxyl can activate microbe by oxidizing phospholipid in membrane cell, where OH radical has ability to oxidize 1.000 – 10.000 times more effective than the inhibition ability of disinfectant in market such as chlorine, ozone and chlorine dioxide [10]. The application of TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan as anti fungal are safe for consumer, easy to apply in textile industry, economical value, compatible with other finishing agents and environmentally friendly. Rilda *et al.* 2015 [11] has synthesized TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan nanoparticle by modification with chitosan which can increase the photocatalytic activity and has good anti microbial property if doped with CTABr surfactant. Chitosan has non toxic, biocompatibility, biodegradability, anti fungal activity and chemical reactivity properties [12,13].

Fungi is a microbial species which can survive in high moisture textile. The growth of fungi can occur in both new and stored textile. The growth of textile is difficult to handle. Some chemical compounds which have been used to inhibit fungal growth can't work effectively because the difficulty to degrade fungal cell. The use of TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan is an alternative solution to overcome the problem in textile because its effectiveness to any kinds of bacteria (positive and negative gram) [11,14,15]. The aimed of this research is to modify the function of textile fiber by TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan coating in preparation of anti fungal textile.

## MATERIALS AND METHODS

### Materials

Materials used were cotton textile (64 g/cm<sup>2</sup>), chloride acid (HCl), Acetic acid (CH<sub>3</sub>COOH), diethanol amine (C<sub>4</sub>H<sub>11</sub>NO<sub>2</sub>), isopropanol (C<sub>3</sub>H<sub>8</sub>O), tetraethoxyortho silicate (C<sub>8</sub>H<sub>20</sub>O<sub>4</sub>Si), Salicylic acid (CH<sub>2</sub>CHCOOH), Cationic surfactant (C<sub>16</sub>H<sub>33</sub>N(CH<sub>3</sub>)<sub>3</sub>Br) Merck, aquadest, titanium isopropoxide (C<sub>12</sub>H<sub>28</sub>O<sub>4</sub>Ti) (Aldrich 97%), commercial chitosan (C<sub>6</sub>H<sub>11</sub>NO<sub>4</sub>)<sub>n</sub> (85% de-acetylated).

### Synthesis and Preparation of Cotton Textile Coating by TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan

TiO<sub>2</sub> sol suspension consists of the mixture of TIP, isopropanol and DEA as additive substance from TIP hydrolysis. SiO<sub>2</sub> as dispersion agent consists of the mixture of TEOS, isopropanol and HCl. The addition of chitosan/acetic acid at 5:1. Sol mixture was made by varying TiO<sub>2</sub> : SiO<sub>2</sub> molar composition (1:1 and 2:1) with the addition of CTAB surfactant as a template of particle distributor. The gel incubation was done at 110° C for 15 h. Gel was calcinated at 500° C for 3 h to get TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan powder. TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan powder suspension was coated at 8x8 size of cotton textile which has been dipped in to acrylic acid solution. The dipping time variation were 6, 12 and 24 h. TiO<sub>2</sub>-SiO<sub>2</sub>/Chitosan coating (1:1 and 2:1) was done by *dip-spin coating* method with 60, 90, 120 and 150 min of time setting with CTABr as dispersion agent. The process was continued by the drying of cotton textile at 80° C for 10 min. The characterization of cotton textile was done by FT-IR, SEM and EDX analysis and non-coated cotton textile was used as a control.

### Anti Fungal Textile Measurement

The suspension of *A. niger* which has been grown for 48 h in PDA medium was made by 10<sup>-2</sup> of dilution method. The suspension then inoculated into Petri dish with PDA medium and a piece of cotton textile (d= 10 mm) coated by TiO<sub>2</sub>-SiO<sub>2</sub>. Incubation was done for 48-72 h at 37° C in laminar flow with UV radiation.

## RESULTS AND DISCUSSION

### The Gain of Textile's Mass Analysis

The analysis of the gain of textile mass is identical as a description that the coating of 8 x 8 cm textile with TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan reached the perfection. Based on the duration of dipping time in acrylic acid binder for 6, 12 and 24 h, it was correlated to synergize the function of acrylic acid binder in TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan coating to the textile fiber. Figure 1 describes the perfection of coating is related with the duration of dipping time in acrylic acid. The result showed that the dipping for 24 h has the highest gain of textile's mass, which is indicated by the more of TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan powder coating. The covalent bond of *cross-linkester* with hydroxyl group of cellulose formed in cotton fiber is relevant with the length of dipping process in acrylic acid and TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan nanoparticle [16].

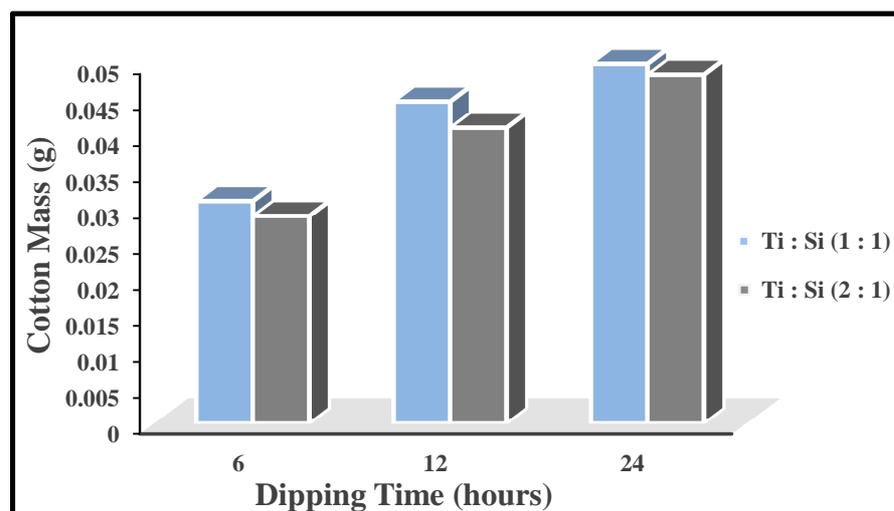


Fig 1. The mass amount of cotton textile after coated with  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  with time variation of dipping in acrylic acid

Figure 2 shows that there was a gain in textile's mass after being coated by  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  with 60 to 90 min dipped with acrylic acid. Meanwhile, after dipped by acrylic acid for 24 h, there was a higher gain in textile's mass. There was no significant gain in textile's mass after dipped by acrylic acid for 120 – 150 min. It is predicted that there was a spreading of  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  suspension to the wall inside spin-coating instrument. The optimum time of  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  coating to the surface of cotton textile fiber is 90 min.

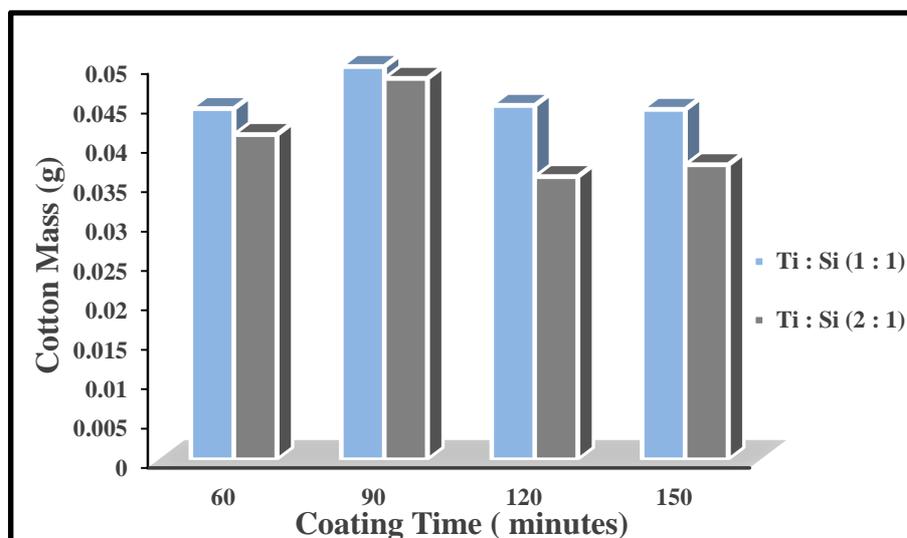
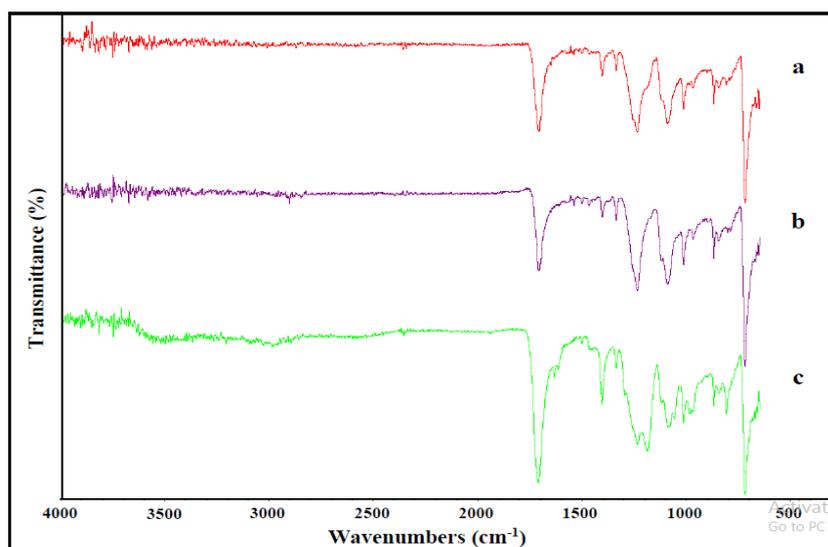


Fig 2. The amount of textile cotton's mass after being coated by  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  1 % with Ti:Si molar composition 1:1 and 2:1 with 24 h dipped in acrylic acid

#### FT-IR Analysis

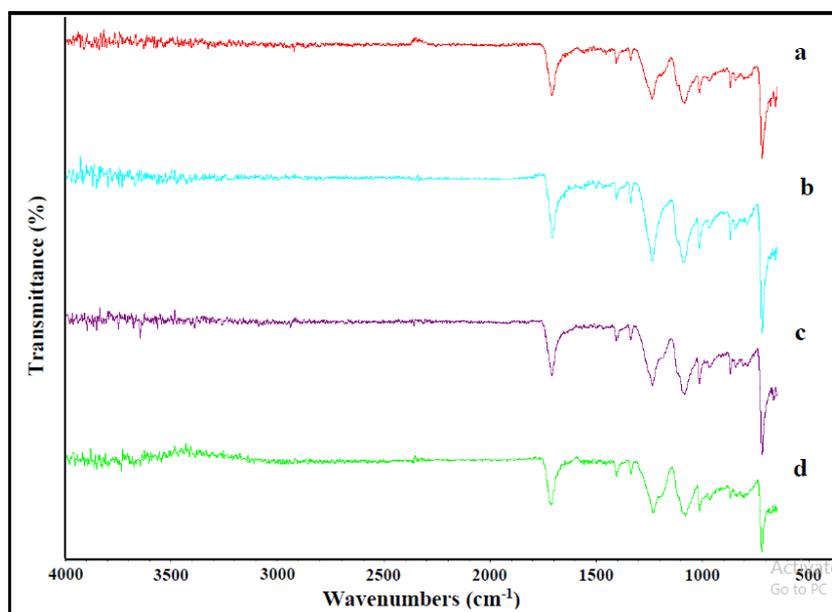
FT-IR analysis can provide information that the perfection of textile fiber coating process with  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  is indicated by the formation of chemical interaction between hydroxyl group from cellulose with carboxylic group from acrylic acid as *cross ink* agent. This interaction happened was indicated by the change of the intensity of C=O stretching functional group at  $1700\text{ cm}^{-1}$  wave number [17].



**Fig 3. FTIR pattern of textile cotton fiber to the time duration of dipping in acrylic acid**

(a) 6 h; (b) 12 h and (c) 24 h.

Figure 4 shows that the intensity of ester covalent interaction which happened at 6 -24 h of dipping process in acrylic acid was at 1427, 1160, 1109, 1054 and 1031  $\text{cm}^{-1}$  of wave number are the indication of cellulose group comes from the fiber (Nasr, H.E, et al., 2009). Meanwhile, at 3 (c) spectrum, there is absorbance at 3000 and 1000  $\text{cm}^{-1}$  which show C=O residue comes from  $\text{CO}_2$  at measurement time [18]. The intensity of C=O stretching group at 1700  $\text{cm}^{-1}$  will be higher depends on the length of dipping time in acrylic acid binder which show the perfection of covalent interaction of ester. This also indicates the perfection of  $\text{TiO}_2/\text{SiO}_2/\text{chitosan}$  coating in the textile fiber.



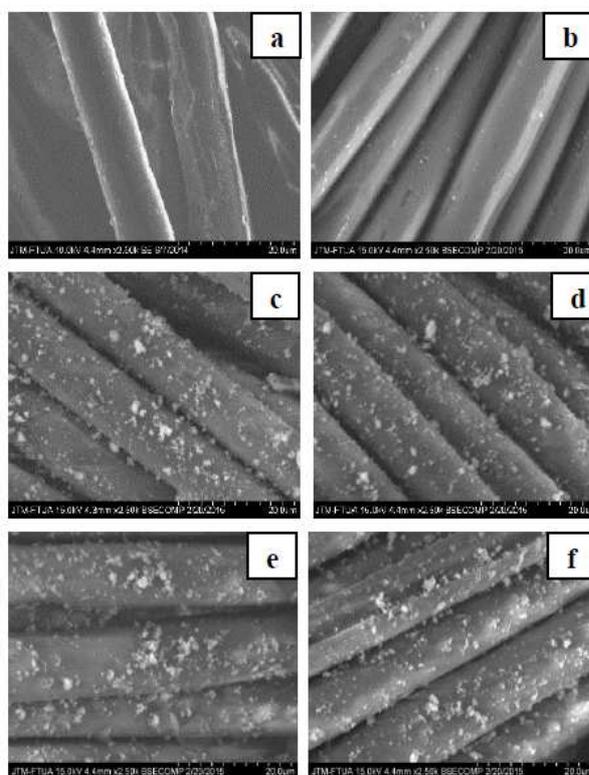
**Fig 4. FT-IR pattern of textile cotton fiber with  $\text{TiO}_2/\text{SiO}_2/\text{chitosan}$  (1:1) coating time (a) 60 min; (b) 90 min; (c) 120 min and (d) 150 min; with 24 h dipped in acrylic acid.**

If the coating time of  $\text{TiO}_2/\text{SiO}_2/\text{chitosan}$  was varied at 90 – 150 min, there was no significant changes in the intensity of C=O group. There was only minor shift of Ti-O-C interaction at 1260  $\text{cm}^{-1}$  [17]. Meanwhile, the absorbance band at 1105 - 950  $\text{cm}^{-1}$  was identical with the formation of Ti-O-Si interaction (Rilda, et al., 2014). It was also confirmed that there was O-Ti-O interaction at 650  $\text{cm}^{-1}$  and vibration of Si-O-Si at 1080  $\text{cm}^{-1}$  [19].

### Morphology and EDX Analysis

SEM analysis can provide the surface morphology of cotton fiber which have been coated by  $\text{TiO}_2/\text{SiO}_2/\text{chitosan}$ . Figure 5 (a) shows the surface morphology of cotton textile after dipped in acrylic acid for 24 h. It can be seen that

the cotton fiber was soft and clean. The surface morphology of textile cotton which has been coated by  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  (2:1) is shown at figure 5 (c), (d) and (e) with 6, 12 and 24 h of time variation of dipping in acrylic acid. It can be seen that textile cotton coated by  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  with 24 h of dipping time in acrylic acid was coated by  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  particle perfectly with well distribution particle by CTABr as dispersion agent.



**Fig 5.** The surface morphology of: (a) non coated textile cotton; (b) textile cotton dipped by acrylic acid; (c) – (e) textile cotton coated by  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  (2:1) with dipping time in acrylic acid: (c) 6 h; (d) 12 h and (e) 24 h; (f)-(h) textile cotton coated by acrylic acid and  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  (1:1) with concentration: (f) 1 %

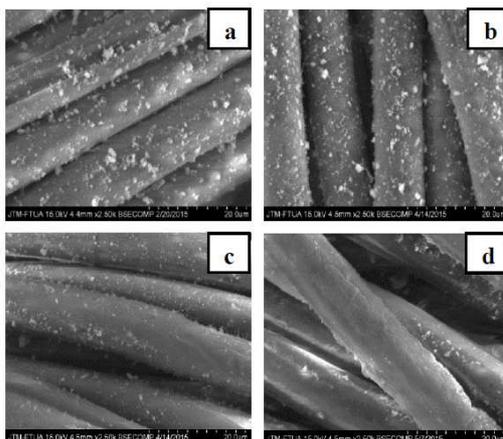
The molar composition of Ti and Si influence the  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  coating process in textile. SEM analysis shows that in Ti:Si (1:1) molar composition, the coating particle was distributed well compared with Ti:Si (2:1).  $\text{TiO}_2$  is an electropositive compound and if the Ti composition is higher than Si, it will produce more positive charge particles and increase the interaction between textile cotton fiber with  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  nanoparticle [20]. Meanwhile,  $\text{SiO}_2$  is a particle which will form *core shell* with  $\text{TiO}_2$  to enlarge the surface area and porosity of  $\text{TiO}_2$  to the fungal cell [21]. Therefore, the Ti composition in  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  gave the influence in the inhibition ability of fungi. The surface morphology of textile cotton coated by  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  with concentration variation (Figure 5 f –h) showed that the significant difference of the amount of  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  coated in the textile cotton which is correlated with EDX data.

The stability of  $\text{TiO}_2\text{-SiO}_2/\text{chitosan}$  coating on the surface of textile fiber can be measured by repetition rinse. Table 2 shows that there was the decrease of textile's mass after repetition rinse.

**Table 2.** The Decreasing Percentage of Textile Cotton's Mass After Rinsed by Water

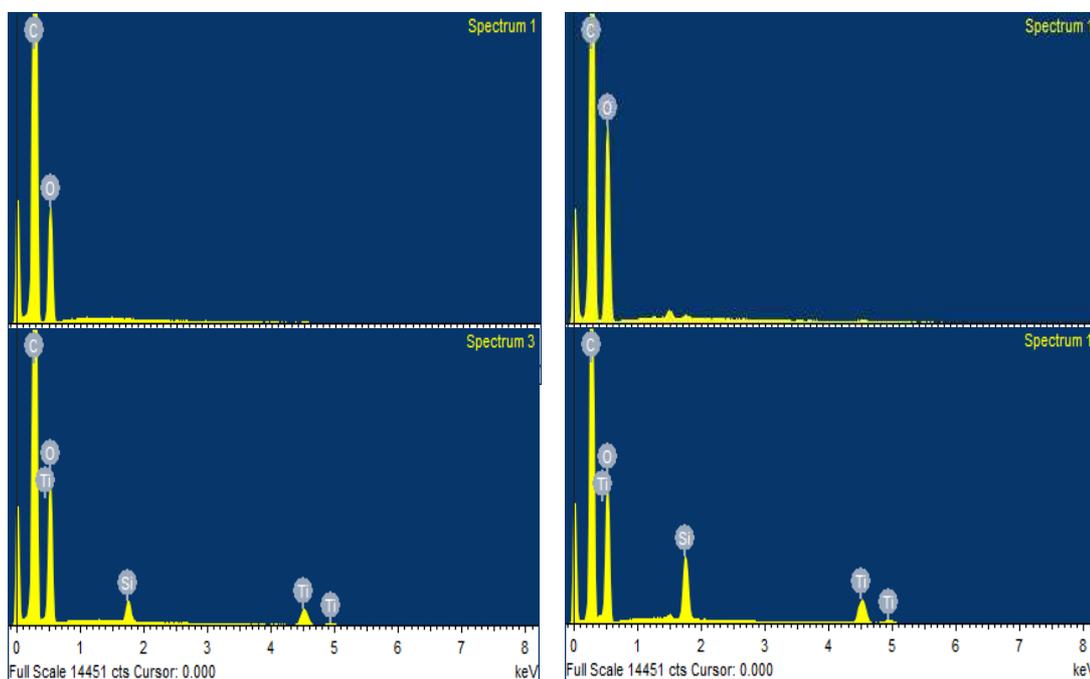
Repetition of Rinsing	Textile Cotton's Mass		Mass Decreasing (%)
	Before Rinsing (g)	After Rinsing (g)	
1	0.1767	0.1736	1.75
3	0.1767	0.1719	2.72
5	0.1767	0.1714	2.99

Figure 6 shows the effect of rinsing to the stability of coating on the textile fiber. Karimi *et al* reported that the rinsing of textile cotton can decrease the mass of  $\text{TiO}_2$  coated on the cotton fiber [16]. Meanwhile, Yu Ming *et al* reported that  $\text{TiO}_2$  nanoparticle is stable on the surface of textile cotton with 30 x rinsing by using *cross-linkpoly*(2-hydroxyethyl acrylate) (PHEA) [21].



**Fig 6. The surface morphology of textile cotton coated by  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$**   
(a) before rinsing; (b) after 1x rinsing; (c) after 3x rinsing and (d) after 5 x rinsing

EDX analysis can be correlated with the substance composition as an indication of the  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  coating on the surface of textile cotton. Difactogram pattern from Figure 7 (a) shows the composition of textile cotton without treatment. There was only substances from cellulose identified such as C and O. Figure 7 (b) shows the pattern of composition of textile cotton after dipped in acrylic acid. Acrylic acid is one of carboxylic acid with  $\text{C}_3\text{H}_4\text{O}_2$  of molecular formula. There was increasing of C and O percentage of textile fiber after treated with acrylic acid to the non treated cotton. Figure 7 (c) shows the cotton textile coated by  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  (1:1) consists of Ti and Si and is confirmed that it has been coated by  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$ . The capacity of  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  on the surface of 8x8 cm textile fiber, by comparing the ability of powder suspension at 1 % concentration, was distributed well on the fiber surface. The loading capacity of the textile cotton to the  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  at 1:1 was higher than 2:1.

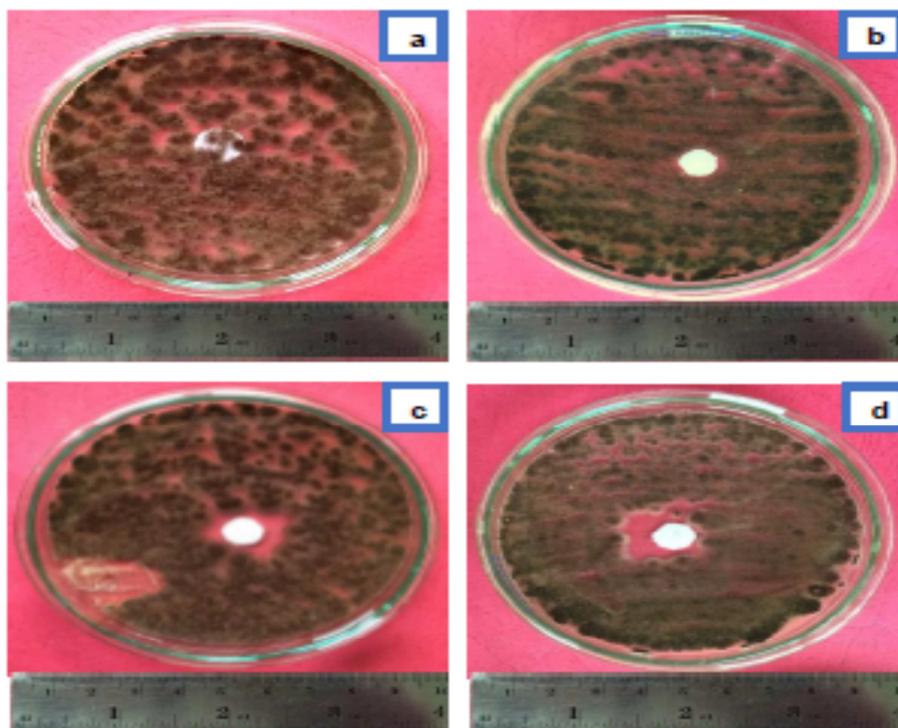


**Fig 7. EDX analysis: (a) non coated textile cotton; (b) textile cotton dipped in acrylic acid; (c) textile cotton coated by acrylic acid and 1 %  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  (1:1) and (d) textile cotton coated by acrylic acid and 3 %  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  (1:1)**

### Antifungal Activity

The antifungal activity of textile was measured by measuring the growth inhibition of *Aspergillus niger*. Figure 8 shows that the textile activity was influenced by the duration of textile dipped into acrylic acid binder and coating of textile cotton by  $\text{TiO}_2\text{SiO}_2/\text{chitosan}$  with different of Ti and Si composition. The result shows that the inhibition

ability of textile to *Aspergillus niger*'s growth was correlated to the amount of  $\text{TiO}_2\text{-SiO}_2$ /chitosan coated on the textile fiber and the gain of textile's mass.



**Fig 8.** The inhibition zone of antifungal activity of textile to *A. Niger* : (a) non treated textile (control -); (b) textile treated by *ketokonazol* (control +); (c) textile coated by  $\text{TiO}_2\text{-SiO}_2$ /chitosan (1:1) and (d) textile coated by  $\text{TiO}_2\text{-SiO}_2$ /chitosan (2:1)

Figure 9 shows the antifungal activity of  $\text{TiO}_2\text{-SiO}_2$ /chitosan to *A. niger* with different composition variation of  $\text{TiO}_2\text{-SiO}_2$ /chitosan. Figure 9 (a) is a negative control which was non coated textile and it can be seen that there was no inhibition zone formed. Figure 9 (b) is a positive control which was textile coated by *ketokonazol* antibiotic and there was inhibition zone formed. Figure 9 (c) and (d) is textile coated by  $\text{TiO}_2\text{-SiO}_2$ /chitosan with each composition (1:1) and (2:1). It can be seen that the antifungal activity of  $\text{TiO}_2\text{-SiO}_2$ /chitosan with UV irradiation was higher than *ketokonazol*. The formed inhibition zones in textile coated by  $\text{TiO}_2\text{-SiO}_2$ /chitosan were 14 mm and 12 mm. In the composition Ti and Si (2:1), where the amount of  $\text{TiO}_2$  is higher than  $\text{SiO}_2$ , the ability of  $\text{TiO}_2\text{-SiO}_2$ /chitosan to form free radical hydroxyl ( $\bullet\text{OH}$ ) and superoxide anion ( $\bullet\text{O}_2^-$ ) is higher than  $\text{TiO}_2\text{-SiO}_2$ /chitosan compound with Ti:Si (1:1) composition. Free radical hydroxyl ( $\bullet\text{OH}$ ) and superoxide anion ( $\bullet\text{O}_2^-$ ) are strong oxidator which are able to destroy the membrane cell of fungi which caused the lysis that inhibit the fungal growth.



**Fig 9.** The morphology of *Aspergillusniger*: (a) before coated by  $\text{TiO}_2\text{-SiO}_2$ /chitosan (1:1) and (b) after coated by  $\text{TiO}_2\text{-SiO}_2$ /chitosan (2:1)

*A niger* has the membrane cell which consists of chitin which is a complex compound and protective spore to the contamination. This causes *A. niger* is more difficult for cell lysis by the interaction with  $\text{TiO}_2\text{-SiO}_2$ /chitosan. The morphological structure of *A. niger* after inhibited by  $\text{TiO}_2\text{-SiO}_2$ /chitosan is shown at figure 9.

## CONCLUSION

The effect of the time setting of dipping in acrylic acid was to allow the synergy among functional groups in the formation of *cross-link*. The optimum time resulted were 24 h and 90 min. FT-IR analysis showed that there was change in the C=O stretching intensity which is identical with chemical interaction of ester covalent bond. The surface morphology analysis by SEM-EDX showed that TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan (1:1) nanoparticle was distributed well by CTABr role. Antifungal textile activity showed that the textile coated by TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan (2:1) had higher inhibition ability which was 14 mm after irradiated with UV (536 lux) for 48 h. The stability of TiO<sub>2</sub>-SiO<sub>2</sub>/chitosan was done by the 5 times repetition of rinsing process. It can be concluded that this study can be applied to small industries due to the simple treatment.

## Acknowledgments

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