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Grafen Oksit Miktarının In₂O₃-İndirgenmiş Grafen Oksit Kompozit Filminin Yapısı ve Morfolojisi Üzerindeki Etkisi

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Öne Çıkanlar:

- In₂O₃-rGO nanokompozitleri elektrokimyasal teknik kullanılarak üretildi.
- Nanokompozit yapısı ve morfolojisi üzerine GO miktarının etkisi araştırıldı.
- Değişen GO miktarları için In₂O₃-rGO nanokompozitleri üretildi.
- En optimum hacim oranı 1In:1GO olarak tespit edildi.

ÖZET:

Bu çalışmada, elektrokimyasal bir teknik kullanılarak ilk defa tek kapta indiyum oksitle indirgenmiş grafen oksit (In₂O₃-rGO) kompozitleri üretildi. Grafen oksit (GO) miktarının kompozit yapının kompozisyonu ve morfolojisi üzerindeki etkisi incelendi. Bu amaçla, GO ve In³⁺ iyonları içeren elektrolit çözeltileri farklı hacim oranlarında karıştırıldı. Biriktirmeler farklı elektrolit kompozisyonlarında sabit potansiyelde gerçekleştirildi. Farklı deneysel parametreler altında hazırlanan kompozit yapıların karakterizasyonları X-ışını kırınım spektroskopisi (XRD), X-ışını fotoelektron spektroskopisi (XPS), Raman spektroskopisi ve alan etkili taramalı elektron mikroskobu (FESEM) teknikleri kullanılarak incelendi. En iyi kompozisyona sahip In₂O₃-rGO kompozitinin hacimce 1:1 GO:In³⁺ elektrolit oranında elde edildiği sonucuna varıldı.

Anahtar Kelimeler:

- Elektrokimyasal depozisyon
- İndiyum oksit
- Nanokompozit
- İndirgenmiş grafen oksit

Influence Of Graphene Oxide Amount on The Structure and Morphology of In₂O₃-Reduced Graphene Oxide Composite Film

Highlights:

- In₂O₃-rGO nanocomposites were produced using electrochemical technique.
- The effect of GO amount on nanocomposite structure and morphology was investigated.
- In₂O₃-rGO nanocomposites were produced for varying GO amounts.
- The most optimum volume ratio was determined as 1In:1GO.

ABSTRACT:

In this study, indium oxide-reduced graphene oxide (In₂O₃-rGO) composites were produced in one-pot using an electrochemical technique for the first time. The effect of graphene oxide (GO) amount on the composition and morphology of the composite structure was investigated. For this purpose, electrolyte solutions containing GO and In³⁺ ions were mixed at different volume ratios. Deposits were carried out at constant potential in different electrolyte compositions. The characterizations of the composite structures prepared under different experimental parameters were investigated using X-ray diffraction spectroscopy (XRD), X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and field effect scanning electron microscopy (FESEM) techniques. It was concluded that the In₂O₃-rGO composite with the best composition was obtained in 1:1 GO: In³⁺ electrolyte by volume.

Keywords:

- Electrochemical deposition
- Indium oxide
- Nanocomposite
- Reduced graphene oxide

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INTRODUCTION

Nanocrystalline metal oxides have many applications in magnetic data storage, batteries, catalysts, and sensors. Among various metal oxides, indium oxide (In₂O₃) is a remarkable n-type semiconductor metal oxide with a wide band gap (3.55–3.75 eV) (Prakash et al., 2011; L. Zhu et al., 2024). In₂O₃ nanostructures have been reported to exist in different morphologies, such as nanowires, nanorods, nanoparticles, nanospheres, nanosheets, microspheres, and microtubes (Zhu et al., 2007; Fang et al., 2022). In₂O₃ nanocrystals are widely used in many potential applications, such as microelectronic device materials in solar cells, transparent conductors, gas sensors, touch panel displays, photocatalysis, sensors, lithium-ion batteries, and light-emitting diodes (Anand et al., 2016; Sekkat et al., 2024).

In₂O₃ offers advantages such as the location of the conduction and valence band edges, which have excellent conductivity and stability for direct photoelectrolysis of water and the correct support of water redox potentials. However, In₂O₃'s large band gap exhibits low conversion efficiency when utilizing visible light. Adding dopants to the In₂O₃ lattice can improve optical and electrical properties. Researchers have shown that doping In₂O₃ with metal cations such as Nb, Al, Sn, W, Ti, V, and Mo significantly affects optoelectronic properties (Mostafa et al., 2018). In addition, carbon-based materials such as graphene, graphene oxide (GO), and reduced graphene oxide (rGO) can also be preferred for this purpose (Doğan, 2019; Doğan et al., 2019; Eryiğit et al., 2022). rGO, the rising star of the carbon family, has a thick planar layer consisting of a sp²-bonded carbon structure. Like graphene, rGO is a type of material with some extraordinary properties, such as ultra-large specific surface area, flexibility, high electrical conductivity, superior chemical stability, and exceptional mechanical strength. The rGO structure can be easily obtained by reducing GO using various techniques such as chemical, electrochemical, and hydrothermal. The literature describes different techniques for the synthesis of In₂O₃-graphene composites, such as supercritical fluid (Ioni et al., 2021), hydrothermal synthesis (Mao et al., 2024), microwave-assisted synthesis (Alaizeri et al., 2023), and etc. However, one-pot electrochemical synthesis of In₂O₃-graphene nanostructures has not been performed so far. Nitrate ions have an important role in the nanocomposite formation mechanism between In₂O₃ and rGO during electrodeposition (Kurt Urhan et al., 2019). The reduction of GO to rGO simultaneously occurs through the formation of hydroxide and the conversion to the metal oxide form.

In general, spin coating, hydrothermal synthesis, spray pyrolysis, electrodeposition, microwave-assisted synthesis, and inkjet printing coatings are among the most widely used techniques for the synthesis of metal oxide thin films (Mostafa et al., 2018). Most of these methods are performed with toxic organic solvents and surfactants (Vakh & Koronkiewicz, 2023; Younis & Osman, 2023). Moreover, the reaction parameters include high temperature and long reaction times. The electrodeposition technique has many advantages, such as direct production on the electrode surface, low cost, and easy application.

In this study, In₂O₃-rGO nanocomposites were synthesized by the one-pot electrochemical deposition method. In₂O₃-rGO nanocomposites with different GO amounts were prepared, and structural and morphological characterizations of all samples were performed. Among these samples, the nanocomposite structure in the GO:In³⁺ electrolyte mixture with a volume ratio 1:1 was found to have the best morphology and composition.

MATERIALS AND METHODS

Synthesis of In₂O₃-rGO

10mM indium chloride (InCl₃) salt was used as the source of In³⁺ ions. Graphene oxide (GO), InCl₃, and potassium nitrate (KNO₃) were supplied by Sigma. To prepare the GO suspension, 1mL containing 2mg/mL GO (2mg GO/1mL H₂O) was added to 0.1 M 10mL KNO₃ solution. GO and In³⁺ solutions were mixed in varying volumetric ratios such as 1:1, 2:1, and 1:2. BASi100 model potentiostat was used for the electrochemical deposition of In₂O₃-rGO nanocomposites. The working, counter, and reference electrodes in the three-electrode cell system were indium tin oxide coated glass (ITO), Pt wire, and Ag/AgCl electrodes. The deposition potential of In₂O₃-rGO nanocomposites was determined as -1.4 V by recording a cyclic voltammogram (CV).

Characterization of In₂O₃-rGO

For the structural characterization of the produced In₂O₃-rGO nanocomposites, x-ray diffraction spectroscopy (XRD, Rigaku MiniFlex Benchtop Powder XRD Diffractometer 2005E302 model), x-ray photoelectron spectroscopy (XPS, Specs-Flex trademark) and Raman spectroscopy (WITech alpha 300R) techniques were used. Morphological characterization was performed using field emission scanning electron microscopy (FE-SEM, Zeiss Sigma 300). XRD analyses were recorded using Cu K α radiation ($\lambda = 1.5405 \text{ \AA}$) at 2 θ between 10 and 80 degrees with 4-degree steps. Core-level XPS analyses were measured with a sensitivity of 0.1 eV. Raman spectrum was analyzed using a 532 nm and 100mW laser source.

RESULTS AND DISCUSSION

Structural Analysis

To determine the phase structure of In₂O₃-rGO nanocomposite, XRD patterns of materials deposited in various volume ratios were collected in Figure 1a. All XRD spectra presented in Fig. 1a show well-defined peaks at $2\theta = 21.53^\circ, 30.65^\circ, 51.07^\circ, 60.70^\circ$ corresponding to (211), (222), (440) and (622) planes of In₂O₃ (Shifu et al., 2010, JCPDS card No. 89-4595). In addition, diffraction peaks related to the graphite crystal structure originating from rGO are also present. Besides, no diffraction peaks belonging to impurity species such as metallic In or In(OH)₃ are obtained in the XRD spectrum of the nanocomposite.

To examine different C types, number of graphene layers, and structural defects of In₂O₃-rGO nanocomposite, Raman spectroscopy was recorded as shown in Fig. 1b. In the 2In:1GO nanocomposite as volume ratio, the Raman spectrum of In₂O₃ film appeared as a plateau curve. When the volume ratio was changed to 1In:1GO, a spectrum with some peaks related to the vibration modes E_{1g} (131 and 304 1/cm), E_{2g} (626 1/cm), A_{1g} (494 1/cm) and 364 1/cm associated with In₂O₃ was obtained in the Raman spectrum (Gan et al., 2013; Wiranwetchayan et al., 2018). In addition, D (1330 1/cm) and G (1560 1/cm) bands originating from rGO were also observed. However, only the peaks belonging to rGO were present when the volume ratio was 1In:2GO. As a result, both In₂O₃ and rGO peaks in the deposited films occurred in the 1In:1GO mixture.

XPS analysis was performed to determine the chemical composition and bonding state of the elements in the nanocomposite film. Figure 2a shows the XPS spectrum of In 3d core levels. The binding energy of In 3d detected at 443.9 eV corresponds to In 3d_{5/2}. The peak corresponding to In 3d_{3/2} was detected at 451.5 eV. The difference between the In 3d_{3/2} and In 3d_{5/2} peaks is 7.6 eV, confirming the presence of In³⁺ in In₂O₃ nanoparticles (Sawant et al., 2021). Moreover, the XPS spectrum of In3d for In₂O₃ has broader spectral components than metallic In. For these reasons, it can

be said that the In₂O₃-rGO nanocomposite contains the In₂O₃ structure. Figure 2b shows the XPS O 1s core level spectra of the nanocomposites prepared at different concentration ratios. It is generally known that the oxygen 1s core levels of crystalline metal oxides are located in the relatively narrow binding energy range of 529.0 eV–531.1 eV (Zatsepin et al., 2019). The 530.3 eV band belongs to the XPS O 1s core level signal coming from the regular oxygen sublattice of In₂O₃ (Gurlo et al., 1997). In addition, the peak in the 529.0 eV region allows us to conclude that adsorbed OH groups or GO is due to the presence of entirely non-reducing oxygen groups.

To determine the reduction of GO to rGO, C1s core level XPS spectrum was recorded (Figure 2c). C1s spectrum for GO contains two main peak regions: C-C/C=C at 284 eV–286 eV and C-O functional groups at 286 eV–289 eV (Al-Gaashani et al., 2019; Öztürk Doğan & Kurt Urhan, 2023). In the spectrum in Figure 2c, as the GO ratio increases in In₂O₃-rGO nanocomposite, the peak in the 287 eV region was obtained more dominantly. Oxygen functional group peaks also decrease depending on the decrease of GO amount in the In:GO ratio. Similarly, increasing the GO amount also increased the peak intensity of the C-C bond. For In₂O₃-rGO nanocomposites prepared at varying concentration ratios, it was determined that the most suitable composition for the reduction degrees of GO was a 1In:1GO ratio. When the In3d, O1s, and C1s XPS spectra of the nanocomposites prepared at varying concentration ratios were examined, the 1In:1GO ratio was chosen due to the presence of In³⁺ in the In3d peak, the lattice O1s peak, and the absence of oxygenated functional groups of GO. Furthermore, the atomic percentage values of each element were investigated using the data in the general scanning XPS spectra. Table 1 shows the changes in the ratio of the In, O, and C elements in the In₂O₃-rGO nanostructures produced at different concentration ratios. As seen in Table 1, different concentrations also affected the element distribution within the nanostructure. It was supported that the most suitable composition for the In:O:C composition ratio was 1In:1GO.

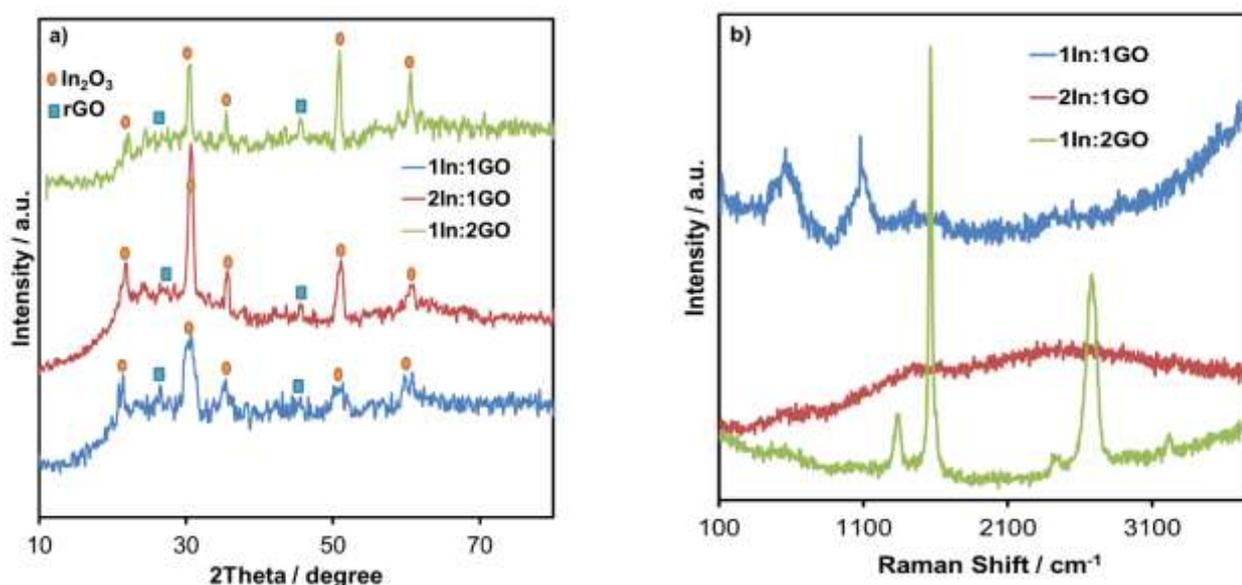


Fig. 1. XRD (a) and Raman (b) spectrum of In₂O₃-rGO nanocomposite

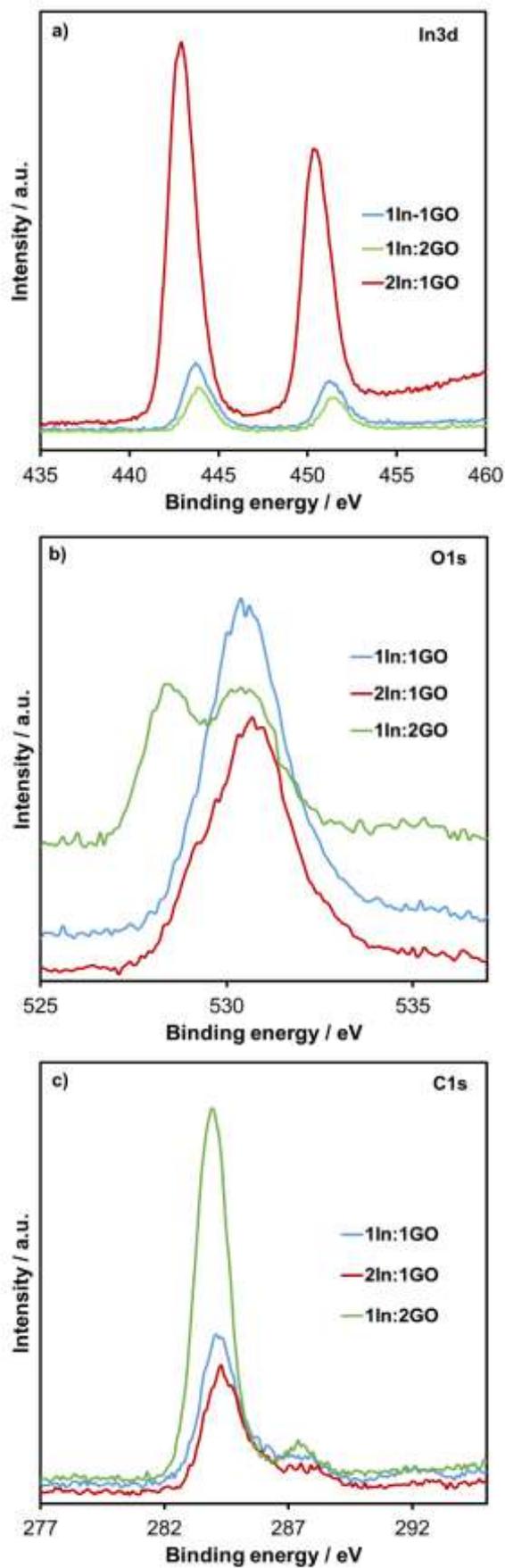


Fig. 2. High-resolution XPS of In 3d (a), O 1s (b), C 1s (c) core-levels for In_2O_3 -rGO nanocomposite

Table 1. Changes of the In, O, and C elements in the In₂O₃-rGO nanostructures produced at different concentration ratios

Volume ratio of In:GO	In	O	C
1:2	12.35	26.59	61.06
2:1	36.6	38.27	25.13
1:1	23.25	49.47	27.28

Morphological Analysis

The surface morphology of the prepared In₂O₃-rGO nanocomposites was investigated using FESEM. Figure 3 shows the FESEM images of the nanocomposites produced with varying amounts of GO. In Figure 3a, it is seen that bulk graphene structures are formed on the surface due to the increasing amount of GO for the FESEM image of the 1In:2GO sample at x10000 magnification. In addition, the presence of In₂O₃ structures is also evident in the lower layer of these rGO layers for x40000 magnification. As shown in Figure 3b, spherical In₂O₃ structures distributed uniformly on the surface were obtained due to the increase in the amount of In (for 2In:1GO). This may be due to the faster kinetics of In³⁺ ions accumulating on the electrode surface during electrochemical deposition compared to rGO. In the FESEM image given in Figure 3c, it is clearly seen that the In₂O₃-rGO nanocomposite for the 1In:1GO ratio contains both nanoparticles and graphene sheets with suitable morphology.

EDX spectra recorded for all samples (Figure 3) show that In, O, and C elements are present in the chemical composition of In₂O₃-rGO nanostructures without any impurity elements. Moreover, the elemental compositions showed variations that confirmed the ratios in the composite. When the amount of GO increased, the percentage of C increased; when the amount of In increased, the percentage of In increased. FESEM images and EDS analysis are in good agreement with previous results (Table 2). All results show that In₂O₃-rGO nanostructures were successfully prepared using an electrochemical technique.

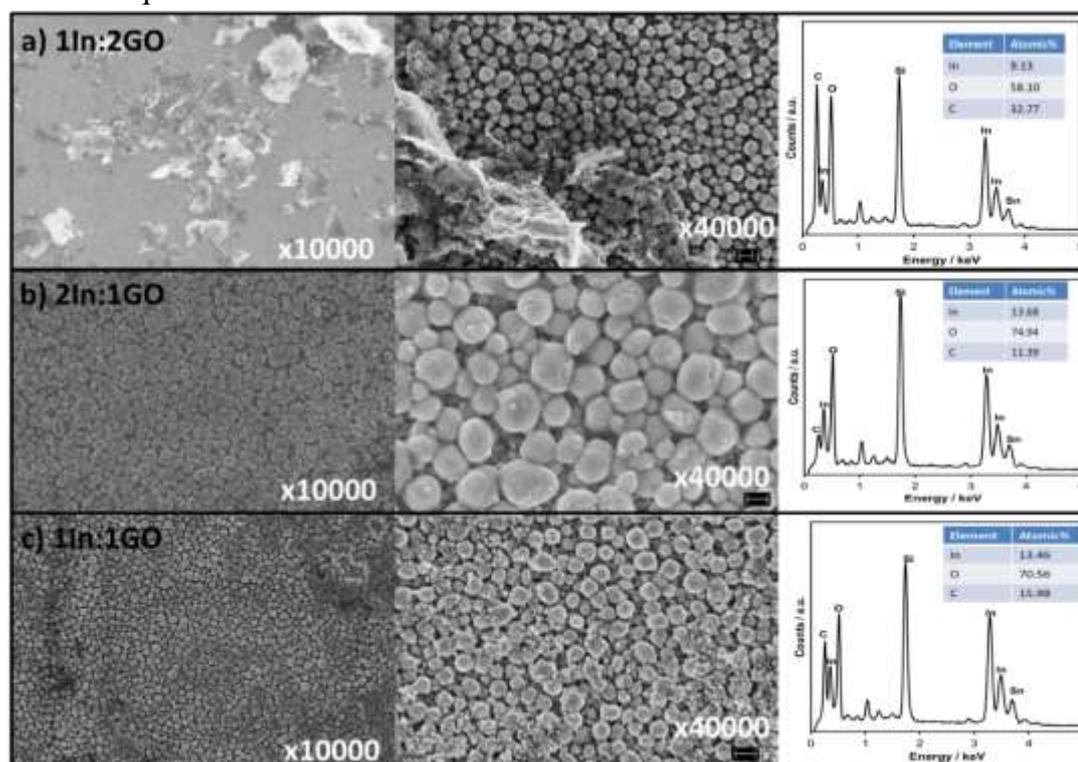


Fig. 3. FESEM images and EDS spectra of In₂O₃-rGO nanocomposite for volume ratios of 1In:2GO (a), 2In:1GO (b), 1In:1GO (c)

Table 2. Morphology of In₂O₃ nanostructures depending on the production technique

Material	Technique	Morphology	Reference
In ₂ O ₃	Chemical vapor deposition	Nanowire	(Tuzluca et al., 2018)
N-doped rGO-In ₂ O ₃	Hydrothermal	Nanocube	(Shanmugasundaram et al., 2017)
In ₂ O ₃ -functionalized rGO	Chemical	Nanorods	(Guo et al., 2023)
In ₂ O ₃	Ultrasonic-assisted synthesis	Nanoparticle	(El-Khouly et al., 2020)
In ₂ O ₃ /RGO	Microwave-assisted hydrothermal synthesis	Nanoparticle	(Alaizeri et al., 2023)
In ₂ O ₃ -rGO	Electrodeposition	Nanoparticle	This study

CONCLUSION

This study presents the effect of changing the amount of GO on the composition and morphology of the nanocomposite for electrochemical production of In₂O₃-rGO nanocomposites. For this purpose, In:GO concentration ratios were tested as 1:1, 1:2, and 2:1. It was determined that the optimum nanocomposite composition and morphology could be produced at the 1In:1GO ratio. XRD, Raman and XPS characterizations supported the fact that the composite composition was composed of In₂O₃ and rGO. As a result, it was seen that In₂O₃-rGO nanocomposites with the best composition could be synthesized using an electrochemical technique when the In:GO concentration ratio was 1:1.

Conflict of Interest

The article authors declare that there is no conflict of interest between them.

Author's Contributions

The authors declare that they have contributed equally to the article.

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