



# The preparation of reduced graphene oxide-TiO<sub>2</sub> composite materials towards transparent, strain sensing and photodegradation multifunctional films



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## ABSTRACT

In this study, reduced graphene oxide-TiO<sub>2</sub> (rGO-T) composite films fabricated by a spray coating method are proposed to develop a multifunctional material for the application of strain sensing and photodegradation. This work provides a simple and versatile approach to prepare rGO-T as a multifunctional material. The drastic synergy effect was found simply by mixing graphene oxide with TiO<sub>2</sub>. The obtained multifunctional rGO-T composite films showed relatively high optical transmittance (around 60% at a 550 nm wavelength), excellent strain sensing property with gauge factors (GF) of 12–23 and enhanced photocatalytic property. The remarkable performance could be attributed to the combination of electrical and mechanical properties of rGO nanosheets and photocatalytic performance derived from TiO<sub>2</sub> nanoparticles. Overall, this work could provide new insights into the research of the graphene-TiO<sub>2</sub> composite materials and facilitate their application in a broad range.

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## 1. Introduction

Since the discovery of graphene [1], researchers have been fascinated by its superior electrical conductivity, excellent mechanical properties, large surface area, highly optical transparency, excellent thermal/chemical stability, and high carrier mobility [1–5], as well as its versatility for a wide range of applications. These characteristics have so far stimulated its use in many applications, such as transparent conducting films, sensors, catalyst supports, supercapacitors, batteries, solar cells, nanoelectronics and many others [6–11]. A particularly promising research direction would be to incorporate graphene sheets in composite materials to achieve materials with improved or extended functionalities. Nevertheless, graphene is poorly soluble in water and polar organic solvents, which makes it difficult to synthesize graphene-based composites in an aqueous environment. Unlike hydrophobic graphene, graphene oxide (GO) is hydrophilic due to the oxygen containing functional groups on the sheet surface, which favors its good solubility in solvents and provide fertile opportunities for the processability of this nanomaterial and the

construction of GO-based hybrid composites [12].

Titanium dioxide (titania, TiO<sub>2</sub>) nanomaterials are generally believed to be the most available and commercially cheapest photocatalyst because of their high efficiency, inexpensive, non-toxicity, chemical stability and friendly to environment [13–15]. The overall efficiency of photon utilization by TiO<sub>2</sub> is however limited by electron-hole recombination, photon scattering and the intrinsic physical properties of TiO<sub>2</sub> ( $E_{\text{bg}} = 3.2$  eV for anatase) [16]. The considerable efforts have been made to enhance the photocatalytic behaviour by hybridizing TiO<sub>2</sub> with other materials such as noble metals or carbon allotropes [17–21]. Generally, there are three fundamental approaches to improve the photocatalytic activity of TiO<sub>2</sub>: band-gap tuning and/or extension of excitation wavelength by use of photosensitizers, minimising charge carrier recombination, and promotion of the forward reaction and adsorbance of reactants on the surface of photocatalysts [11]. Obviously, the combination of GO or graphene (a large theoretical specific surface area, a high intrinsic electron mobility) with TiO<sub>2</sub> therefore presents the opportunity to simultaneously cover at least two of the mechanisms of photocatalytic enhancement. Recently, both graphene and GO have been used to synthesize series of graphene- or GO-TiO<sub>2</sub> hybrid materials by various methods, and their photocatalytic activated were studied [22]. A range of

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different methods have been proposed to synthesize the graphene-TiO<sub>2</sub> composite materials, including hydrothermal method [23], solvothermal method [24], and sol-gel method [25]. Those techniques are somehow not simple, require specialized equipment, and it may be not easy to quantify the ratio between composite compounds. Therefore, it is highly desirable to seek for other methods which are easy aqueous processing potential for large-scale fabrication with cost efficiency. In addition, conventional powder photocatalysts, however, have a serious limitation—the need for post-treatment separation in a slurry system. After the photocatalytic process, the photocatalyst has to be removed by processes not always easy and fast. The recovery of TiO<sub>2</sub> nanoparticles from solution is still a difficulty in widely extending its application in practice. Furthermore the catalyst cannot be easily used again. A possible solution is to prepare composite films, which can be removed from solution and regenerated for further use after purification processes. Although powders can produce excellent photocatalytic materials, for many practical applications films are more appropriate [26]. Recently, a simple process of spray coating is proposed to form graphene films, which is very beneficial to the substrates with a large surface area and/or complicated surface structures [27,28], and this method is considered as a promising candidate for creating graphene-TiO<sub>2</sub> composite films.

Up to now relatively little focus has been on the effects of TiO<sub>2</sub> on the structure and properties of graphene, and as yet virtually no attention has been directed to exploring the other properties of graphene-TiO<sub>2</sub> composite materials. In addition, there is an increasing interest in extending functional film properties to multifunctionality, which can substantially enhance the added value for commercial use. It is widely known that graphene-based thin film have been widely applied, especially in strain sensing fields [28–30]. Strain sensors are ubiquitous smart devices which have a wide range of application where the mechanical deformation or structure change has to be detected, such as damage detection, characterization of structures, fatigue studies of materials and the internal activities in human body. As the strain sensor market in 2013 is expected to exceed 4.5 billion USD, graphene strain sensors have enormous potential value in this field. The gauge factor (GF) is a common figure of merit to show the sensitivity of the transduction from mechanical to electrical parameters. The relation between the change in electrical resistance and the applied strain is revealed by GF, through

$$GF = (\Delta R/R_0)/\varepsilon \quad (1)$$

where  $\Delta R/R_0$  is the normalized electrical resistance variation, and  $\varepsilon$  is the mechanical strain. The combination of TiO<sub>2</sub> and graphene may give rise to a multifunctional film with enhanced or extended functionalities, which has potential industrial applications.

Herein, we prepared multifunctional films based on reduced graphene oxide-TiO<sub>2</sub> composite materials by a direct mechanical mixing method and followed by a simple spray coating method. Compared with other deposition methods, spray coating is shown to be fast and low-cost with controllable and simple process to fabricate rGO-T composite films with tunable properties which is ideally suitable for mass production. Fabrication of multifunctional rGO-T composite films, which combine the strain sensing property of graphene and photocatalytic property of TiO<sub>2</sub>, are of great interest to material science. In this paper we show that it is possible to retain the strain sensing property of the graphene while enhancing the functionality of the sample with the high photoactivity and TiO<sub>2</sub>. The photocatalytic performance and the strain sensing property of the multifunctional films were systematically investigated. Results presented in this paper will confirm the multifunctionality of rGO-T composite films.

## 2. Experimental

### 2.1. Materials

Graphite oxide (Average size: 5  $\mu\text{m}$ ) was purchased from The Sixth Element Co. Ltd., Changzhou, China. Hydriodic acid (HI, 45%), TiO<sub>2</sub> (Anatase, 25 nm), methyl orange (MO) and anhydrous ethanol were purchased from Sinopharm Chemical Reagent Co., Ltd, China. Deionized water was used throughout the work.

### 2.2. Preparation of multifunctional films

The suspension of GO (0.1 mg/mL) was prepared by dispersing the graphite oxide powder into water with the aid of ultrasonication for 2 h. TiO<sub>2</sub> solution (0.1 mg/mL) was prepared by dispersing TiO<sub>2</sub> powder into deionized water with the help of ultrasonication for 1 h. GO-T composite materials were prepared by simple mechanical mixing and sonication in accordance with corresponding proportion. And anhydrous ethanol was used to improve the wettability of the dispersion. After that, spray coating was carried out with a commercial airbrush (Ustar CD-601, Taiwan). GO-T composite films were fabricated both on the rigid (poly carbonate) and flexible (rubber) substrates using spray coating method with a good dispersion of mixture solution. The films were deposited on poly carbonate to facilitate the testing of photocatalytic performance, and in order to characterize the performance of the strain sensing more clearly and obviously, other samples are deposited on the thin and stretchable rubber. The reduction of GO-T films by HI vapor was carried out by vaporizing HI solution at 100 °C. The weight ratio of initial GO to TiO<sub>2</sub> was from 0 to 100%. Two groups of the rGO-T composite materials were prepared which was recorded as T-x%rGO (the amount of T is fixed to 0.15 mg) and rGO-x%T (the amount of GO is fixed to 0.15 mg), respectively. The former group was used to study the photocatalytic performance of the composite film while the latter was used to investigate the strain sensing property.

### 2.3. Photocatalytic experiments

The photocatalytic property of the as-prepared T-x%rGO composite films was evaluated by measuring the degradation of methyl orange (MO, with a concentration of 1 mg/L) in aqueous media under UV irradiation which was carried out with a 175 W ultraviolet lamp. During the photocatalytic experiment, one piece of the film (4 cm<sup>2</sup>) was dipped into 50 mL of the above MO solution. The lamp was positioned 10 cm away from the beaker. An aliquot of 3 mL of suspension was taken at given time intervals. In order to characterize the stability and the reusable of the film, 5-cycle photodegradation for MO was carried out. Each cycle lasts for 5 h. After each cycle, the films were washed by water and anhydrous ethanol for several times, and then re-dipped in fresh MO solution. The degradation of MO was evaluated by the Lambert-Beer law via the absorbance peak at 465 nm.

### 2.4. Strain sensing experiments

The static strain sensing characteristics were evaluated in static testing and the strain behavior was tested by an electronic tensile testing machine (DXLL-5000, Shanghai D&G Measure Instrument Co. Ltd.). The rGO-x%T composite film was stretched by the tensile testing machine (loading speed, 5 mm/min), meanwhile the digital multimeter (UNI-T UT203) was applied to measure the change of electrical resistance during the tensile deformation of the film.

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