Fabrication and Analysis of Optical Properties of Double Layer Glass Surface; Self Cleaning and Glowing Glass

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Abstract: This research was conducted in order to produce a self-cleaning and glowing double layer glass and also to analyze its the optical properties. The glass was engineered by coating the surface with two layers, the thin film of carbon phosphor dots urea - citric acid and TiO₂. Methods of the glass fabricating and coating use simple methods that were sol gel and dip coating techniques respectively. Based on the test analysis of the optical properties by performing the wettability test, the glass that we produced has a contact angle of 0° (superhydrophilic). Once we dripped water onto the glass, we observed that has the water has a spreading wetting. The glass that has been coated with TiO₂ was resistant to fog or vapor (antifogging) as we did not observed any fog or droplets on it. This indicates that the surface already has self-cleaning properties. We then used UV to see glowing effect, and we observed that the glass has fluorescent effect with emission wavelength around 570-590 nm which confirmed that we managed to get glowing glass.

1. Introduction

Dust and particulates in the air can make material especially those made of glass become dirty faster, causing some constraints in transparency as well as visual aspects of the material. Therefore it requires intensive cleaning to maintain cleanliness and transparency on the glass. Conventional cleaners require detergents containing chemicals which can endanger the environment.

The Titanium dioxide has been known having two unique properties, which are hydrophilic and superhydrophilic during UV irradiation and become hydrophobic when not irradiated by UV [1]. One of the uses of the hydrophilicity is to engineer the properties of glass surfaces so that it can degrade organic pollutants on the surface of the glass [2]. This is in line with the research that conducted by Feng (2004), TiO₂ photocatalysts have a self-clean and anti-fog properties that have been demonstrated by the ability of TiO₂ to clean the pollutant by itself [3,4]. In phenomenon of superhydrophilicity, when TiO₂ exposed by photon, pairs of electrons and holes will be generated. The electrons tend to reduce Ti (IV) cations to Ti (III) cation state, and the holes oxidizes the O₂ anion. In this process, the oxygen atom will be apart resulting the emptiness of oxygen. After that, the water molecule can occupy the vacancy of oxygen, and produce an adsorbed OH group, which tends to make...
a hydrophilic surface. The longer the surface irradiated by the UV light, the contact angle between water and the surface will be smaller to near zero. It means that the water has a tendency to spread perfectly on the surface which is coated by TiO$_2$ [1]. Carbon nanodots (CND) provide a broader photoluminescence profile and lower quantum efficiency values (<3%) compare to quantum dots [5]. The addition of citric acid as a carbon source and 1,2-ethylenediamine (EDA) as a surface passivation agent and it is one of the amine molecules, resulted CND with better photoluminescence properties and an unprecedented and have a quantum efficient value 30.2% [6]. By utilizing the properties of photoluminescence in a thin layer of phosphorus carbon dots urea-citric acid, the glass surface will have a glowing properties.

2. Material and Synthesis

Learning In this experiment the thin layers was depositioned twice to the glass substrate (glass slide) with dimension (75x25x0.5) cm that had been cleaned with distilled water and then soaked into solution of ethanol for 5 minutes and then dried at room temperature. The first layer is Phosphor Carbon Dots Urea - Citric Acid to get the glowing properties and the second layer was TiO$_2$ that serves as self cleaning agent. The solution of Phosphorus Carbon Dots Urea-Citric Acid was made by mixing 6 grams of urea [CO(NH$_2$)$_2$] and 0.03 grams of citric acid (C$_6$H$_8$O$_7$) in a solid phase by mashing the two materials without using water. Then the mixture was heated in a microwave with a power of 380 watts for 90 seconds. The heating process resulted a solution of carbon phosphorus dots urea and citric acid. The solution was initially liquid when removed from the microwave, but after being at room temperature and interacting with the air, the solution then became solid. Citric acid is used as a carbon source because citric acid has a uniform molecular weight distribution and is reactive to carbon sources [7]. The TiO$_2$ solution that was made, derived from titanium tetrachloride (TiCl$_4$) as the precursor and ethanol solvent (C$_2$H$_5$OH) with a precursor and solvent ratio of 1: 5 and stirred using a magnetic stirrer for 30 minutes. TiCl$_4$ which was used was 4 mL and C$_2$H$_5$OH was 20 mL.

The glass substrates were pre-conditioned, so that were free of impurities, before being coated with the first solution (carbon phosphorus dots urea - citric acid). The coating of the carbon dots phosphorus solution on the glass substrate was carried out by dip coating method by immersing the glass substrate into the instantaneously heated phosphorous solution after the phosphorus solution was removed from the microwave. It aimed to make the phosphor material being deposited on a glass substrate perfectly. Then the glass substrate was cooled and dried at room temperature. The coating results on the glass substrate are showed in Figure 1 (a). Figure 1 (b) is the glass substrate after several cleanings.

After the coating was cooled, a second coating was made using TiO$_2$. This was performed by using the simple coating method by dripping and flattening the entire substrate surface. In this research, we used five drops of TiO$_2$ which were dripped onto the substrate and then flattened. After the TiO$_2$ deposition it was dried in the open air for 15 minutes to vaporize the remaining solvent remnants [8]. The coating was then calcined with temperature variations of 100°C, 150°C, and 200°C with a calcination time of 60 minute. This process aimed to grow crystals, increase grain homogeneity, and strengthen adhesion forces between layers with substrate [9].
3. Discussion
Development Wettability testing was performed on two substrate types. The first substrate was a substrate which was uncoated with TiO$_2$, as a control, and the second substrate is a substrate which was coated with TiO$_2$. The wettability test was performed in five glass sections to see the consistency of hydrophobicity or hydrophilicity on the glass as shown in Figure 2.

![Figure 2. Water droplets on the glass substrate without TiO$_2$ coating](image)

The contact angle can be formed from water droplets on the ideal solid surface. This occurs because of the mechanical balance of water droplets against the action of three surface tensions, which are liquid-gas ($\gamma_{LV}$), solid-gas ($\gamma_{SV}$), and solid-liquid ($\gamma_{SL}$) then formed a three-phase contact line called the wetting line as shown in Figure 3.

![Figure 3. Illustration of the contact angle formed by water droplet on the homogeneous solid surface](image)

The Young equation correlates the contact angle and the surface tension. Young’s equations form the basis of the quantitative description of the wetting phenomenon as shown by equation (1) [10].

\[
\gamma_{LV} \cos \theta = \gamma_{SV} + \gamma_{SL} \quad (1)
\]

The contact angle can be calculated by rearranging equation (1) into equation (2).
\[
\cos \theta_y = \frac{\gamma_{SV} + \gamma_{SL}}{\gamma_{LV}} \quad (2)
\]

If the solid-gas surface tension is higher than the solid-liquid surface (\(\gamma_{SV} > \gamma_{SL}\)), the right side of Young’s equation will be positive so it must be positive and the contact angle will be less than 90°. Consequently liquid moistened solid surface partially.

**Figure 4.** Effect of calcination temperature (after coating) with the surface contact angle.

On the other hand, if the solid-liquid surface is larger than the solid-gas surface (\(\gamma_{SV} < \gamma_{SL}\)), the contact angles between the water and the substrate were then measured using equation (2). The control substrate have an average contact angle 25°, while the substrates coated with TiO₂ had varied contact angle due to the temperature of calcination of TiO₂ layer. Figure 4 showed the contact angle of coated glass calcinated on temperature of 100°C, 150°C, and 200°C. It is clear that the higher the temperature given, the higher hidrophilic value is. It is proves that the substrate which coated with TiO₂ are increasingly hidrophilic. Particularly at a substrate with calcination temperature 200°C, the measurement of contact angle between the substrate and the water was close to 0°. Shortly after the water was dripped onto the substrate, the water had a spreading wetting which mean the water spread perfectly on the substrate as shown in Figure 5. In phenomenon of superhydrophilicity, when TiO₂ exposed by photon, pairs of electrons and holes will be generated. The electrons tend to reduce Ti (IV) cations to Ti (III) cation state, and the holes oxidizes the O₂ anion. In this process, the oxygen atom will be apart resulting the emptiness of oxygen. After that, the water molecule can occupy the vacancy of oxygen, and produce an adsorbed OH group, which tends to make a hydrophilic surface. The longer the surface irradiated by the UV light, the contact angle between water and the surface will be smaller to near zero. It means that the water has a tendency to spread perfectly on the surface which is coated by TiO₂ [1].

**Figure 5.** Water droplet on the substrat surface with TiO₂ coating
The water spraying tests for substrates that uncoated and coated with TiO$_2$ were also carried out. The results showed that for uncoated substrate, there is a water droplets. On the other hand, the ones that coated with TiO$_2$ made the water spreading wetting and the water always leaded to the lower part of the glass for all temperature variations. This revealed that TiO$_2$ can change the glass surface properties, so it now has self-cleaning properties. The spray test results were presented in Figure 6.

Besides wettability test to see the self-cleaning performance of the substrate, we also performed a visual test to see the resistance of the substrate to the fog/vapor (antifogging). Antifogging test was done by placing the two glass inside the refrigerator for 20 minutes and then we took them out to room temperature. On the substrate uncoated with TiO$_2$, the water granules was formed and then disappered after 20 second. Whereas on the substrate which coated with TiO$_2$, no water grain was found, as shown in Figure 7.

The observation under UV light was performed to see the effect of luminescence on the substrate glass when exposed to UV light. The glass substrate was irradiated by UV light with a wavelength of 365 nm. Figure 8 showed fluorescence effect that appeared on the glass substrate when irradiated with UV light. The edge of glass substrate showed visible yellow luminescence emission wavelength between 570 - 590 nm. Carbon nanodots (CND) provide a broader photoluminescence profile and lower quantum efficiency values (<3%) compare to quantum dots [5]. The addition of citric acid as a carbon source and 1,2-ethylenediamine (EDA) as a surface passivation agent and it is one of the amine
molecules, resulted CND with better photoluminescence properties and an unprecedented and have a quantum efficient value 30.2% [6].

![Figure 8. Glass substrate coat with carbon dots;](image)

(a) without ultra violet radiation; and
(b) during ultra violet radiation

The use of citric acid as a carbon source to synthesize Boron Carbon Oxynitride (BCNO) phosphorus by a simple heating method will produce a uniform luminescence. Whereas if using Polyethylene Glycol (PEG) as a carbon source, the luminescence is uneven [7]. By utilizing the properties of photoluminescence in a thin layer of phosphorus carbon dots urea - citric acid, the glass surface will have a glowing properties.

4. Conclusion
Based A double thin layer as self-cleaning and glowing glass has been produced by coating the glass substrate with TiO$_2$ layer and phosphor carbon dots urea - citric acid. The results of the optical properties analysis on glass substrate coated with phosphorus layer carbon dots urea - citric acid is that the glass showed yellow luminescence effect with emission wavelength of about 570 - 590 nm, and the TiO$_2$ layer changes the glass surface to have superhydrophilic and antifogging properties. Particularly at a substrate with calcination temperature 200°C, the measurement of contact angle between the substrate and the water was close to 0°, on the substrate uncoated with TiO$_2$, the water granules was formed and then disappered after 20 second. Whereas on the substrate which coated with TiO$_2$, no water grain was found.

Acknowledgments
This work was supported by Research Grant from DIPA FMIPA Tanjungpura University, Number 022/SP2H/PPM/ DRPM/II/2016 in the fiscal years of 2016.

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