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Spray pyrolysis of graphene oxide based composite for optical and wettability applications

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Abstract

In this study, silica–graphene oxide nano–composites were prepared by sol–gel technique and deposited by spray pyrolysis method on glass substrate. The effect of changing the graphene/silica ratio on the optical properties and wetting of these nano–structures has been investigated. The structural and morphological properties of the thin films have been studied by x–ray diffraction spectroscopy (XRD), field emission scanning electron microscope (FESEM), energy dispersive x–ray spectroscopy (EDS) and atomic force microscope (AFM). XRD results show that silica structures present in the synthesized films exhibit amorphous character and there is a poor arrangement in graphene plates along their accumulation directions. The relationship between the contact angle of the water drop and the surface of thin films was analyzed by surface roughness. The results show that the contact angle is also decreased by decreasing the surface roughness. Absorption and transmittance spectra obtained from (UV–vis) of the studied films were used to compute and determine some optical parameters such as absorption coefficient, transmittance rate, optical gap, refractive index and extinction coefficient of the films. The calculated optical band gaps of films decrease by increase the silica contents in these structures.

1. Introduction

A macroscopic combination of two or more separate materials with a certain common surface between them is called a composite [1]. The composite is composed of two main parts: matrix and amplifier. The matrix keeps the amplifier in its relative location by enclosing the amplifier. The amplifier improves the mechanical properties of the structure. In fact, we combine or composite them to modify and optimize the physical and chemical properties of the materials. A nano–composite is also a composite with one or more components less than 100 nm in dimension. The nano–composites are composed of two phases. The first phase is a crystalline structure that is actually the base or matrix of the nano–composite and may be polymer, metal, or ceramic. The second phase is the nanometer-scale particles that are distributed within the first phase (base material) as amplifier (filling material) for the specific purposes such as strength [2], resistance [3], electrical conductivity [4, 5], magnetic properties [6, 7]. Nano–composites have the potential applications in technology [8, 9], information [10], communication [11], medical care [12], and water disinfection [13]. Nano–composites made of nano–particles placed on carbon substrates such as graphene oxide [14–17] have been one of the topics of interest to recent research in science and engineering. These carbon substrates have a very higher surface area than volume, making them suitable for a variety of practical applications [18]. In 2020, Zhang et al reported ultrasonic-assisted electrodeposition was used to fabricate the nickel/graphene oxide composite coatings with high hardness, low friction coefficient, and high wear resistance [19]. According to their results, disruption of graphene with polystyrene increased the electrical conductivity of the composite. The different techniques such as chemical reduction method [20, 21], modified Hummers’ method [22], hydrothermal [23], ultrasonic-assisted electrodeposition [24], chemical method [25], molecular dynamics simulation [26] and in situ sulfuration process [27] are used for the synthesis of nano–composites. In this study, we intend to prepare the...
silica-graphene oxide nano–composites by sol-gel technique and investigate the effect of percentage change in graphene oxide on the optical properties and wetting of these structures.

By definition, wetting means how much liquid is absorbed by a solid surface. The wetting rate of a surface is measured by calculating the contact angle between a solid and a liquid. Young equation is generally used to calculate the wetting rate of a substrate [28]. But this equation is based on the fact that the solid surface is completely flat, but as we know, at the microscopic scale, all surfaces have some roughness. Therefore, other models have been presented to describe the surface wetting, taking into account the surface roughness. The first model was introduced by Wenzel [29]. He assumed that the liquid penetrates into the surface roughness. Another model is Cassie-Baxter model, assuming that surface roughness traps air beneath the liquid surface [30]. In this work, we attempt to study the structural, optical and wettability properties of Graphene oxide and Graphene oxide based composites. We also try to investigate the level of surface roughness and its relationship to contact angle.

2. Experimental procedure

2.1. Sample synthesis
Graphene oxide nano–fluid at 1 mg ml\(^{-1}\) concentration (Borhan NanoScale Company, Iran), Tetraethyl orthosilicate (TEOS) (Merck), ethanol and deionized water were used as the primary material denoted as GO. 10, 20, 30 ml of graphene oxide at 1 mg ml\(^{-1}\) concentration was mixed with ethanol and stirred. 2 ml of TEOS was added to each of the above solutions and stirred. The final solution was diluted by adding the deionized water and left at room temperature for 24 h. These solutions were denoted by SG10, SG20 and SG30, respectively.

In addition to graphene oxide solution at 1 mg ml\(^{-1}\) concentration, 35 ml of the above solutions were deposited on glass substrate by spray pyrolysis device. The distance of nozzle about 30–40 cm and the deposition rate of 2–2.5 were selected. The plate temperature was 150 °C for graphene oxide and 400 °C for SG composites.

2.2. Characterization techniques

2.2.1. X-ray diffraction (XRD) examination
X-ray diffraction is a low-cost and widely used technique for investigating the structural characteristics of materials. XRD technique is used to determine the properties of crystalline structure such as lattice constant, lattice geometry, qualitative determination of anonymous materials, and phase determination of crystals. In addition to size determination of crystals, single crystal orientation, stress, tension and lattice defects. XRD machine of D8 Advance Bruker YT model were operated to characterize the structure of tested films and nanopowders synthesized using CuK\(\alpha\) radiation (\(\lambda = 1.5418 \text{ Å}\)) at room temperature over 2\(\theta\) values between 5° and 80°.

2.2.2. Field emission scanning electron microscope (FESEM) test
FESEM is one of the most popular microscopic methods that can be utilized to study the morphology and topography of a material. The surface microstructure and morphology of GO, SG10, SG20, SG30 films were investigated using field emission scanning electron microscope (FESEM), MIRA3TESCAN-XMU model. The voltage used to capture these images was 10 KV.

2.2.3. Atomic force microscope (AFM) scan
In atomic force microscope, the force between the scanning needle and the sample surface, which causes the cantilever to bend, is measured by the detector. These microscopes can also be used for studying the mechanical properties, abrasion or scratching, in addition to the types of nanolithography and production of nanostructures and nano-machining. In this study, AFM image was prepared to obtain the information on topographic properties and the level of surface roughness of GO, SG10, SG20, SG30 thin films.

2.2.4. Contact angle measurements
The self-cleaning phenomenon depends on the contact angle. The contact angle is formed at the interface of the three phases of solid, liquid, and gas at the contact point of the liquid drop with the solid surface. In general, if the contact angle is less than 90°, the surface is called hydrophilic, while if the angle is more than 90°, the surface is called hydrophobic. Surfaces with the contact angle near zero are called superhydrophilic, and surfaces with the contact angle more than 150° are called superhydrophobic. Hydrophobic surfaces have very low surface energy, while surface energy of hydrophilic surfaces is too high [16].

In this study, the contact angle property of GO, SG10, SG20, SG30 thin films are investigated. Imaging of a 2 \(\mu\)l drop with a magnification of 50 at 20 °C was performed using AM-7013MZR, Dino-Lite, Taiwan.
3. UV–vis measurements

The optical absorption and transmittance spectra were obtained by UV–vis (U3500 spectrophotometer) for the studied films to determine the optical parameters such as absorption coefficient, transmittance rate, optical gap, refractive index and extinction coefficient of the films.

4. Results and discussion

4.1. Structural and morphology analysis of GO and SG composites

X-ray diffraction spectra of GO, SG10, SG20, SG30 samples are shown in figure 1. As can be seen from the figure, the graphene oxide thin film has a peak of (001) at an angle of 10.9°. This peak is belonging to graphene oxide [31]. This finding is in agreement with the literature [32–34]. Applying Bragg equation (nλ = 2d sin θ), the interlayer distance in graphene oxide amounts to 8.147 Å. This value is greater than the reported ones for graphite, i.e. 3.36 Å [35]. The reason for this increase is maybe because of the existence of functional groups including oxygen such as carboxyl, hydroxyl and epoxide on both sides of the graphene oxide plates and its edges. This peak (10.9°) disappears in the diffraction spectra of SG samples, indicating that it has been converted into reduced graphene oxide as a result of heating. SG10 sample shows a peak at 31.77° with an interlayer distance of 2.926 Å. This peak is belonging to cristobalite (102), mineral polymorph of silica [36, 37]. Presence such a peak is attributed to introducing silica to the system. The aforementioned peak vanished when the ratio of graphene increases, and this is ascribed to lowering oxygen contents of graphene oxide as a result of graphitization—occurring [35]. As it can be seen in figure 1, in SG10, SG20 and SG30 nano-hybrids, a broad peak is seen at angles of 26.1°, 23.45°, 23.95°, that is related to the silica structure (SiO₂) (JCPDS File no. 47–1301). The presence of this broad peak indicates that the silica structure is amorphous and there is poor arrangement in the graphene plates along their accumulation directions.

In other words, these structures are composed of stacked multilayer graphene sheets with silica nanoparticles, which can be clearly seen in FESEM images [38, 39].

Figures 2(a) and (b) display morphology and topography features of the films. As can be seen from figure 2(a), that is related to graphene oxide thin film, the monolayer plates with a lateral size about several micrometers are observed.

The surface of the films is relatively smooth. Graphene oxide sheets are stacked and randomly accumulated during the heating process. In figure 2(b), small white circles are noticed, which are related to SiO₂ nanoparticles on the graphene oxide surface and are very small in size and below 50 nm. These nano-particles are randomly scattered on GO surface. The surface roughness increased after the decoration of the surface of GO plates with SiO₂ nano-particles. In SG10 sample, which has the highest Si/GO ratio, the highest number of silica nano-particles was observed. Nucleation and growth of spherical SiO₂ nano-particles on GO surface occurs due to the reactions of hydrolysis and condensation of TEOS with oxygen groups present on graphene oxide plates.
The cross-section images of the samples of graphene oxide and SG10 thin films are shown in figure 3. The surface of graphene oxide cross-section also displays a relatively flat surface. SG10 cross-section image also shows spherical silica nano-particles.

Field emission scanning electron microscope (FESEM) with energy dispersive x-ray (EDX) system was used to determine the elemental composition of the films. The voltage used to perform these tests was between zero and 10 kV. The results of energy dispersive x-ray spectroscopy of GO and SG10 thin films are shown in figure 4. X-ray energy diagrams are plotted based on the amount of x-ray energy received from each energy level. Each of the peaks shown in these diagrams is allocated to a specific atom. The elements identified in the results of energy dispersive x-ray (EDX) test also are completely anticipated and confirm the successful synthesis of these compounds. In graphene oxide thin film, only carbon and oxygen are reported. In SG10 thin film, in addition to carbon and oxygen atoms, Si atom is also observed, confirming the presence of SiO₂ nano-particles.

As can be seen from figures 4(a) and (b), the intensity of Si raises from 1100 in GO film to 2000 in SG10 film. This is attributed to that Si peak in GO thin film is related to the glass substrate used for the deposition and the intensity in SG10 thin film increased because Si nanoparticles inserted into the composite structure in addition to the silicon glass substrate.

The obtained images by AFM are depicted in figure 5. As shown in figure 5, the sample of graphene oxide thin film has a relatively homogeneous surface, with high roughness.
By incorporating silica nano-particles into SG10 sample, the surface roughness is increased and the silica nano-particles are clearly visible. In SG20 thin film sample, where the ratio of silica to GO is decreased, the surface roughness is also decreased and the silica nano-particles are scattered. By further decreasing the SiO₂/GO ratio in SG30 sample, silica nano-particles are not visible and most of the surface is covered by graphene oxide films. Since GO nano-particles are randomly accumulated, the surface roughness is increased compared to SG20 sample and this finding agrees well with the previous work [40, 41, 43].

4.2. Contact angle measurements

The contact angle property of GO, SG10, SG20, SG30 thin films are investigated and shown in figure 6. The contact angle of the drop with the surface in GO, SG10, SG20, SG30 samples record 55.0°, 55.6°, 12.1°, 37.3° respectively. The different factors affect the contact angle, such as the amount and type of impurity, surface roughness and surface homogeneity. Surface roughness is one of the most important factors affecting the interaction between the liquid and the surface. The higher the surface roughness, the greater is the angle of contact. As we know, graphene oxide is hydrophilic. AFM images also show that its surface roughness is relatively high, so the contact angle of its thin film is less than 90° and equals to 55°, indicating the surface hydrophilicity of this film. Silicon oxide is hydrophobic but here the silica nano-particles content is very low and
therefore it does not cause much change in the contact angle, and AFM images show that the surface roughness is high and therefore the contact angle of SG10 is similar to that of GO. In SG20 and SG30 samples, silica nanoparticles are smaller and have less surface roughness, so they have a lower contact angle. SG20 sample, as shown in figure 5, has the lowest roughness and thus the lowest contact angle and is a relatively good hydrophilic surface.

Figure 5. 2D and 3D AFM Images of GO, SG10, SG20, SG30 thin films prepared by spray pyrolysis method.
5. Optical properties of studied films

Figures 7 and 8 show the absorption and transmittance spectra of GO, SG10, SG20, SG30 thin films prepared by spray pyrolysis method, respectively. These figures show the permitted optical transitions of the electron between the occupied states of the valence band and the empty states of the conduction band. Researchers have reported that graphene oxide thin film has an absorption band at a wavelength of about (230–270) nm [43].

In graphene oxide thin film prepared in this study, the maximum absorption peak is at 360 nm and a shoulder peak is also seen at 382 nm. These peaks are related to the $\pi-\pi^*$ and n-$\pi^*$ transition of C=C and C=O bonds, respectively. These peaks have shifted to longer redshift wavelengths compared to other reports, indicating the conversion of graphene oxide to reduced graphene oxide as a result of heating during the deposition process. The absorption of light occurs in the ultraviolet region and blue part of the spectral range, relating to n-$\pi^*$ transition of carbonyl groups [44–47]. Comparing the absorption and transmittance spectra of pure graphene oxide and SG nano-composite shows that incorporating the silica into graphene oxide plates increases the transmittance rate and decreases the absorption of the films, which is the expected result because the pure silica is similar to glass and in UV–vis wavelength range, it is very transparent. In SG30 thin film, which has the lowest Si/GO ratio, it is observed the highest absorption and lowest transmittance compared to two SG10 and SG20 samples. SG10 thin film with the highest Si/GO ratio has the highest absorption and the least transmittance between the films.
In order to calculate the optical gap and determine its type, the optical absorption coefficient of the films, \( \alpha(\lambda) \), is calculated using the equation

\[
\alpha(\lambda) = 2.303 \frac{A}{t}
\]

(1)

Where, \( A \) is the measured UV–vis absorption spectrum of the samples and \( t \) denotes their thickness. From the famous Tauc formula, \( E_g \) can also be obtained \[48\]

\[
\alpha h\nu = \beta (E_g - h\nu)^n
\]

(2)

In this equation, \( \beta \) is an energy-independent constant, \( E_g \) is the optical band gap, and \( n \) is the constant that specify the type of optical transition and its value for direct transition is \( 1/2 \) and for indirect transition is \( 2 \) \[49\]. Accordingly, in order to calculate the optical band gap, the diagrams of \( (\alpha h\nu)^2 \) must be plotted versus \( h\nu \). Diagram of \( (\alpha h\nu)^2 \) versus \( h\nu \) for GO, SG10, SG20, SG30 thin films prepared by spray pyrolysis method at different wavelengths is plotted in figure 9.

In GO sample, the highest absorption occurs in UV region, with a band gap of about 2.25 eV. Incorporating silica nano-particles resulted in decreasing the band gap from 2.25 to 1.75 eV in SG10 sample and this value roughly remains unchanged for the other samples (SG20 and SG30). These significant change in band gap size may be attributed to the presence of silica nano-particles, whereas band gap size has not been affected by decreasing the ratio of nano-particles to graphene oxide \[50\].

The refractive index is a complex function. The imaginary part of the refractive index is called the extinction coefficient, which reflects the absorption of electromagnetic beam by that material. The refractive index, \( n \), and the extinction coefficient, \( k \), of GO, SG10, SG20, SG30 thin films were calculated using the transmittance and reflection spectra, which are depicted in figures 10 and 11.

The refractive index of the samples can be obtained by this equation \[51\],

\[
n = n_s \left( 1 + \frac{\sqrt{R}}{1 - \sqrt{R}} \right)^{1/2}
\]

(3)

Where, \( n_s \) is the refractive index of the substrate and \( R \) is reflection. The refractive index for the glass substrate used is 1.51. The extinction coefficient, \( k \), is also obtained by this relation \[52\],

\[
k = \alpha \lambda / 4\pi
\]

(4)

Where, \( \alpha \) is the absorption coefficient of the films and \( \lambda \) is the light wavelength. Figure 10 shows the refractive index changes for these samples in terms of wavelength. As can be seen in the figures, the refractive index of GO film is in the range of \( (1.4–1.6) \) eV, which is consistent with the results reported by others. In the visible range, the refractive index value of the GO film is 1.5 and it increases approximately to the value of 1.8 by adding the silica nano-particles. Refractive index value is 1.5 for the GO film. By adding the silica nano-particles, refractive index value first increases to approximately 1.8 and then decreases slightly to the value of 1.7. In regions where the refractive index increases with increasing the frequency, this behavior is called normal dispersion, which is the usual behavior of all transparent materials. In regions where the slope of the diagram of refractive index is negative, it is related to absorption. In this region, a longer wavelength light breaks when passing through the material compared to a shorter wavelength light, which this behavior is called abnormal dispersion. GO, SG20,
SG30 samples at (300–350) eV and SG10 samples at (350–400) eV exhibit the abnormal dispersion behavior. As shown in figure 11, the extinction coefficient of all samples is consistent with the absorption coefficient. If the electromagnetic wave passes easily through the structure, the material has a low extinction coefficient and vice versa, if the beam hardly penetrates into the material, it has a high extinction coefficient. At the energy range of (400–800) eV, GO thin film has the highest extinction coefficient compared to the other films, and SG10 sample, which has the highest ratio of silica particles, exhibits the lowest extinction coefficient.

Figure 9. Diagram of \((\alpha h \nu)^2\) versus \(h \nu\) for GO, SG10, SG20, SG30 thin films prepared by spray pyrolysis method at different wavelengths.

Figure 10. Refractive index, \(n\), of GO, SG10, SG20, SG30 thin films prepared by spray pyrolysis method.
In this study, the effect of changing the graphene/silica ratio on the optical properties and wetting of the synthesized thin films is investigated. The XRD patterns find out the presence of graphene oxide monolayer in GO thin film, whose characteristic peak is observed at an angle of 10.9°. XRD results also show that the silica structure formed in these films is amorphous and that there is poor arrangement in graphene plates along their accumulation directions. Analysis of EDX spectra of the samples were also investigate that there are no additional elements in these structures. The contact angle measurements of the studied films reveal that SG20 sample has the lowest contact angle (12.1°) and is a relatively good hydrophilic surface and according to AFM images the aforementioned sample has lowest roughness. Examining the optical absorption transmittance spectra derived from (UV–vis) of GO film shows light absorption in GO thin film occurs in ultraviolet region and blue part of the spectral range, which related to the n-π* transition of the carbonyl groups. Transmittance spectra of pure graphene oxide and SG nano-composites explore that introducing silica into graphene oxide plates increases the transmittance rate and decreases the absorption of the films. In SG30 thin film, the highest adsorption and lowest transmittance compared to SG10 and SG20 samples. The optical band gap of all thin films was calculated using the Tauc equation. The band gap of SiO₂ decreases with increasing Si/GO ratio. These significant changes in band gap size may be due to the presence of silica nano-particles. The value obtained for the refractive index of graphene oxide film is in the range of (1.4–1.6) and it is a relatively good hydrophilic surface and according to AFM images the aforementioned sample has lowest roughness. Examining the optical absorption transmittance spectra derived from (UV–vis) of GO film shows light absorption in GO thin film occurs in ultraviolet region and blue part of the spectral range, which related to the n-π* transition of the carbonyl groups. Transmittance spectra of pure graphene oxide and SG nano-composites explore that introducing silica into graphene oxide plates increases the transmittance rate and decreases the absorption of the films. In SG30 thin film, the highest adsorption and lowest transmittance compared to SG10 and SG20 samples. The optical band gap of all thin films was calculated using the Tauc equation. The band gap of SiO₂ decreases with increasing Si/GO ratio. These significant changes in band gap size may be due to the presence of silica nano-particles. The value obtained for the refractive index of graphene oxide film is in the range of (1.4–1.6) eV, which is consistent with the results of others. In the visible range, the refractive index value of the GO film is 1.5 and it increases approximately to the value of 1.8 by adding the silica nano-particles. In SG20 and SG30 films, which the ratio of silica particles is low, the diagram of refractive index is similar to that of GO film and the value of refractive index does not change a lot. Examining the extinction coefficient of the samples shows that at the energy range of (400–800) eV, GO thin film has the highest extinction coefficient and SG10 sample has the lowest extinction coefficient.

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