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Recent graphene oxide/TiO₂ thin film based on self- cleaning application

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Abstract. Graphene oxide/TiO₂ (GO/TiO₂) thin films works as self-cleaning device have been developed in various method onto selected substrates. It was noticeable that graphene oxide is the best form in the group of graphene family. Under self-cleaning application, the wettability test and electroconductivity of the sample was the main characteristic for self-coating study. As planned, by addition of graphene to TiO₂ films produce a highly conductivity, transparent and produced promising enhanced photocatalytic activities. Moreover, superhydrophilic properties of GO/TiO₂ film surface film exhibits more as compared to TiO₂ film only within a short period of time. Thus under this finding, the photocatalytic activity of GO/TiO₂ films will be enhance as a result of improve charge separation efficiency because of the electron injection to graphene from TiO₂ conduction band. Other factor that contribute to self-cleaning activity is the electrical conductivity of the graphene added to TiO₂ thin film. The graphene added to TiO₂ films have a lot of potential in various indoor application due to its fantastic characteristics such as not expensive, transparent, highly conductive, exhibits superhydrophilic properties, and very much photocatalytically active.

1. Introduction

Self-cleaning technology has been given remarkable attention recently. This is due the self-coating and windows have the commercialization factors whereby had promoted many studies to improve this technology. For self-cleaning windows, a glass surface is coated with thin layer coating having about 10 to 25 nm of titania or silica through several deposition methods. The efficiency of self- cleaning performance was controlled by the wettability of the surfaces to obtained good self-cleaning coating [1].

Initially, two types of coating surfaces that should be considered. First, a surface layer of film has a superhydrophilic surface when a liquid drops onto the surface of the film. Second is superhydrophobic surface where a drop of water will maintain its spherical shape on the solid surface or substrate. Figure 1 shows a schematic representation of superhydrophobic and superhydrophilic surface while in Figure 2 shows schematic views of superhydrophilic, hydrophilic, hydrophobic, and superhydrophobic surfaces



(left to right). Other than that, electroconductivity of the coating also give effects to the self-cleaning effect. This happens by the effect of antifouling effect in contrast to the contaminants [2]. Superhydrophilicity surface could avoid fog as soon as water droplets spread form a thin sheet-like water membrane. For self-cleaning purposes, three major mandatory of films quality need to be applied. First, films should possess superhydrophilicity, good photocatalytic activity and high durability. Secondly, the substrate of the film should optical transparency for the coating application especially glass windows. Lastly, an antistatic property is needed in order to decrease the attachment of the contaminants on the surface can be achieved by having a good electroconductivity exhibits in the film [3].

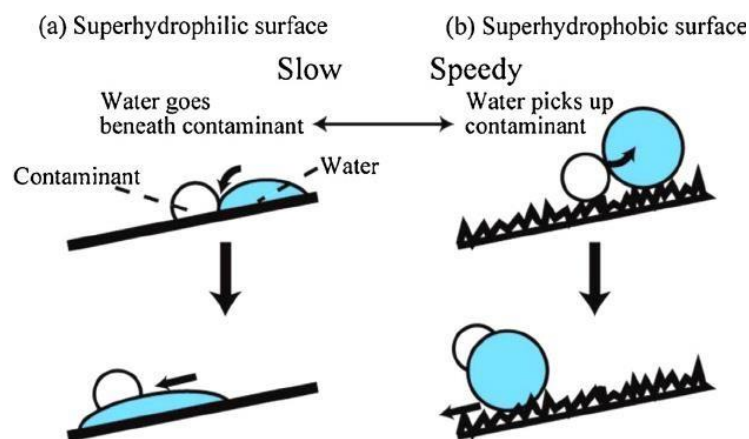


Figure 1. Schematic illustration of self-cleaning processes on (a) a superhydrophilic and (b) a superhydrophobic surface [4].

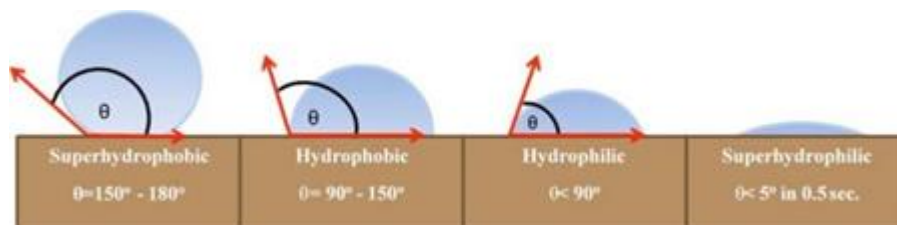


Figure 2. Schematic views of superhydrophilic, hydrophilic, hydrophobic, and superhydrophobic surfaces (left to right) [5].

Studies of titanium dioxide (TiO_2) related to self-cleaning materials has gained remarkable attention for both their powerful characteristics and practical applications in energy and environmental areas [4] [6]. TiO_2 thin film can be developed by several methods such as sol-gel [7] [8], electrochemical deposition [9] and chemical vapor deposition (CVD) [10]. The sol-gel method was mostly developed as a general and powerful approach for preparing inorganic materials such as ceramics and glasses. The TiO_2 thin films may be deposited on the surface of various substrates, such as metals, glass, textiles, ceramics, cement, fibre or bricks to produce a layer that display self-cleaning properties, when there is a source of light [4].

In early 1970's Honda-Fujishima has started a research on semiconductor photocatalyst which is TiO_2 [11], the most favourable photocatalyst with a lot of applications in the environment [12]. TiO_2 has attract great attentions to the researchers because of its excellent properties such as low cost, good stability, favourable chemical and high photo activity. It has been employed in almost studies of photocatalytic degradation of water pollution.

There are three main or common phases that TiO_2 could exist which are anatase, brookite and rutile. Among those phases, anatase phase of TiO_2 , is generally regarded to be the most active under

UV irradiation. However, low activity under visible light irradiation is still the limitation for photooxidation process to occur for the wide application of TiO_2 . To overcome these issues, the reactivity has to be enhanced as well as visible photoresponse of TiO_2 should be improved. Several efforts taken such as noble metal deposition, cationic and anionic doping, sensitization and addition of sacrificial agents to overcome these matters [13]. Though the noble metal deposition has several advantages over others, high cost and low abundance of the noble metals restrict their use in large scale applications [14]. On that regard, many efforts have been made to replace noble metal with other material which has capability to self-cleaning application.

Graphene is one of the suitable elements to replace the noble metals since it has high surface area, sufficient carrier mobility which was found to be $200,000 \text{ cm}^2 \text{ V}^{-1} \text{ S}^{-1}$, high electrical conductivity (10^6 S cm^{-1}), and efficient electron (e^-) transfer from TiO_2 to graphene. This is because its redox potential is just below the conduction band (CB) edge of TiO_2 [15]. Thus, the photocatalytic applications of semiconductor-graphene composites have been extensively studied recently. Decorating semiconductor materials with graphene can enhance their electronic, optoelectronic, electrocatalytic and photocatalytic properties [2]. There are several reports available on reduced graphene oxide (rGO)- TiO_2 pair having better performance [16].

Recently, most of the studies were highlighted on the self-cleaning property of graphene- TiO_2 hybrids. Up to now, the fabrication and application of graphene- TiO_2 hybrids has been scarcely reported especially involving of broadband light absorption and self-cleaning properties. In addition, the self-cleaning property of TiO_2 is depending on the mutual effect between photocatalysis and photoinduced hydrophilicity. Moreover, TiO_2 surface also displays photoinduced superhydrophilic conversion, and can be used as promising material for removal of organic contaminants on the surface of TiO_2 during the photocatalytic oxidation reaction.

Therefore, a simple strategy to fabricate a highly conductive and transparent graphene-loaded TiO_2 thin film will be determined. Figure 3 shows the mechanism of self-cleaning property of graphene- TiO_2 under influence of UV light irradiation.

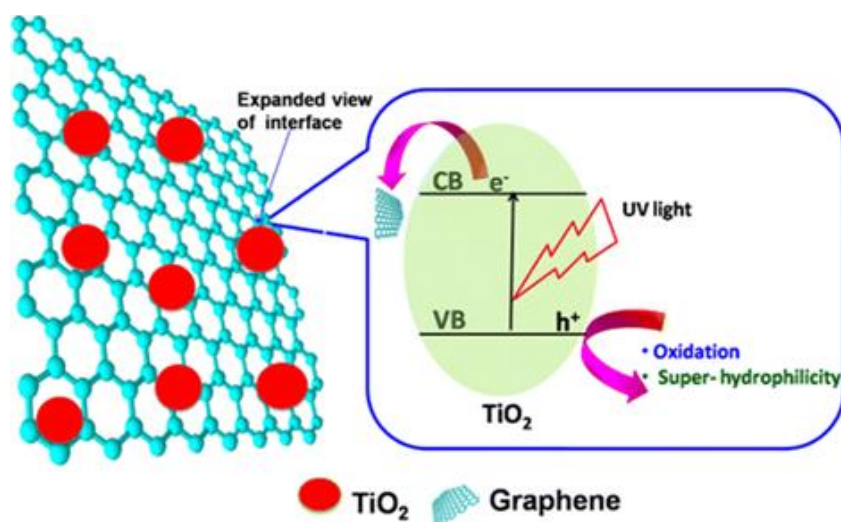


Figure 3. The mechanism of photocatalytic activity graphene/ TiO_2 [17].

The modification of TiO_2 with Graphene element had shown superhydrophilicity within a short time even under a white fluorescent light bulb, as compared to a pure TiO_2 film. The increment of photocatalytic activity of graphene/ TiO_2 films is attributed to its efficient charge separation which provides a pathway for the electron to promote injection from the conduction band of TiO_2 to graphene. Besides that, the electroconductivity of the graphene-loaded TiO_2 thin film also contributes to the self-cleaning function by its antifouling effect against particulate contaminants. The present study reveals

that graphene has the ability to be as a low cost cocatalyst instead of expensive noble metals (Pt, Pd), and the function of graphene can be expanded further by showing its capability of the application of self-cleaning coatings under irradiation of low energy sources.

2. Methods to prepare self-cleaning coatings on various substrates

Graphene oxide/ TiO₂ thin layer was deposited on the transparent substrate to prepare a hydrophilic or hydrophobic interaction. The fabrication of thin films can be coated onto various type of substrates such as glass, silicone, tiles, textile, plastic and many more. The quality of coating of the films were dependent on the selection of method employed. This coating method had given variety responses on the result of self-cleaning property.

Mostly used method to develop a thin films coatings are Chemical vapor deposition (CVD), atomic layer deposition (ALD) and sol-gel method. According to sol gel method, there are other techniques to deposited the coating which are dip coating techniques, spin coating technique or spray coating technique. Concisely, to apply sol-gel method as surface coatings offers a simple and convenient route to synthesis the advanced material systems.

2.1 Chemical Vapour Deposition (CVD)

In 1993, M. Jose-Yacamán *et al.* was the first researcher who introduced this Catalytic chemical vapour decomposition (CCVD) method to produce CNTs [18]. As shown in Figure 4, a simple representation of the chemical vapour deposition process [19]. CVD is a process whereby disconnection and chemical reactions of gaseous product will develop the thin films coating when it is being activated by heat, light or plasma irradiation from the environment. The deposition involves heterogeneous chemical reaction which occurred on a heated surface while in the gas phase occurs homogenous gas reaction. These reactions form powders or films respectively. Indeed, this method does not required of post-treatment process which is suitable to deposit the best crystalline phase of the thin films [1].

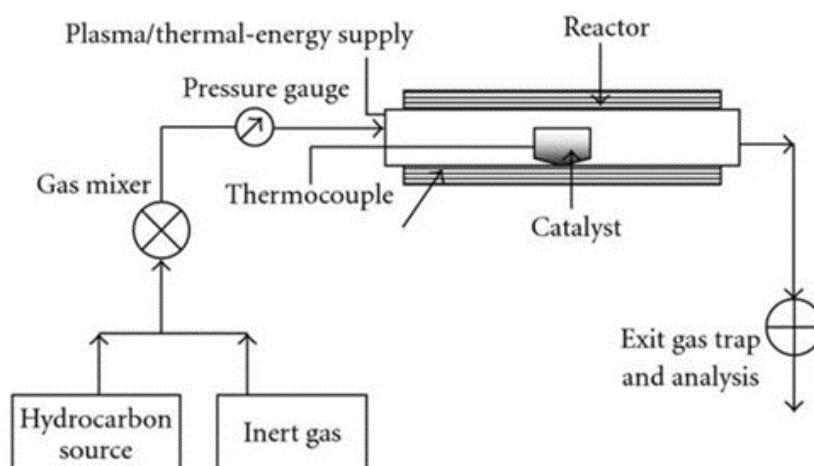


Figure 4. A schematic CNT synthesis diagram by using CVD process [19].

Having the thickness of the film on the sidewall is almost the same as the thickness on the top of the substrate surface proving that CVD has a number of advantages as a method for depositing thin films [20]. The structure of the film can simply be restrained by deposition techniques while film with porous structure will be obtained simultaneously.

In its simplest explanation, CVD involves flowing a precursor gas or gases into a chamber containing one or more heated objects to be coated. Chemical reactions occur on and near the hot surfaces, resulting in the deposition of a thin film on the surface. This is accompanied by the

production of chemical by-products that are exhausted out of the chamber along with unreacted precursor gases. As would be expected with the large variety of materials deposited and the wide range of applications, there are many variants of CVD.

CVD method is commonly used in producing TiO₂ coatings on various substrates. According to Lee *et al.* (2011) studies, TiO₂ nanoparticles that was coated on the glass substrate by CVD was found that coating time influence the amount of TiO₂ deposited and obtained a uniform shape of coating [21]. While Kim *et al.* (2004) obtained a thin layer coating of TiO₂ on Al₂O₃ substrate using CVD. An ultrasonic atomizer self-cleaning glass was used to spray TiO₂ mist on the substrate. The observation showed that the thickness of the film was not dependent to the substrate temperature but dependent on the deposition time. The substrate temperature and the deposition time influence the surface roughness as it was gradually increased with increasing of substrate temperature and the deposition time [22]. Wang *et al.* (2014) has synthesize TiO₂ films by chemical vapor deposition using the recently synthesized precursor Ti(H₃BNMe₂BH₃)₂ with H₂O as the co-reactant at modest temperature. Films grown at substrate temperatures of 350-450 °C consist of a mixture of anatase and rutile; the rutile content increases with temperature [23].

CVD also has a number of disadvantages. One of the primary disadvantages lies in the properties of the precursors. Ideally, the precursors need to be volatile at near-room temperatures. CVD precursors can become a highly toxic material like Ni(CO)₄, also an explosive material like B₂H₆ or display corrosive effect which is SiCl₄. The byproducts of CVD reactions can also be hazardous (CO, H₂, or HF). Some of these precursors, especially the metal-organic precursors, can also be quite costly. The other major disadvantage is the fact that the films are usually deposited at elevated temperatures [20].

2.2 Atomic Layer Deposition (ALD)

As an impressive technique which allows the coating of complicated and high surface area nanostructures in a conformal and homogeneous manner, atomic layer deposition (ALD) also having a precise control of the grown film thickness at nanometer scale [21]. Previously, there have been some ALD depositions on carbon substrates mostly were done on CNTs [24]. However, there have been also trials using graphene and graphene oxide. These latter studies involved depositing oxides (e.g. Al₂O₃ [25]; TiO₂ [26]) and metals (e.g. Pt [27]). Recently an amorphous ALD of TiO₂/fullerene composite had remarkable photocatalytic activity whereas the photocatalytic performance of ALD grown amorphous TiO₂/GO composites were not studied [28].

Yang *et al.* (2016) studied that TiO₂ quantum dots graphene hybrid photocatalyst with high performance toward the degradation of methylene blue has been successfully realized by ALD. This nanocomposite prove that photocatalytic rate has been compared to commercial P25 nanoparticles. ALD has its own strength in photocatalysis study [29]. As reported by Lupen *et al.* (2018), by using atomic layer deposition method, TiO₂ ultra-thin films with a thickness starting from only 15 nm were successfully deposited and chemical properties investigated, showing that anatase phase was the present [30].

2.3 Sol-Gel

Around 1900s, the sol-gel technology was applied by the Schott Glass Company in Jena, Germany where initially this method started in 1800s. The schematic view of sol-gel process for thin film fabrication is given in Figure 5 [31].

Commixture of few numbers of methods for synthesizing materials from solutions which involves gel formation as one of its stages of processing is termed as sol-gel process [32]. Generally, sol-gel method is a the most common process used to produce both hydrophobic and hydrophilic coatings. In this process, first step is to obtain a colloidal solution by hydrolysed or polymerized a precursor material. Next step is to obtain a solid film by heating the sol that has been deposited on the substrate. In order to get ultrafine or spherical shaped powders, thin film coatings, fibrous, porous or dense

materials and extremely porous aerogel materials, the sol-gel process is the powerful method [1].

Studies by Alam and Cameron (2002) demonstrate that with the presence of air, oxygen and nitrogen during annealing process at different temperature, titanium dioxide thin films have been successfully deposited by the sol-gel process and the films are transparent. At higher annealing temperature, as deposited TiO_2 films which basically having an amorphous structure was converted to anatase crystalline phase that was proven in the XRD results. These results suggest that the titanium dioxide thin films can deposited by the sol-gel technique may be used as a high permittivity insulator in thin film electroluminescent devices [33]. On different studies, Suci *et al.* (2009) used sol-gel process and spray coating technique using titanium alkoxide to prepare TiO_2 thin films. In XRD analysis of our titanium precursor powder in Figure 6 shows that the peaks of TiO_2 anatase become more intense starting from 500°C to 600°C of annealing temperature due formation of the TiO_2 anatase crystalline phase [34].

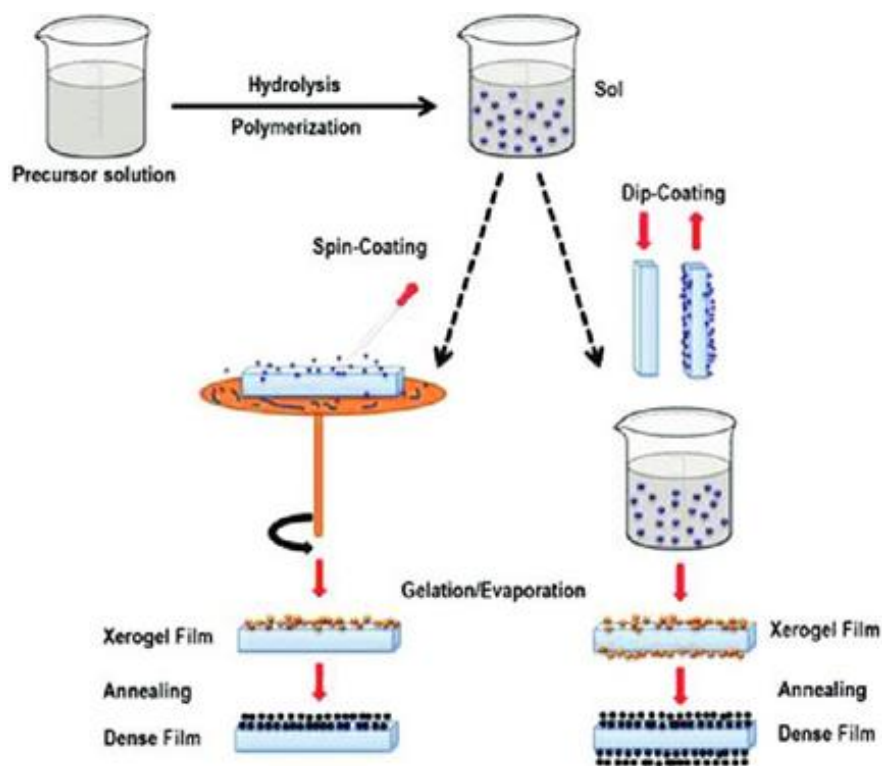


Figure 5. Sol-gel process for the thin film processing [31].

Herein, this sol-gel technology has provided many essential advantages for prepare thin oxide coating. Mostly the interest of this technique towards to the application of sol-gel method whereby involving several advantages such as good homogeneity, ease of composition control, low processing temperature, large area coatings, low equipment cost, and good photocatalytic properties [35].

Although sol-gel method was an awesome synthesis route, there are several serious drawbacks regarding to the sol-gel technique. Large amount of pores and the elimination of undesirable residuals such as hydroxyl and organics occurred during the gelation time whereby most of the gel being the dried and the extreme volume shrinkage. Perhaps, lack of knowledge about the process of sol-gel is the disadvantage that creates wide range of complexities in the process [32]. Moreover, this technique is quite substrate dependent. Metal alkoxides are the most preferred precursor but they are expensive.

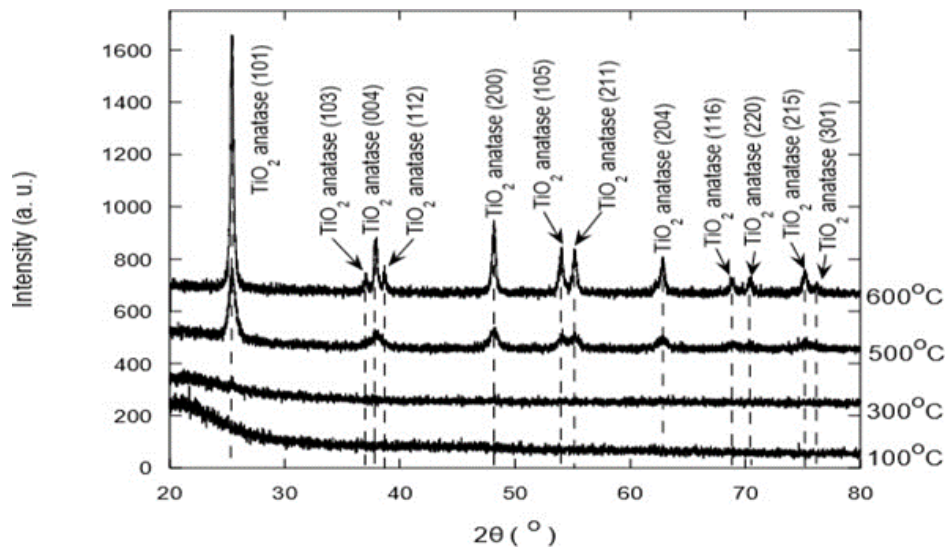


Figure 6. The X-ray diffraction pattern of the titania precursor dried at 100°C, and calcined at 300°C, 500°C and 600°C [34].

Currently, there are some limitations to the applications of TiO₂ film for outdoor use self-cleaning due to the photoinduced superhydrophilicity where self-cleaning effects only occur when UV light having similar intensity to that of natural sunlight was irradiated. Thus, it is most significant source to get visible light active TiO₂ self-cleaning films for indoor applications. Therefore, GO material was applied to TiO₂ to enhanced the photocatalytic activity which was irradiated under visible light as GO is the appropriate replacement of noble metal.

3. Fabrication of graphene oxide/TiO₂ thin film on glass

There are multiple methods to fabricate the prepared sol-gel on the substrate. The mostly used methods are dip or spin coating methods.

3.1 Dip Coating

Process of dip coating involve immersion of substrate containing colloidal solution, remaining the substrate in the solution at fix time, deposition of thin layer coating, drainage of excess liquid and evaporation of solvents at once forming the thin layer coating. Finally, coating thickness is influenced mainly by two major factor which are the deposition speed and solution viscosity. Besides these two major factors, the functionalization of the initial substrate surface, immersion period, withdrawal speed, number of cycles dipped, solution composition, concentration and temperature, number of solutions in each dipping sequence and environment humidity have an influence on the final coating. Basically, to coat on complex structure, dip coating is the best choice [1].

Dip coating deposition have several advantages such as low cost, uniform coating of large areas and high quality of the coated films. [36]. These high quality films are achieved due to the layer-by-layer growth during each dipping process in the aqueous solution [37].

T. Bai *et al.* (2017) successfully fabricate graphene TiO₂ ceramic film by dip coating technique resulted that in GT ceramic composite films, graphene can perform as an efficient reinforcement and lubricant factor [38].

3.2 Spin Coating

Spin coating is a procedure of creating thin films and was widely used to deposit uniform thin film on the substrate. Figure 7 represented a schematic diagram of spin coating process. There are three steps in spin coating process. First is dispense, in which the resin is deposited on the substrate. Secondly,

high speed of rotation in order to thin the resin. Lastly is to remove the excess solvent from the film by drying it. The speed and time of spinning have great influence in the final coating thickness. The higher speeds and longer times of spinning will produce a thinner films [1].

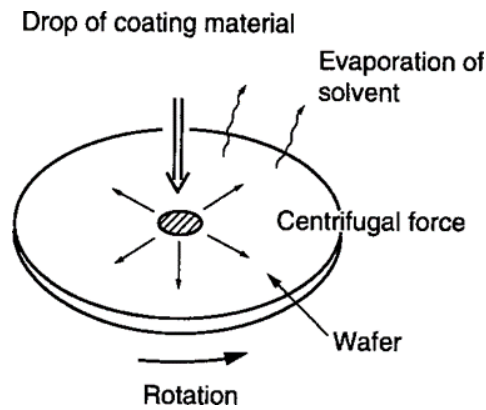


Figure 7. Spin coating process diagram [39].

Small cost, simple operation, manageable control of the chemical composition and facile preparation of large area films has made spin coating technique the most useful and friendly method in depositing a thin film on various substrates. Moreover, it also provides an easy and precise technique and controllable doping ratio and uniform distribution of components in the films. This is very helpful to regulate the microstructure, optical properties and electrical properties of films [40].

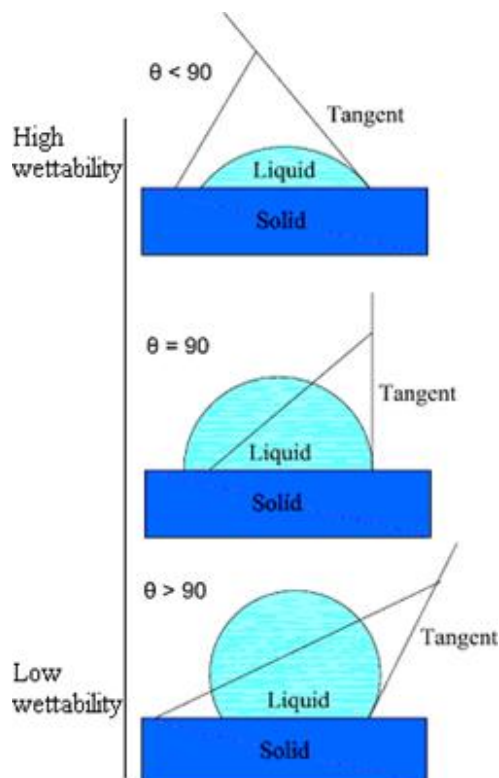


Figure 8. Contact angle and wettability measurements schematic diagram [43].

A. Timoumi *et al.* (2018) has deposited TiO₂-graphene oxide solution on a substrate by spin coating techniques and obtained a homogenous thin film with 250nm thickness. Based on the Transfer

Matrix Method (TMM) model in the theoretical study, it was shown that film thickness strongly influences the optical properties of the film. Moreover, amorphous structure was shown in the TiO₂ and TiO₂-GO thin films [41].

4. Wettability test

Wettability test is a test to measure the ability of water to maintain contact with the surface and this was measured by the water contact angle. Hence, the activity of a photo-induced self-cleaning surface could be evaluated. As the primary data, measurement of contact angles indicates the degree of wetting when a water and surface interact. Small contact angles ($\ll 90^\circ$) indicated to high wettability, while large contact angles ($\gg 90^\circ$) indicates to low wettability. By observing the variation of contact angle during the UV light irradiation, photoinduced superhydrophilicity of the TiO₂ surface can be investigate. This can be easily obtained by using a contact angle meter. Figure 8 shows a schematic diagram of contact angle and wettability measurements [42,44].

There are a few requirements need to be considered to get a standard measurement of contact angle such as:

First, organic substances should be removed from the surface by irradiated it under f UV light for at least 24 hours before the pre-treatment

Surface should be free from contamination while handling it.

To obtained initially high contact angle, the surface can be pretreated with oleic acid.

To observed the decreasing of contact angle, the initial contact angle should be more than 20°.

The measurement of the contact angles was replicate at least five times at five different points and calculate the mean to obtain an accurate result. This should be done measurement should be done within 3 s to 5 s after dripping the distilled water.

5. Conclusion

Graphene and graphene family (eg. graphene oxide and reduced graphene oxide) based composite have unique properties especially for its electronic, biological, mechanical, as well as optical properties. This review highlights the fabrication of the films using the moss common method which is sol-gel dip coating. This selection method provides a good contact of surfaces and by electron transfer from a conduction band of TiO₂ to graphene produce an efficient charge separation in TiO₂. Thus, this is attributed to the enhanced photocatalytic oxidation and photoinduced superhydrophilicity. By control this two matter which was their superhydrophilicity surface and also its electroconductivity behavior has contributed to the very useful application of self- cleaning coating especially under the extremely low UV irradiation for example like indoor applications.

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