A Comparison Study of Different Photocatalyst Preparation Methods: A Review on RGO-BI_2MoO_6 Photocatalyts Synthesis Methods

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Abstract**—** The use of photo catalysts for the degradation of organic pollutants in water is widely growing and the application of photodegradation process has been seen to be effective in most organic pollutants. Different photocatalysts have been studied and various methods for photocatalysts preparations have been developed and investigated. Different methods have been experimented by different researchers in order to see how they improve the physicochemical properties of the photocatalysts since they have an influence on the on their performance. This paper will review different methods for the synthesis of commonly used photocatalysts while focusing more on the RGO-Bi_2MoO_6 as it is one of the new photocatalysts attracting a lot of attention in research. Comparison of different methods will be done to see how each methods affect the final structure of the materials. Moreover, advantages and disadvantages of each method will be compared. The methods to be reviewed will be classified into physical and chemical synthesis methods. Finally, this study will identify the best method to use when developing RGO-Bi_2MoO_6 for degradation purposes.

Keywords**—**Chemical methods, photocatalysts, physical methods, RGO-Bi_2MoO_6, synthesis.

I. Introduction

Photocatalysis has been widely applied in the photodegradation of most pollutants such as dyes, polycyclic aromatic hydrocarbons and other chemicals that are not easily degradable. Various photocatalysts have been employed to degrade contaminants occupying the environment (e.g pesticides and PAHs). Owing to the fact that, the application of photocatalysis in degradation of pollutants has been found to be effective [1]. Photocatalytic degradation of pollutants has been widely studied and different photocatalysts have been explored [2-6]. A great number of photocatalyst have been investigated and different synthesis methods have been tested. Literature indicates that one photocatalyst could be prepared using different methods in order to alter and improve its physicochemical properties [7-9]. This review will provide an overview of photocatalyst preparation methods in general and then narrow down the review to focus more on specific methods used to synthesize RGO-Bi_2MoO_6. There is plenty of literature on the review of TiO_2 preparation methods, but according to the knowledge of the author, there is no review done on the preparation methods of RGO-Bi_2MoO_6.

II. Photocatalysts: Brief description

A photocatalyst is a material that is capable of absorbing light and take it to higher energy in order to drive a chemical reaction. Photocatalysts exist in a form of powder, solids, films, or small particles [10]. A photocatalyst can be synthesized through the incorporation of different kind of materials. There are photocatalysts that are prepared using material that are in different phases and these photocatalysts are referred to as heterogeneous photocatalysts. Some photocatalysts are called homogenous photocatalysts because they are prepared using materials of similar phases [11-12]. A good photocatalyst must depicts the following characteristics: low band gap, high carrier mobility, and non-toxic, chemically stable, inexpensive and efficient light absorption. Photocatalysts can be prepared via physical or chemical methods [12]. The choice of preparation method depends highly on the desired end use of the material. Some of the common preparation methods are discussed below.

III. Physical Methods

Physical synthesis techniques for photocatalysts utilize physical technologies including sputtering, grinding and laser techniques [1]. Physical methods are not very common in developing photocatalytic materials. These methods were used mostly in the olden days. As result a brief discussion will be provided.

A. *Ball milling Methods*

Ball milling methods involve the process of applying great force of rotation and vibrations on the raw big materials to transform them into micro or nanoparticles. The synthesis is commonly carried out in a ball miller. A fusion of different

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materials is transferred in the milling tank that has agate balls of different sizes [13]. Different materials are mixed together in different ratios depending on the structure or form of the desired photocatalyst. If it is desired or necessary to incorporate the photocatalyst with some dopants, then after mixing the raw materials for certain period of time, dopants of choice can be added in the mill [14]. After ball milling the materials at a certain speed, the undesired sizes and materials can be removed using a sieve or any other suitable separation method. The milled sample will then be taken for characterization or analysis to confirm if the materials is successfully synthesized. The disadvantage of this method is that the prepared sample may contain a lot of unknown contaminants and it can only be used for the synthesis of solid materials. TiO_2 is one example of the photocatalysts that have been prepared using ball milling technique [15-16].

B. *Magnetron sputtering Method*

Magnesium sputtering method is another physical method that can be used to prepare photocatalysts. It involves depositing thin films on the surface of the material by sputtering process [17-18]. Sputtering is a process whereby molecules or atoms of a material are ejected from a target and then gets deposited in to a substrate by bombarding the target with high energy ions of non-reactive gases. Magnesium sputtering is classified under physical vapour deposition (PVD) methods [19, 20]. The most important parameters in sputtering technique are substrate, metal precursor, pressure, distance between substrate and target, sputtering pressure and substrate temperature[7]. This technique has been widely used for the modification of photocatalysts due to its low process temperature, good uniformity of thickness distribution and good film adhesion [22]. Its limitation is that it is mostly efficient in preparing thin films photocatalysts.

C. *Pulsed based deposition (PLD) Method*

Pulsed laser deposition method also falls in the category of PVD methods and it also involves the deposition of thin films like the magnesium sputtering method [23]. PLD method is a technique that uses a high-power pulsed laser beam to focus in a beam chamber in order to strike a target material to be deposited. The material to be deposited is vaporised after the laser energy reaches the ablation threshold and creates a plasma plume [24-26]. Then plasma plume subsequently forms a thin film. The pulsed laser depositions usually take place in the presence of background gas such as oxygen [25-27].

IV. Chemical Preparation Methods

In chemical preparation methods, materials are transformed into other materials using chemical reactions. All types of chemical reactions such as precipitation, hydrolysis and pyrolysis can be used to synthesize photocatalysts. Chemical preparation methods that are commonly used to synthesize newly discovered photocatalyst will be briefly discussed in the next sections. These methods include sol-gel, solvothermal, hydrothermal, microwave and sonochemical method.

A. *Sol-gel Method*

Sol-gel method is one of the mostly used wet chemical method in the preparation of nanocomposite photocatalysts and thin films. Different researchers have attempted to improve and modify sol-gel methods and they came up with modified methods such as acid catalyzed and one step sol-gel method [28]. Briefly speaking, sol-gel method involves preparation of a homogenous solution that is converted in to a sol by treating it with a suitable reagent [24]. Sol-gel process takes place according to the following steps in the very same order: (a) preparation of a homogenous mixture, (b) conversion of homogenous mixture into a sol by treatment with an appropriate reagent (usually water with base or acid or without) (hydrolysis &condensation), (c) aging, (d) shaping and (e) thermal treatment[1]. Specifically, the sol-gel method uses inorganic salts or metal alkoxide as precursors, which are converted into a sol via hydrolysis and condensation. Therefore, the obtained sol is dried to obtain solid gel with a desired shape. Finally, the dried gel undergoes a thermal treatment to produce the photocatalyst of desired form [29, 30]. Hydrolysis and condensation are the main steps in the sol-gel process and their general chemical reactions are represented by equation (1) and (2), respectively [52]. The main advantages of sol-gel process include easy preparation of films with high purity, less energy consumption: no need to reach the melting temperature, network structure can be achieved at relatively low temperatures near Tg. Better homogeneity: due to mixing at the molecular level, easy to operate, no requirements for special equipment/apparatus and controlling the film phase structure. In as much this method has some great advantages, it also has some draw backs such as the use of organic solutions that can be very toxic, long time of processing, formation of fine pores, residual carbon and/or hydroxyl groups and the contraction that takes place during processing [52].

$$M - OR + H_2O = M - OH + ROH \tag{1}$$

$$M - OR + HO - M = M - O - M + R - OH \tag{2}$$

B. *Solvothermal and hydrothermal Method*

Hydrothermal and Solvothermal techniques have the same principles and characteristics of photocatalyst preparation process, namely elevated temperature and pressure, and they both require solvents. By definition, solvothermal or hydrothermal method is a process that takes place in a closed reaction vessel (autoclave) inducing a chemical reaction between raw materials in the presence of aqueous or non-aqueous solvents under high pressure and temperature [31-33]. Depending on the reaction conditions, hydrothermal or solvothermal systems can be heterogeneous or homogenous [31]. The only difference between the two techniques is that, in hydrothermal the precursor solution is always aqueous and non-aqueous for solvothermal. In both techniques, precursor solutions are prepared first, then transferred in to an autoclave where crystals develop. After a certain period of time, the mixture is washed and dried to obtain a photocatalyst. Factors such as temperature affects the final structure of the photocatalyst. For instance, if the temperature is not high enough it might prevent the formation of the desired structure of that particular photocatalyst [50]. These two methods have advantages such as low crystallization temperature, controllable crystalline products with different composition, structure and morphology, low energy consumption, environmentally friendly and low costs [34]. The large scale production of photocatalyst using hydrothermal method is achievable, the large scale production of single-crystal potassium tungsten bronze nanowires was successfully achieved using hydrothermal method under mild conditions [53,50]. Due to the advantages mentioned above, a great number of photocatalysts have been prepared using hydrothermal or solvothermal methods.

C. Sonochemical Method

The sonochemical method is a process that allows raw materials or molecules to undergo a chemical reaction through the application of sonochemistry principles which utilize powerful ultra-radiation [35]. Sonochemistry is about providing energy in a particular system through irradiation of liquid with high intensity ultrasonic waves in order to form regions of extreme conditions [36-37]. This method allows the use of milder conditions to form amorphous or crystalline materials. The sonochemical technique has advantages such as no chemical reducing agent required, fast reactions, very small metal nanoparticle can be obtained [38]. However, sonochemical methods are commonly known for only producing spherical metal nanoparticles which limit its application.

D. Microwave Method

Microwave method is essentially a process that involves heating up reactants using microwave radiation for the reaction to start taking place. This process transfers electromagnetic energy to thermal energy. The conversion of electromagnetic energy into heat energy using microwave method is based on two mechanisms, namely, ionic conduction and dipolar rotation [49]. Ionic conduction occurs when polar molecules are experiencing an electric dipole moment aligned with the rotating electromagnetic field. In this process, molecular friction and dielectric loss cause the energy to be released as heat [49,53]. Ionic conduction is a result of dissociated charge ions. In comparison to the former mechanism, this process allows for faster energy transfer and the absorption of microwave irradiation is efficient. The energy transfer is able to uniformly heat material, it also has an ability to heat even thicker materials in a short space of time because the microwaves can penetrate the material and stores energy [39-42]. The heat transfer in a material via microwave method is uniformly distributed, as a result less by-products are formed, if the equipment is well- designed in addition, this method provides a uniform and speedy reaction environment to form materials with homogenous morphology. Microwave-assisted method has been greatly used to synthesize mesoporous materials due to its transferring of energy instead of heat, ability to produce high purity materials, shorter reaction time, and energy saving and heating of selective material.

V. Comparison of Methods for RGO-Bi_2MoO_6 Synthesis

The comparison of different RGO-Bi_2MoO_6 synthesis methods is shown in Table I and it depicts that hydrothermal method is the one that is mostly used for preparing RGO-Bi_2MoO_6. From performing a comparison study, it was discovered that only chemical methods are used, to the best knowledge of the author, no physical methods have been previously used to synthesize RGO-Bi_2MoO_6. The studies done by [45] and [47] shows that both authors used the same preparation method, which is the hydrothermal method. However, the operating conditions were slightly different, as it is evident from Table I that the reaction time in the study done by [45] was 12 hours while in the study by [47], the reaction time was 48 hours. The above observation implies that the reaction time has a great influence on the final structure of the photocatalyts. The preparation method that had longer reaction time (48 hrs) resulted in the formation of nanoplates photocatalyst. In the study done by [43], hydrothermal method was used but a slightly higher temperature of 150℃ was used and the reaction time was made shorter, the reaction ran for 10 hours only. These conditions led to the formation of irregular sheets.

TABLE I: Review of RGO-Bi_2MoO_6 Preparation Methods

Ref.	Method	Use	Reaction conditions	Precursors	Structure
[43]	One-pot hydrothermal	Acted as an anode	150℃, 10 hrs.	C_{12}H22BiN_3O_{14}, Na_2MoO_4.$_2H_2O$	Irregular sheets
[44]	solvothermal	Removal of Methylene blue	180℃, 12 hrs.	$Bi(NO_3)_3$. $5H_2O$, Na_2MoO_4. $2H_2O$	microspheres
[45]	Hydrothermal	Removal of Methylene blue	140℃, 12 hrs.	$Bi(NO_3)_3$. $5H_2O$, $(NH_4)_6Mo_7O_{24}.4H_2O$	microspheres
[46]	One-pot solvothermal	For doping with other metal	160℃, 20 hrs.	$Bi(NO_3)_3$. $5H_2O$, Na2MoO4.2H2O	Not specified
[47]	Hydrothermal	Degradation of Bacteria (E.coli)	140℃, 48 hrs.	$Bi(NO_3)_3$. $5H_2O$, $(NH_4)_6Mo_7O_{24}.4H_2O$	nanoplates
[48]	Facile UV light reduction	Water oxidation	5 of hrs. irradiation	N/A	Microspheres + nanosheets

VI. Conclusion and Recommendations

The use of photocatalysts is a very broad concept in a sense that it can be applied in the removal of contaminants in the environment, in water splitting and it can also be used as an anode. In addition, there is vast number of contaminants in the environment that cannot be easily removed by traditional methods and requires the application of photocatalyst. However, for effective application of photocatalysis, the synthesized photocatalyst must exhibit certain physico-chemical properties which are conducive for the photo-degradation of the contaminants. Therefore, different researchers will always use different methods during the preparation of photocatalysts such as to ensure that the desired physico-chemical properties are obtained. The choice of synthesis method will depend on the type of pollutant to be degraded or the end-use of the photocatalyst. Based on the literature it can be concluded that chemical methods are mostly used in the preparation of RGO-Bi_2MoO_6 and among chemical methods, hydrothermal method is very common and it has been proven to produce a more efficient, chemically stable RGO-Bi_2MoO_6 [51,52].

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