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# Optical Characteristics Of Nano-Talc/$TiO_2$ Thin Layer: A Preliminary Study

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**Abstract:** This study tested the effect of 50%:50% concentration nano-Talc/$TiO_2$ on optical and photocatalytic properties thin layer activity. The goal is to understand how 50%:50% concentration affects absorption, reflection and transmission as well as photocatalytic activity of thin layer. This research is experimental, starting with grinding Talc and $TiO_2$, followed by dilution layer synthesis using the spin coating technique. Characterization includes cross-section morphology analysis using SEM, absorption, reflection, transmission and energy measurements slit with a UV-Vis DR spectroscope, as well as photocatalytic activity test. The research results show that the coating process managed to increase absorption by (92-98)% and energy band gap of 2.93 eV while reflection and transmission are reduced by (0.5-4)%, as well as the lowest rate of photocatalytic activity degradation rate of 0%, causing low ROS under light sunlight protective layer.

**Keywords:** Talc; $TiO_2$; Photocatalytic Activity; Spin Coating; Concentration.



## 1. Introduction

Titanium dioxide ($TiO_2$) is the active material of a sunscreen that is used as one of the active ingredients of sunscreens. The advantage of this material is that it can absorb and reflect sunlight because it has a wide spectrum of protection, which is in the area of UVA and UVB [1]. Thus, $TiO_2$ can protect the skin from user risks from exposure to UV rays, UVB causes skin burns and UVA plays a role in premature aging.

In addition to the advantages of $TiO_2$, there is also one of the weaknesses that can cause white spots or white cast [1]. One solution to this is to decrease the size of $TiO_2$ on a nanoparticles scale. The size of the particle in nanometers has a large surface area and decreasing the particles size makes the product have different properties that can improve the quality of the material [2]. But when $TiO_2$ is on the nanoparticles scale, it has a high photocatalytic effect and

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can trigger the formation of free radicals that will react with the skin [3]. Thus, the way to solve this problem is by coating nano-$TiO_2$ with nano-Talc.

Talc ($Mg_3Si_4O_{10}(OH)_2$) is a white-coloured mineral that is insoluble in water [4] and is slightly soluble using dilute mineral acid with Mohs strength 1 [5]. The advantage of talc over other materials such as calcium carbonate ($CaCO_3$), mica $(KF)_2(Al_2O_3)_3(SiO_2)_6(H_2O)$ and sarpentine ($C_{21}H_{20}N_2O_3$) is that it has been proven to be an efficient filler in a composite [6], [7]. Talc's advantages in nanoparticles size are large surface areas, easy to disperse, suitable as fillers and stabilizers [8], capable of absorbing and reflecting sunlight with high efficiency so that it can reduce solar heat penetration, and capable of covering the surface of $TiO_2$ forming a protective layer that can inhibit photocatalytic reactions with the target substrate or molecule [9]. So it can reduce direct contact between $TiO_2$ and a substance that is potentially degraded by a photocatalytic reaction [10]. Therefore, nano-$TiO_2$ coating with nano-talc is an action that needs to be tried.

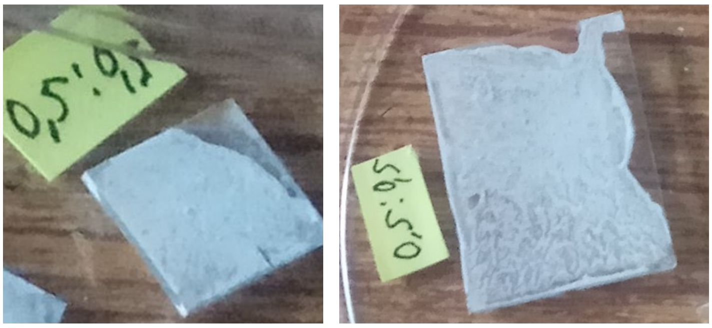
In realizing this research, we used spin coating techniques to synthesize nano-Talc/$TiO_2$ in the form of a thin layer. The sample will be designed with a concentration of 50%: 50%. Next, to determine the cross section shape of the thin coating characterized using SEM (Scanning Electron Microscopy) and UV-Vis spectroscopy for optical properties, determination of band gap and speed of photocatalytic activity. The main objective is to see the structure and cross-sectional morphology of the thin layer as well as understand the optical properties of the resulting sample and describe in detail the optical response of the sample to the concentration of the constituent materials.

## 2. Materials and Method

The materials used are $TiO_2$ (rutile) with a particle size of 280 nm obtained from the LUG (Laju Usaha Gemilang) Chemical Store (Southern Tangerang), the Talc used has a particulate size of 153.46 nm that is sintering at a temperature of 500ºC using hydrothermal methods, ethanol 96%, Aquades and methylene blue. The tools used are prepared glass as a substratum of 1cm x 1cm and 2cm x 2cm, pinsets, spatules, drop pipettes, petridish, chemical glasses (pyrex), beaker glasses, volumetric flask, filter funnel, glass containers, digital scales, UV light 10 watt ($\lambda$ = 254 nm), 325 mesh sieve, ovens, HEM-E3D, Spin Coating. While the characterization tools used SEM (Scanning Electron Microscopy), UV-Vis spectroscopic and UV-vis DR.

The process begins with the preparation of the material, which is milling Talc and $TiO_2$ for 2 hours, respectively, continued by sift to obtain particles of the same size [11]. Next, clean the substrate using aquades, sterilize with ethanol, and dry at room temperature (6 hours) [12]. Then weighing nano-talc powder 0.5 g and nano-$TiO_2$ 0,5 g [13]. At the preparation stage of the sample is done by suspensing 0.5 g of talc into 2.65 m ethanol and 0.5 g of $TiO_2$ into 12.5 ml of ethanol so that the nano-Talc:$TiO_2$ (%w/w) ratio is 50%:50%.

The next step is to make a thin layer, starting with placing the substrate that has been dried using a pinset over the spin coater plate [14]. For the first layer by dropping $TiO_2$ solution (50%) over the substratum until it covers the surface of the substratum using a drop pipette, closing the spin coater, performing a playback at a speed of 2500 rpm for 15 seconds [15]. Continue to the second layer with dropping a Talc suspension (50%) on top of the substrates until covering the surface with a drop-pipette, shutting the spin coater, doing a playthrough at a rate of 2500 rpm during 15 seconds. Then a thin layer of nano-talc/$TiO_2$ is ovened at a temperature of 110ºC for 15 minutes [16].



(a) (b)

Figure 1. Synthesis of Talc/$TiO_2$ with a concentration of 50%:50% for 2 glass sizes: (a) 1 cm x 1 cm (b) 2 cm x 2 cm

As explained in the materials and methods section, material synthesis was carried out using a spin coating technique with a glass substrate. Figure 1 shows Talc/$TiO_2$ synthesized with a concentration of 50%:50%. The resulting material is white and comes in thin layers in 2 sizes each. The number 50%:50% in the sample name refers to the concentration ratio between Talc and $TiO_2$. The surface morphology and cross section testing of thin layers is performed using SEM (Scanning Electron Microscopy) on the sample (substrate size 1 cm x 1 cm), and the optical properties of the thin Layer (substrate size 2 cm x 2 cm) are tested using UV-Vis DR spectroscopes with output values of absorbance, reflection and transmittance, for band gap values are calculated using the kubelka-munk method.

## 3. Results and Discussion

Research in the field of nanomaterials cannot be separated from characterization activities. Material characterization aims to obtain information regarding the specific properties of the material. The characterization used in this research is Scanning Electron Microscopy (SEM), diffuse UV-Vis spectrophotometer (UV-Vis DR) and UV-Vis Spectrophotometer. SEM characterization was carried out by knowing the surface morphology and cross-section of the thin layer. In testing the surface morphology of the Talc/$TiO_2$ thin layer on the sample using a JSM-6360 SEM instrument with 5000x magnification, the results were obtained as in Figure 2.

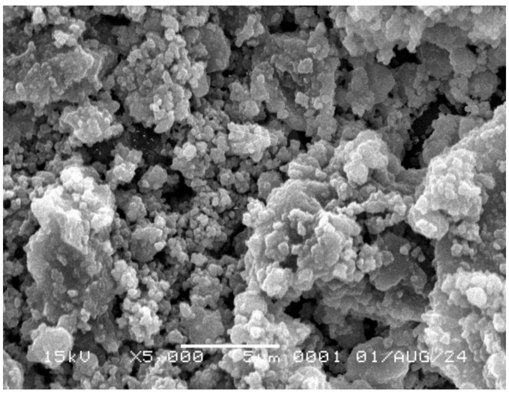



Figure 2. SEM results of the surface morphology of the Talc/$TiO_2$ thin layer

From Figure 2 it can be seen that the cross-sectional imaging results of the Talc/$TiO_2$ thin film shows that the average total particle yield is 145 nm. This The results are in line with existing theory because it is in the range of 10-200 nm [17], [18], so this thin layer can it is said to be nano-Talc/$TiO_2$.

### 3.1. *Cross-section of a thin layer of nano-Talc/$TiO_2$*

In the testing cross-section of the nano-Talc/$TiO_2$ thin layer from the Figure 1(a) sample using SEM instrument JSM-6360 obtained results in Figure 3.

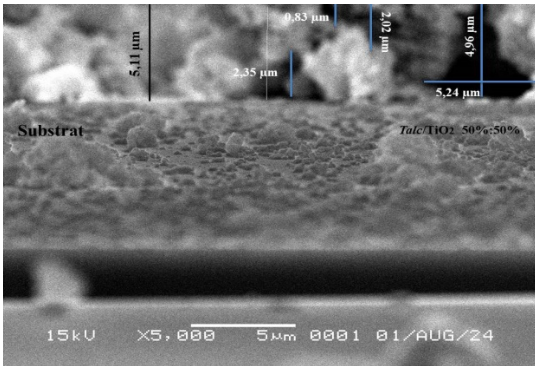
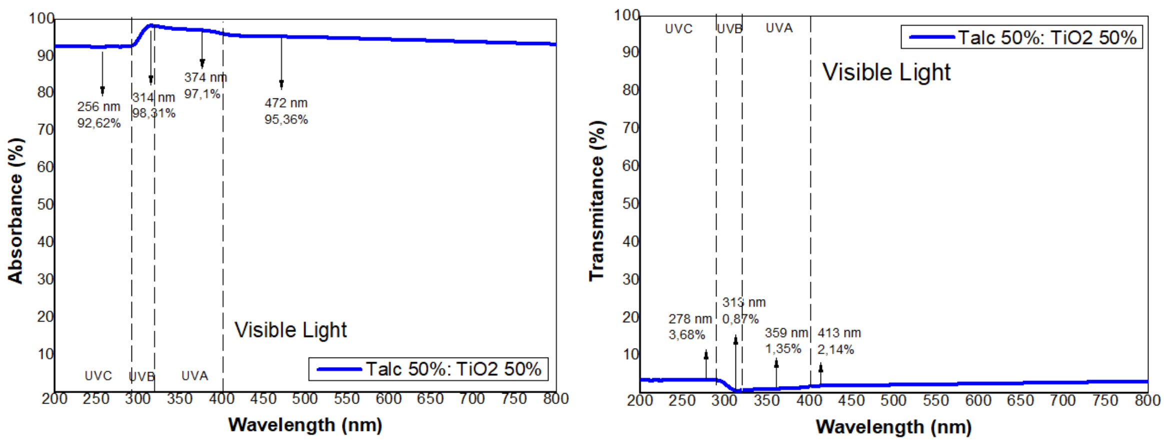



Figure 3. Cross-section thin layer nano-Talc/$TiO_2$ 50%:50%

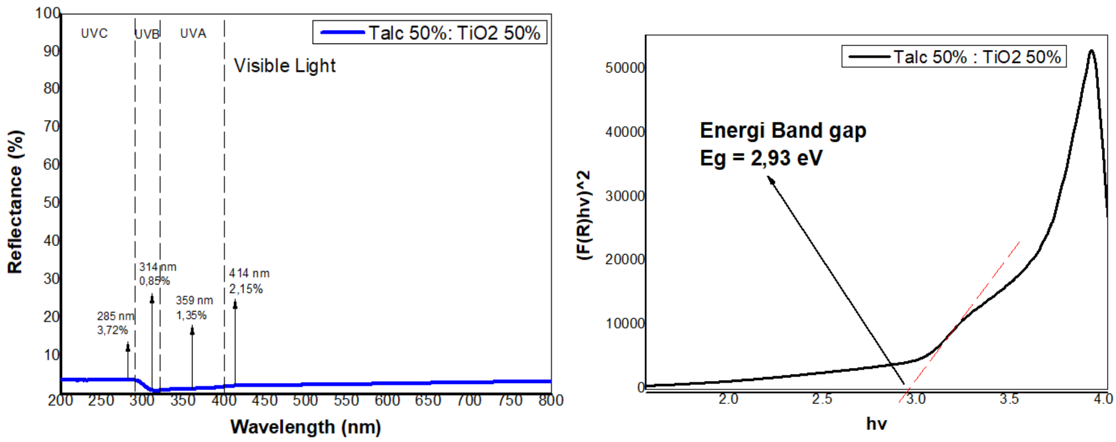
From Figure 3, cross-section thin layer nano-Talc/$TiO_2$ variation 50%:50% has a thickness of 5.11 µm, it is seen that there is still an empty space between the particles and the boundary between the Talc and $TiO_2$ cannot be observed. This layer is not perfectly dense, but has a porosity of (2,35 ; 0,83 ; 2,02 ; 4,96 ; 5,24) µm.

### 3.2. *Optical properties of nano-Talc/$TiO_2$ thin layer*

UV-Vis DR characterization has been carried out using ultraviolet irradiation until visible light appears on the material, then the reflection is explained. In testing the optical properties of the thin layer of nano-Talc/$TiO_2$ on the sample Figure 1(b) using the UV-Vis DR Spectral SPECORD 210 PLUS-223F1936C UV-Vis instrument, obtained data in the form of absorption, reflection and transmission from the sample which can be seen in Figure 4.



(a) (b)




(c) (d)

Figure 4. Results of UV-Vis DR characterization of thin layers of nano-Talc/$TiO_2$ 50%:50%: (a) absorbance (%); (b) transmittance (%); (c) reflectance (%); (d) Band gap energy of nano-Talc/$TiO_2$ thin layer

The results of the UV-Vis DR nano-Talc/$TiO_2$ characterization synthesized with a concentration of 50%:50% are shown in Figure 4. (a) Graph of the relationship between absorption (%) and wavelength (nm) with a wavelength range of 200 nm – 800 nm. From the absorption (%) graph which shows the highest absorption, namely 92.62% in UVC (256 nm), 98.31% in ultraviolet (314 nm), and 97.1% in UVA (374 nm), this result shows that the The highest and most effective peak summation is in the UVB, UVA and Visible Light areas compared to UVC. With a content of 98.31%, this layer is able to prevent skin diseases caused by UV rays, such as sunburn and skin cancer. (b) Graph of the relationship between transmittance (%) and wavelength (nm) with a wavelength range of 200 nm – 800 nm. From the transmission graph (%) it shows transmittance of 3.68% in UVC (278 nm), 0.87% in UVB (313 nm), and 1.35% in UVA (359 nm), and 2.14% in Light Visible (413 nm), these results show that Talc and TiO2 are very effective in protecting against UVC, UVA and visible light. Based on transmission data, the higher the $TiO_2$ concentration in the Talc/$TiO_2$ thin layer, the less transparent the layer is in the UVB to visible light area. Conversely, the greater the concentration of Talc (up to 50%), the more transparent it becomes. The desired nano-Talc/$TiO_2$ thin layer has low transmittance around UVB to visible light to maintain the skin's natural color without residue in the pores. (c) Graph of reflection (%) against wavelength (nm) with a wavelength range of 200 nm – 800 nm. The reflection graph (%) shows a reflection of 3.72% in UVC (285 nm), 0.85% in UVB (314 nm), and 1.35% in UVA (359 nm), and 2.15% in light. Visible (414 nm). these results indicate that Talc and $TiO_2$ are very effective in protecting against UVC and visible light. The results of this study show that the coating process improves the optical properties of nano-$TiO_2$. $TiO_2$ coated with a nano-Talc layer shows higher light absorption, very low reflection and transmission compared to pure $TiO_2$, especially in the visible light area [19], [20]. (d) Graph shows the results of band gap energy analysis of a thin layer concentration of 50%:50% obtained from processing using the Kubelka-Munk method with a band gap energy of 2.93 eV. Based on these results, the greater the $TiO_2$ concentration, the wider the band gap energy value of the resulting material. Band gap energy is the smallest energy to excite electrons from the valence band to the conduction band. $TiO_2$, especially in the Rutile phase with a band gap of around 3.0 eV [21], effectively absorbs UVA and UVB radiation. Higher band gap values are associated with lower reflectivity and higher transparency to visible light, making them ideal for sunscreen bases without white cast.

To analyze the band gap value in Figure 4(d) using the Kubelka-Munk method with a linear relationship graph E (eV) on the x-axis and $(F(R)h\upsilon)^2$ on the y-axis using equation (1) [22], [23] :

$$R'\infty = \frac{R\infty\ (sample)}{R\infty\ (standart)}$$

$$F(R'\infty) = \frac{(1-R'\infty)^2}{2R'\infty}$$

$$F(R'\infty) = \frac{k}{s}$$

$$\frac{k}{s} = \frac{(1-R'\infty)^2}{2R'\infty} \quad (1)$$

Where:

| | |
|---|---|
| R'∞ | = Calculated reflectance |
| F(R'∞) | = Kubelka-Munk |
| k | = Absorption coefficient |
| s | = Diffusion reflectance scattering coefficient |

The band gap energy can be calculated by graphing the relationship between hv (photon energy) and $(F(R)h\upsilon)^2$ and draw a linear graph in the area where the slope is steepest. The band gap energy values are shown in Figure 4.

### *3.3. Photocatalytic activity of nano-Talc/$TiO_2$ thin layer*

#### *3.3.1. The degradation rate is proportional to the absorbance of the UV irradiation time*

In the photocatalytic activity test carried out the degradation rate was comparable to the absorption of methylene blue (3 ppm) with a UV irradiation time of 0-5 hours on a thin layer of nano-Talc/$TiO_2$ 50%:50% obtained in Figure 5.

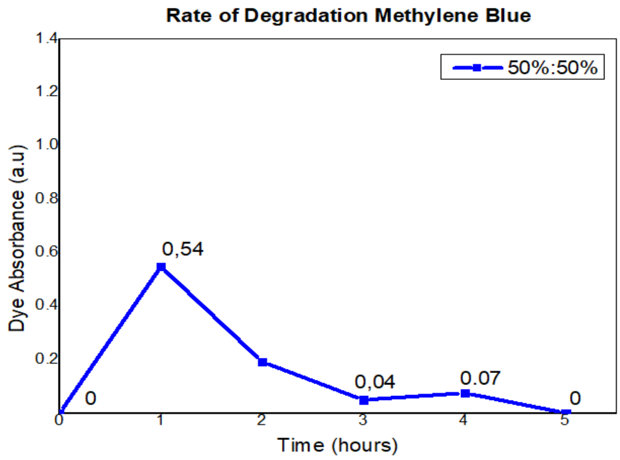



Figure 5. The rate of degradation is comparable to the absorption of methylene blue against the time of UV exposure.

Figure 5 shows a graph that proves the relationship between the degradation rate which is proportional to the absorption and UV irradiation time. The degradation rate value is proportional to the absorption of the nano-Talc/$TiO_2$ thin layer, with a concentration of 50%:50% at various UV light irradiation times for 5 hours. The graph above shows the degradation rate of methylene blue uptake before UV irradiation (0 hour) and 0 au under UV irradiation for 5 hours. There was a decrease in the 3rd hour of 0.04 au and an increase in the 4th hour of 0.07 au. When $TiO_2$ is exposed to UV photons, electrons are excited and react with oxygen to form oxygen radicals. In tests with water and methylene blue, UV light produces hydroxyl radicals from water, causing degradation of methylene blue and high photocatalytic activity as well as high ROS (Reactive Oxygen Species).

### *3.3.2. Degradation rate of Methylene blue absorbance against UV irradiation time*

In determine the constant value of the degradation rate of the thin layer, it can be obtained from the graph of the results of the degradation rate against the UV irradiation time on the photocatalytic activity of the nano-Talc/$TiO_2$ thin layer with a concentration of 50%:50% which can be seen in Figure 6.

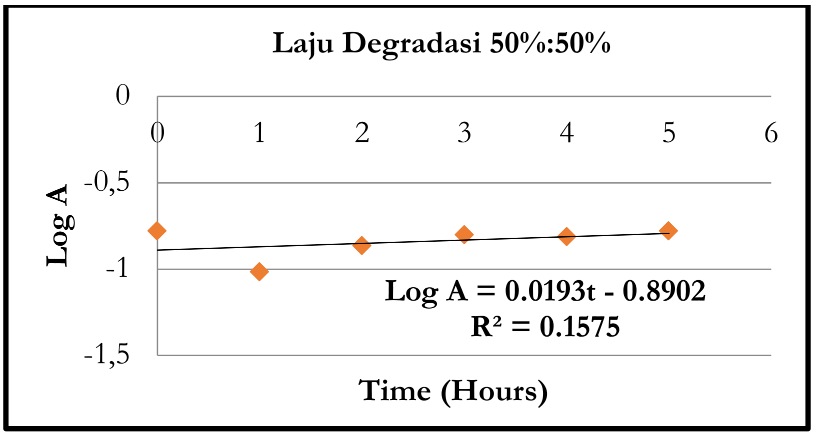



Figure 6. Methylene blue degradation rate versus UV light exposure time

The photocatalytic activity of the samples was evaluated quantitatively using a first-order kinetic model derived from the Langmuir–Hinshelwood equation for quantitative determination of the catalytic efficiency of the synthesized nano-Talc/$TiO_2$ thin films by calculating each first-order rate constant (k) according to the following equation (2) [22]:

$$\ln\left(\frac{A}{A0}\right) = -kt$$

$$\text{Log A} = \text{Log } A_0 - kt \qquad (2)$$

Where A is the absorption at a certain time interval
$A_0$ is the absorption before degradation.

In Figure 6 the data has a good approximation with an $R^2$ value of 0.1575. From this graph, it can be seen that the reaction rate constant (k) is 0.0193/hour. The reaction rate constant value describes how fast the photocatalytic process occurs. In this case, the 50%:50% nano-Talc/$TiO_2$ thin layer has a reaction rate of 0.0193/hour, which means that the 50%:50% nano-Talc/$TiO_2$ thin layer is able to reduce the A value by 0.0193 in one hour. Based on the research results obtained, it shows that the photocatalytic activity in thin layers is caused by the effect of concentration. Large concentrations affect the thin layer of nano-Talc/$TiO_2$ which in turn also affects the photocatalytic activity of the material. The smaller the amount of Talc, the higher the photocatalytic activity. This happens because the more $TiO_2$ compared to Talc that absorbs a material, the greater the surface area that reacts directly with UV light, so that the photocatalytic reaction process will occur more quickly. The higher the degradation, the higher the ROS and this should be avoided in sunscreen products. The research results show that the lowest degradation rate when exposed to UV radiation is 50%:50% with a Talc concentration comparable to $TiO_2$, resulting in a layer with a slower degradation rate. The results of this research show that the coating process succeeded in reducing the photocatalytic activity of nano-$TiO_2$ compared to pure $TiO_2$, with a high level of photocatalytic activity [24].

*3.3.3. UV irradiation time on % degradation thin layer*

The following is the graphical result of UV irradiation time against % Degradation in the photocatalytic activity of nano-Talc/$TiO_2$ thin films varying in concentration 50%:50% at 3 ppm which can be seen from Figure 7.

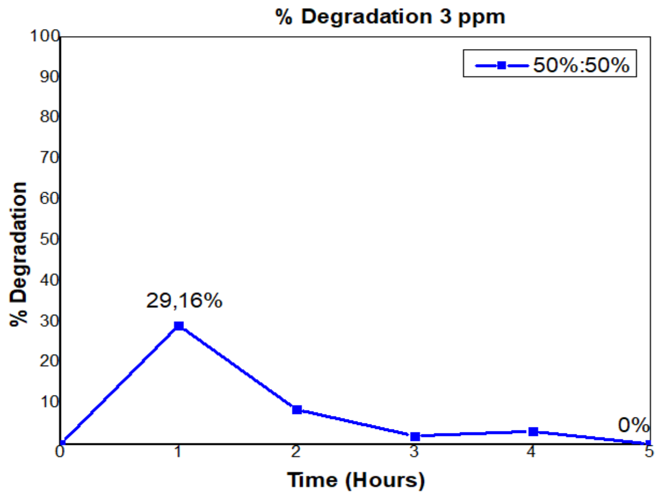



Figure 7. Graph of the relationship between % Degradation and duration of UV irradiation at a concentration of 50%:50%.

Figure 7 shows a graph that proves the relationship between UV exposure time and % degradation. For the photocatalytic activity absorption value of the nano-Talc/$TiO_2$ thin layer 50%:50% with a UV irradiation time of 5 hours. The graph above shows the % degradation before UV irradiation (0 hours) and during 5 hours of UV irradiation, namely 0%. Apart from that, the lowest % degradation value in UV irradiation was at 5 o'clock which produced a value of 0%. The higher the degradation, the higher the ROS and this is something to avoid in sunscreen products. The research results showed that the lowest degradation rate when exposed to UV radiation was 50%:50% with a Talc concentration comparable to $TiO_2$, resulting in a layer with a slower degradation rate. The results of this research show that the coating process succeeded in reducing the photocatalytic activity of nano-$TiO_2$ compared to pure $TiO_2$, with a high level of photocatalytic activity [25].

## 4. Conclusion

Based on research on the optical characteristics of nano-Talc/$TiO_2$ thin layer, it was concluded that the average particle size was 145 nm. Cross Section analysis shows that the layer is not perfectly solid, but there is empty space. Nano-Talc/$TiO_2$ concentration 50%:50% provides the highest absorption, reflection and transmission of UVB, UVA and visible light at low UVC and visible light levels. Furthermore, the influence of the nano-Talk/$TiO_2$ concentration ratio before and during UV irradiation influences the determination of the % degradation of Talk/$TiO_2$ in methylene blue. Providing a Talc concentration comparable to $TiO_2$ results in the lowest % degradation. It can be proven that $TiO_2$ is not reactive when coated with a protective layer of Talc. Large concentration of nano-Talc/$TiO_2$ at 50%:50% reduces % degradation. Providing a Talc concentration comparable to $TiO_2$ resulted in the lowest methylene blue degradation rate, proving that $TiO_2$ becomes non-reactive when coated with Talc. Thus, a concentration of 50%:50% can reduce the rate of degradation and ROS (Reactive Oxygen Species) in the thin layer of sunscreen.

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