

## Review: Synthesis of rGO in Its Use as Thin Films with Various Polymer Matrices

Rahma Alya Hidayah<sup>1</sup>, Nenni Mona Aruan<sup>1</sup>, Nur Aini Fauziyah<sup>1</sup>, Reffany Choiru Riskiarna<sup>1</sup>, Alfriana Margareta<sup>2</sup>

<sup>1</sup> Physics Study Program, Universitas Pembangunan Nasional "Veteran" Jawa Timur, Surabaya, Indonesia, 60294

<sup>2</sup> Biotechnology Study Program, Institut Teknologi Del, Toba, Indonesia, 22381

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#### Corresponding Author:

Name of Corresponding Author,  
Email:  
[23037010013@upnjatim.ac.id](mailto:23037010013@upnjatim.ac.id)

### ABSTRACT

Reduced graphene oxide (rGO) is a two-dimensional carbon material that has been widely developed as a functional filler in polymer-based thin films due to its high surface area, good electrical conductivity, and adequate interface compatibility. This article presents a literature review on the synthesis of rGO and its use as thin films with various polymer matrices, namely polyaniline (PANi), poly(vinylidene fluoride) (PVDF), poly(vinylidene fluoride-trifluoroethylene) [P(VDF-TrFE)], and chitosan. The review was conducted on open access scientific articles discussing rGO synthesis methods, thin film fabrication techniques, and structural, morphological, thermal, and electrical characterization of composite materials. The results of the study show that rGO can improve the functional performance of polymer thin films through the formation of conductive networks, strengthening of interface interactions, and crystal nucleation effects. In the PANi matrix, rGO increases electrical conductivity and stability, while in PVDF and P(VDF-TrFE), rGO promotes the formation of electroactive crystalline phases that enhance piezoelectric properties. Meanwhile, in chitosan, strong interfacial interactions improve electrochemical response and the potential for environmentally friendly sensor applications.

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## 1. INTRODUCTION

The development of advanced materials research in Indonesia shows a significant increase, particularly in carbon-based materials such as graphene oxide (GO) and reduced graphene oxide (rGO). The rGO material has attracted much attention from researchers because it has good electrical conductivity, a large surface area, and relatively high chemical stability. In addition, rGO materials can also be synthesized using conventional laboratory equipment available at most universities in Indonesia. The ability of rGO to be produced from local carbon sources makes it a strategic material in supporting national research independence. Therefore, rGO materials have become one of the leading materials that can be applied with various additives, such as polymer matrices.

rGO material can be produced from GO synthesis using various methods and adjusted to suit the GO material. In general, rGO synthesis is carried out using a Hummers-based chemical

approach and its modifications. The Hummers method is chosen because it is capable of producing graphene oxide with a high oxidation level and can be reduced to rGO with controlled characteristics. In producing rGO material synthesis, it can also be produced from coconut shell charcoal. The process will confirm the success of the reduction process and the formation of the characteristic structure of rGO using FTIR, UV-Vis, and XRD characterization. This research has been conducted by (Hidayat, Setiadji, and Hadisantoso 2019) successfully synthesized rGO from coconut shell charcoal. This result proves that local biomass raw materials have great potential for the development of functional carbon materials in Indonesia. After rGO was successfully synthesized, many studies focused on its use in the form of polymer matrix films. The polymer matrix plays an important role in providing mechanical flexibility, structural stability, and ease in forming a homogeneous film. The matrix material must have high adhesion to the fibers so that the bonds formed are strong enough for the fibers to be effectively bound within the composite structure. With these strong bonds, the matrix functions optimally in transferring and distributing the load received by the composite to the fiber reinforcement elements (Mardiyati 2018). In thin film application systems, polymers function as a dispersing medium for rGO so that carbon sheets do not easily agglomerate. The combination of rGO and polymers enables the transfer of rGO's electrical and functional properties into a stable macroscopic system. The application of rGO with polymers used for thin films is a growing topic in research.

Thin films based on rGO can be formed by adding various types of polymers to achieve the desired end result. The types of polymers used are conductive polymers or natural polymers. Conductive polymers are polymers that are easily degradable and can conduct electricity (Maulana, Syahbanu, and Harlia 2017). Meanwhile, natural polymers are polymers that originate from nature, such as plants, animals, and microorganisms. In general, known natural polymers include amyllum, cellulose, chitin, and chitosan (Dewi, Widhiantara, and Sandhika 2023). Based on research conducted by (Thu et al. 2018) Forming films from rGO with polyaniline. The integration of rGO into the polyaniline matrix can improve the conductivity and signal stability of the sensor. The presence of rGO forms a more effective electron percolation pathway compared to pure polyaniline. Thin rGO-polyaniline films show a more stable electrochemical response to environmental changes (Thu et al. 2018).

In addition to conductive polymers, rGO can also be applied in ferroelectric and piezoelectric polymer matrices. Piezoelectric materials are a type of material that can convert mechanical energy into electrical energy or vice versa, while ferroelectric materials are a special type of material that has spontaneous electrical polarization. In rGO and P(VDF-TrFE) (Poly(Vinylidene Fluoride)) composite materials with very low rGO concentrations, it can increase  $\beta$  phase formation, crystallinity, and dipole orientation in thin films. These structural improvements directly impact the increase in piezoelectric performance and electrical output of piezoelectric nanogenerator devices. It can be said that rGO can function as an effective nucleation agent in a semi-crystalline polymer matrix (Yaseen and Park 2023). A similar approach was also applied to the PVDF system, showing that rGO synthesized through rapid thermal reduction was able to improve the crystallinity and thermal resistance of PVDF without the use of a compatibilizer. The interface interaction between the rGO sheets and the PVDF polymer chains acts as a nucleation center that promotes the formation of a more stable crystalline phase, although at high concentrations, agglomeration phenomena begin to appear, which reduces the performance of the material (Tienne et al. 2022).

On the other hand, the development of natural polymer-based rGO thin films is becoming an increasingly relevant approach in the context of sustainable materials. In research conducted by (Sembiring, Nainggolan, and Andriyani 2023) shows that chitosan and rGO composite films have a higher electrochemical response than pure chitosan. The interaction between the active groups of chitosan ( $-NH_2$  and  $-OH$ ) and the rGO surface results in a more efficient electron transfer pathway, so that the composite film has the potential to be used as an environmentally friendly thin film-based sensor material. However, if the rGO concentration is too high, it will cause agglomeration and reduce the film's performance. Thus, it can be seen that natural polymers can form a stable interface with rGO through hydrogen bonds and electrostatic interactions (Compton and Nguyen 2010).

In the formation of rGO composites with polymer matrices, it is necessary to use characterization tools to observe the structure formed in the polymer and to observe the presence of defects or polymer flaws. The characterization tools used can be adjusted to the needs of the application of rGO composites with different types of polymers. In general, these composites use FTIR, XRD, UV-Vis, FE-SEM, and Cyclic Voltammetry (CV) characterization. Overall, the performance of rGO thin films with polymer matrices is influenced by the rGO synthesis method, the type of polymer matrix, and the film fabrication technique. Each type of polymer provides different characteristics in terms of the mechanical, electrical, and stability properties of the film. Therefore, this literature review was compiled to summarize and analyze the development of research related to rGO-polymer thin films.

## **2. METHOD**

### **2.1 Types of Research Methods**

This study uses a literature review as the main approach to thoroughly examine the development of research related to the synthesis of reduced graphene oxide (rGO) and its use in thin films based on various polymer matrices. This method was chosen because it is able to integrate the results of previous studies scattered across various scientific publications into a single, comprehensive, and systematic framework of understanding. Through a literature review, the relationship between rGO synthesis methods, polymer matrix types, and material functional properties can be analyzed comprehensively. This approach also allows for the identification of research trends, similarities in findings, and differences in results between studies. Thus, a literature review serves not only as a summary of previous research, but also as a conceptual basis for more in-depth scientific analysis (Snyder 2019).

### **2.2 Sources and Literature Database**

The research data sources were obtained from peer-reviewed scientific articles available in open access through scientific databases such as Google Scholar and the Directory of Open Access Journals (DOAJ). These databases were selected based on their broad coverage of scientific disciplines and ease of access to reputable scientific publications. The articles collected were from national and international journals relevant to the fields of functional materials and materials physics. The use of open access sources ensured that all literature was fully accessible so that the evaluation process could be carried out thoroughly. This strategy was in line with methodological recommendations for conducting transparent and reproducible literature reviews (Xiao and Watson 2019).

### 2.3 Data Analysis and Synthesis

Each selected article was analyzed qualitatively by examining the rGO synthesis method, polymer matrix type, thin film fabrication technique, and material characterization results. The information obtained was then classified based on main themes, such as interface interactions, crystal structure, and functional properties of the film. A comparison process between studies was carried out to identify common patterns and differences in research results. Furthermore, these findings were synthesized narratively to build an understanding of the structure-property relationship of materials. This approach is in line with the practice of narrative synthesis in scientific literature reviews (Snyder 2019).

## 3. RESULTS AND DISCUSSION

Reduced graphene oxide or rGO is a material synthesized from GO material with great potential for application in several fields. In the synthesis process, there are several types of synthesis that can be used to produce rGO from GO material. The synthesis process includes the Hummers method, microwave irradiation, ultrasonics, and conventional reduction methods, but these require a long time and involve the use of hazardous materials (Aryani and Mu'awanah 2023). based on research that has been conducted (Hidayat et al. 2019) It is known that graphene is a two-dimensional material made of  $sp^2$  carbon. Graphene has excellent electronic, mechanical, and atomic structural properties, making it suitable for use in various strategic technological applications such as batteries, sensors, polymer fillers, and energy conversion devices. However, the availability of rGO is still limited, so a synthesis method is needed. The Hummer method is one of the most popular techniques for producing graphene oxide (GO), which can then be reduced to rGO. However, the use of  $NaNO_3$  in the original method can produce harmful  $NO_2$  and  $N_2O_4$  gases, so a safer method must be modified with coconut shells selected as an alternative carbon source due to their abundant availability and high graphitic composition. In addition, this study aims to assess the feasibility of coconut shell charcoal as a cheap and easily obtainable alternative carbon source for rGO production. Three main characterization techniques were used to determine the success of the rGO composite using the Hummers method with coconut shell charcoal: FTIR (Fourier Transform Infrared Spectroscopy), UV-Vis (Ultraviolet-Visible Spectrophotometry), and XRD (X-Ray Diffraction). FTIR is used to detect changes in functional groups by identifying the loss of hydroxyl groups and a decrease in the intensity of other oxygen groups, which indicates the reduction process of GO to rGO. UV-Vis spectrophotometry is used to measure the absorption of  $\pi \rightarrow \pi^*$  electron transitions.

In the study conducted, it was found that rGO was successfully synthesized from coconut shell charcoal using the modified Hummer method. Figure 1. shows the results of characterization testing using FTIR. The FTIR spectrum shows a peak at a wavelength of  $1604\text{ cm}^{-1}$ , indicating the presence of aromatic C=C bonds as the main indicator of rGO formation and the disappearance of the OH group peak at  $3400\text{ cm}^{-1}$ , indicating that most of the oxygen groups have been reduced. The FTIR spectrum shows absorption at a wavelength of 272 nm, which is characteristic of rGO due to transition to  $\pi \rightarrow \pi^*$  transition. The XRD results show a diffraction peak at an angle of  $2\theta$  around  $224^\circ$ , indicating the characteristic graphitic structure of rGO. Although there are still indications of incomplete reduction due to the presence of residual C-O and C=O groups, overall the results show that rGO has been successfully formed.

Table 1. Thin Film from rGO-Polymer Matrix Composite

No	Polimer Matrix	Synthesis Method and Fabrication	Characteritation	Results
1.	Chitosan (CS)	rGO is mixed with chitosan using the simple coating method.	FTIR, XRD, CV	The CS-rGO interaction is physical, as indicated by the shift in the FTIR band and amorphous XRD pattern; the addition of rGO increases the conductivity and electrochemical sensitivity, making the film effective as a sensor material (Sembiring et al. 2023).
2.	Poliananilin (PANI)	Method In-Situ Polymerization	FTIR, SEM, Raman, LAMP	rGO acts as an additional conduction pathway to significantly increase electrical conductivity due to the formation of an rGO-PANI percolation network (Thu et al. 2018)
3.	P (VDF-TrFE)	Langmuir Schaefer (LS)	XRD, FTIR, SEM, PFM, Output listrik	The addition of rGO acts as a nucleating agent that increases the $\beta$ phase fraction of P(VDF-TrFE), improves dipole orientation, and enhances the piezoelectric response and output power of the nanogenerator (Yaseen and Park 2023)
4.	PVDF	Modified Hummers + Solution Casting	XRD, FTIR, DSC, TGA, SEM	rGO acts as a heterogeneous nucleating agent that increases the crystallinity of PVDF and promotes the formation of the $\beta$ phase, thereby significantly improving the thermal and structural properties of PVDF (Tienne et al. 2022).

rGO material can be applied in various applications by adding various types of polymer matrices according to the needs of the application to be used. The polymer matrices that can be used are PVDF, P(VDF-TrFE), polyaniline (PANI), and chitosan. Each type of polymer matrix has its own properties and advantages and can be adjusted to suit the needs. In the Poly(vinylidene fluoride) or PVDF matrix, to improve thermal properties, crystallinity, and morphological performance without using compatibilizers and changes in the interaction between the rGO and PVDF structures in various concentrations from 0.1-5%, as well as to determine the strengthening or weakening of composite properties due to agglomeration or interface saturation (Tienne et al. 2022). In the P(VDF-TrFE) matrix, the Langmuir-Schaefer (LS) technique can increase  $\beta$  phase formation, improve crystallinity, and improve dielectric properties (Yaseen and Park 2023). In the matrix, polyaniline is a conductive polymer with several advantages, namely it is easily synthesized, can be synthesized in large quantities using relatively simple methods, and is reversible and stable for thousands of cycles (HIDAYAT and WAHYU ALAMSYAH, IMAN RAHAYU 2016). In this material, chitosan acts as the main matrix of the film because it is biodegradable, easy to process, and rich in active groups such as  $-NH_2$  and  $-OH$  (Sembiring et al. 2023).

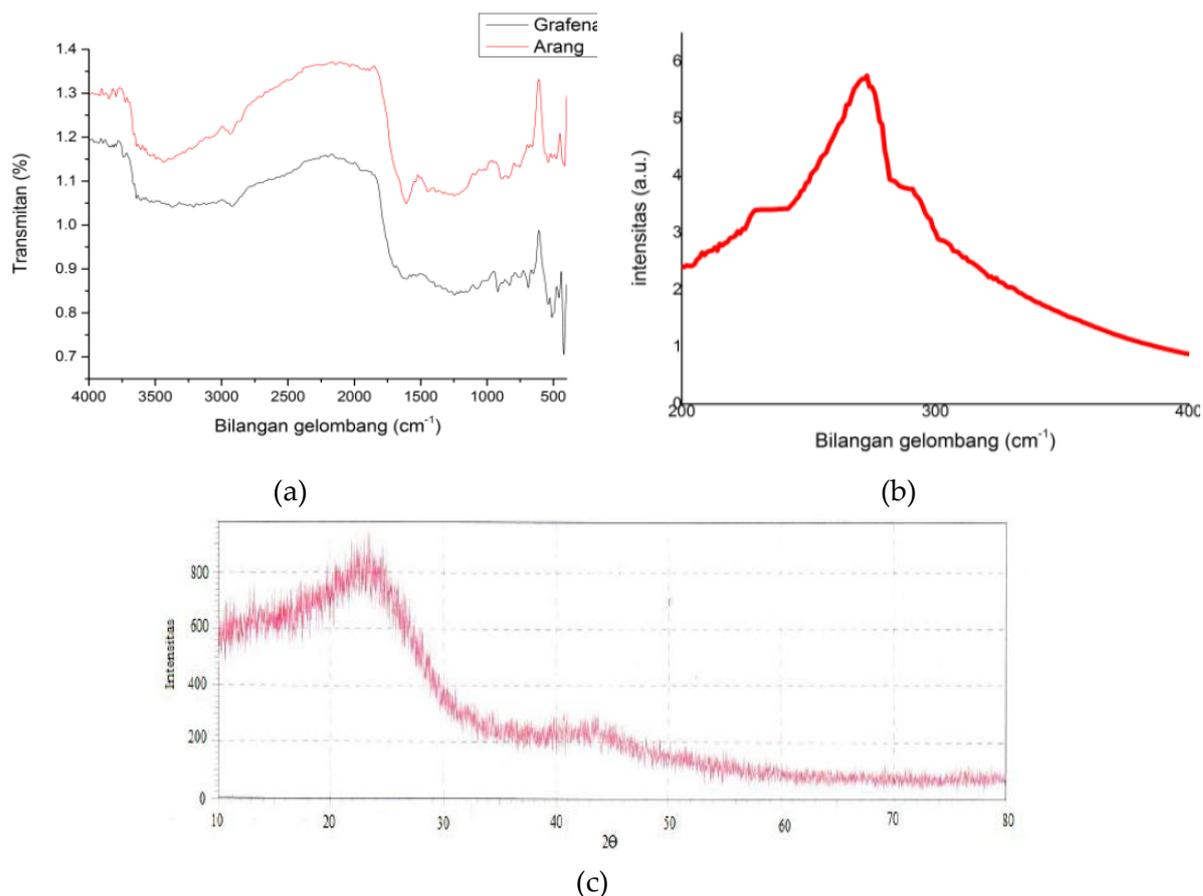


Figure 1. (a) FTIR spectrum of graphene oxide (rGO) from coconut shell charcoal, (b) UV-Vis spectrum of graphene oxide (rGO) from coconut shell, (c) Diffractogram of rGO from coconut shell charcoal

(Hidayat et al. 2019)

In the formation of thin films with rGO and chitosan composites that have been carried out by (Sembiring et al. 2023) shows a significant improvement in functional properties compared to pure chitosan, especially in terms of conductivity and electrochemical response of the material. Chitosan, as a natural polymer matrix, has a polysaccharide chain structure with amine (-NH<sub>2</sub>) and hydroxyl (-OH) groups that play an important role in forming interface interactions with the rGO surface. The presence of oxygen groups in rGO allows the formation of hydrogen bonds and strong electrostatic interactions with chitosan, thereby increasing the compatibility and dispersion of rGO in the polymer matrix. Based on the experiments conducted, it was found that the CS/rGO composite film had formed with a chemical structure that showed interaction between chitosan and rGO, as seen in Figure 2, which is an FTIR spectrum showing a shift in the intensity of the characteristic peak of chitosan and the appearance of a C=C peak. This indicated the presence and integration of rGO in the matrix. XRD showed changes in diffraction peaks and d-spacing, indicating that the crystalline structure of the film contributed to changes in mechanical and electrochemical properties. Then, in Figure 3, testing of the electrode CV coated with a film with a rGO concentration of 250 ppm produced the highest redox current and the most stable response with a very strong linearity value. This indicates that a concentration of 250 ppm is the most effective optimal condition for improving the conductivity and sensitivity of the sensor to ions in PBS. Meanwhile, higher rGO concentrations did not provide a significant improvement because of the possibility of agglomeration, which reduces its effectiveness as a conductive nanofiller.

From an application perspective, the increased conductivity and electrochemical response of thin films from rGO-chitosan include materials with potential for thin-film-based sensors. The strong interface interaction between rGO and chitosan enhances the sensor's stability against environmental variations, while its biocompatible and biodegradable properties provide added value for environmentally friendly and biomedical sensor applications.

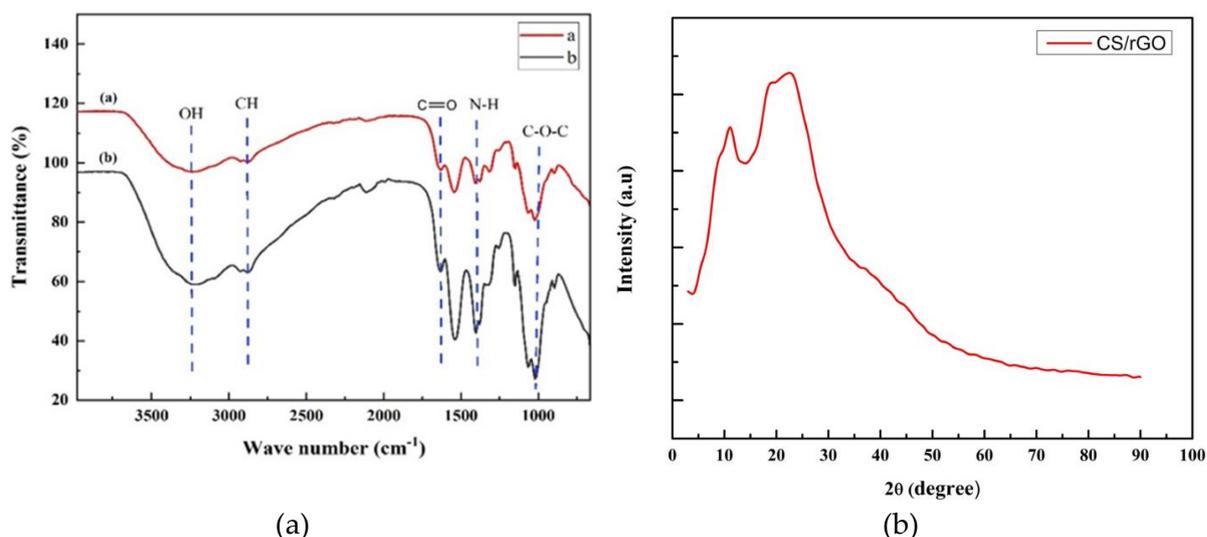


Figure 2. (a) Spektrum FTIR Cs dan Cs/rGO, (b) Diaktogama Cs/rGO

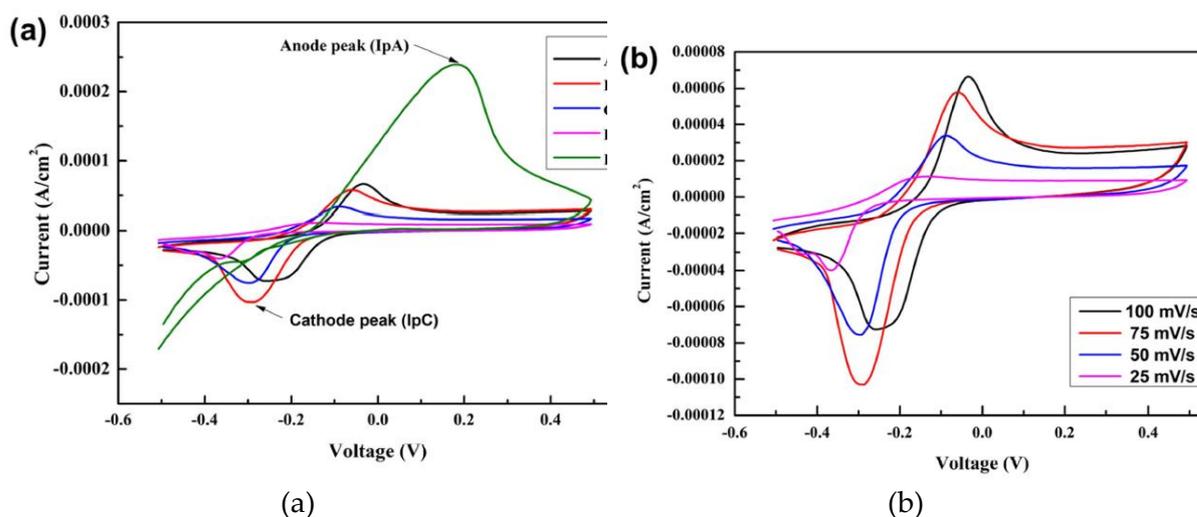


Figure 3. Voltamogram, (a) Cs/rGO with variation in rGO at a scan rate of 75 mV/s and, (b) Variation in scan rate of Cs/rGO modified electrode with rGO concentration of 250 ppm

(Sembiring et al. 2023)

Based on experiments conducted by (Thu et al. 2018), Thin films of rGO-polyaniline composites show consistent performance improvements compared to pure PANi, particularly in terms of electrical properties and functional stability of the material. Polyaniline is known as a conductive polymer with a conjugated bond system that allows charge movement, but its performance is often limited by unstable conductivity and fragile mechanical properties. The addition of rGO contributes significantly by forming a two-dimensional conductive network that shortens the electron transport path. The interaction between the amine groups on PANi

and the oxygen groups on rGO results in a strong interfacial bond, even though it does not involve covalent bonds. The interaction between the amine groups on PANi and the residual oxygen groups on rGO results in a fairly strong interfacial bond that does not involve the formation of covalent bonds. In Figure 4 the SEM image reinforces the findings by showing that the surface of the rGO-PANi film is denser and more uniform than pure PANi. This allows for the formation of interconnected percolation, which is important in improving electrical conductivity and current stability. In Figure 5, FTIR testing shows a shift in the characteristic C-N and C=C bands, indicating the presence of physical interactions that affect the chemical environment of the PANi chain. Figure 6 shows the results of electrical and electrochemical testing, indicating that the addition of rGO significantly reduces resistance. However, if the rGO content is too high, agglomeration tends to occur, affecting the homogeneity of the structure. In addition, rGO is a material that acts as a reinforcement that strengthens the structural integrity of PANi, which is naturally brittle. The combination of PANi's conductivity and rGO's structural stability results in a thinner film that is more durable and resistant to degradation.

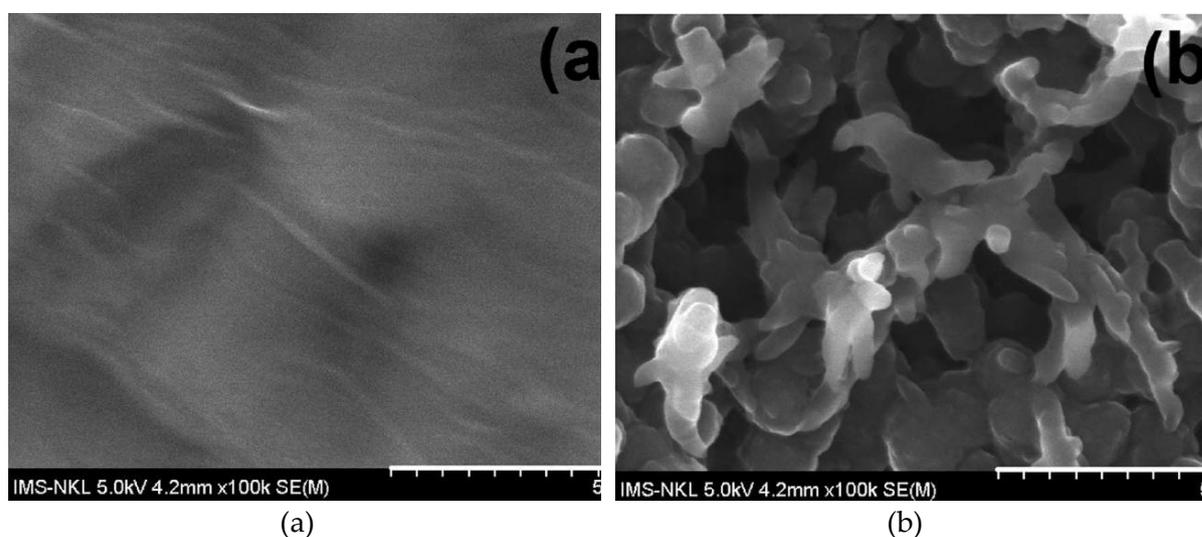


Figure 4. Results SEM, (a) rGO-PANi, and (b) Films

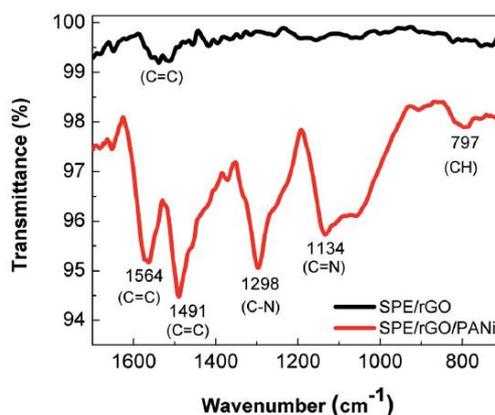


Figure 5. Results ATR-FTIR from rGO (Black) and rGO-PANi (Red)

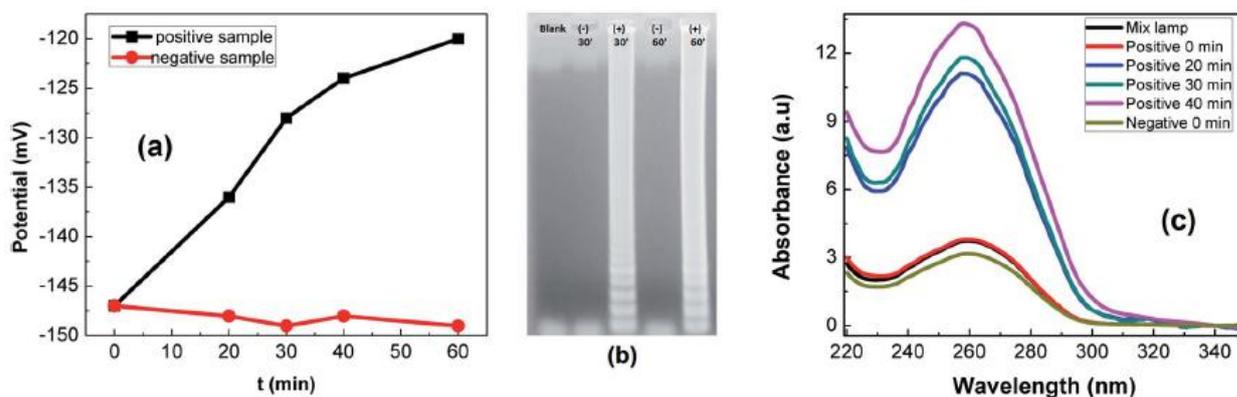


Figure 6. LAMP reaction: OCP detection, (a) Gel electrophoresis, (b) Absorbance measurement, (c) LAMP product

(Thu et al. 2018)

The formation of thin films using piezoelectric polymers, namely P(VDF-TrFE), is useful in significantly improving the piezoelectric properties of thin films, which are directly related to the increase in the  $\beta$  phase fraction and the orientation of the polymer chain dipoles. In experiments conducted by (Yaseen and Park 2023), rGO material acts as a crystal nucleation center that accelerates the formation of the piezoelectric active phase without requiring complex external poling treatment. In addition, the Langmuir-Schaefer technique allows control of film thickness and homogeneity, so that the rGO effect can be maximized structurally. Figure 7 shows that at low concentrations, rGO is well dispersed and does not cause mechanical degradation. Meanwhile, at high concentrations, agglomeration begins to occur. Meanwhile, at high concentrations, agglomeration tends to occur. Composites with rGO-P(VDF-TrFE) material are very promising for energy harvesting applications and flexible piezoelectric devices. The study found that adding rGO at low concentrations to P(VDF-TrFE) films significantly improved the structural, dielectric, and piezoelectric properties of nanogenerator devices, with the optimal composition being 0.002 wt% rGO. Additionally, it shows an increase in the  $\beta$  phase to nearly 98%, higher crystallinity and regular dipole orientation, which can be observed in Figure 8. This resulted in an increase in the piezoelectric modulus  $d_{33}$  and dielectric constant. Electrically, this nanogenerator device can generate a VOC of 88V higher than samples without rGO addition, producing an ISC and maximum power of  $16.5 \mu W/cm^2$  at an optimum load of around  $20 M\Omega$ . However, increasing the rGO concentration above the optimal value causes nanofiller aggregation, a decrease in film regularity, and weakening of the dipole orientation path, which will then reduce piezoelectric performance.

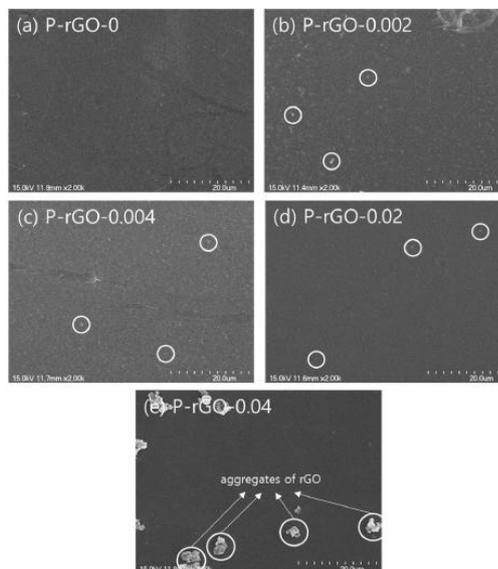


Figure 7. Results SEM form (a) P-rGO-0, (b) P-rGO-0.002, (c) P-rGO-0.004, (d) P-rGO-0.02, and (e) PrGO-0.04 in  $\pi = 5 \text{ mN/m}$ .

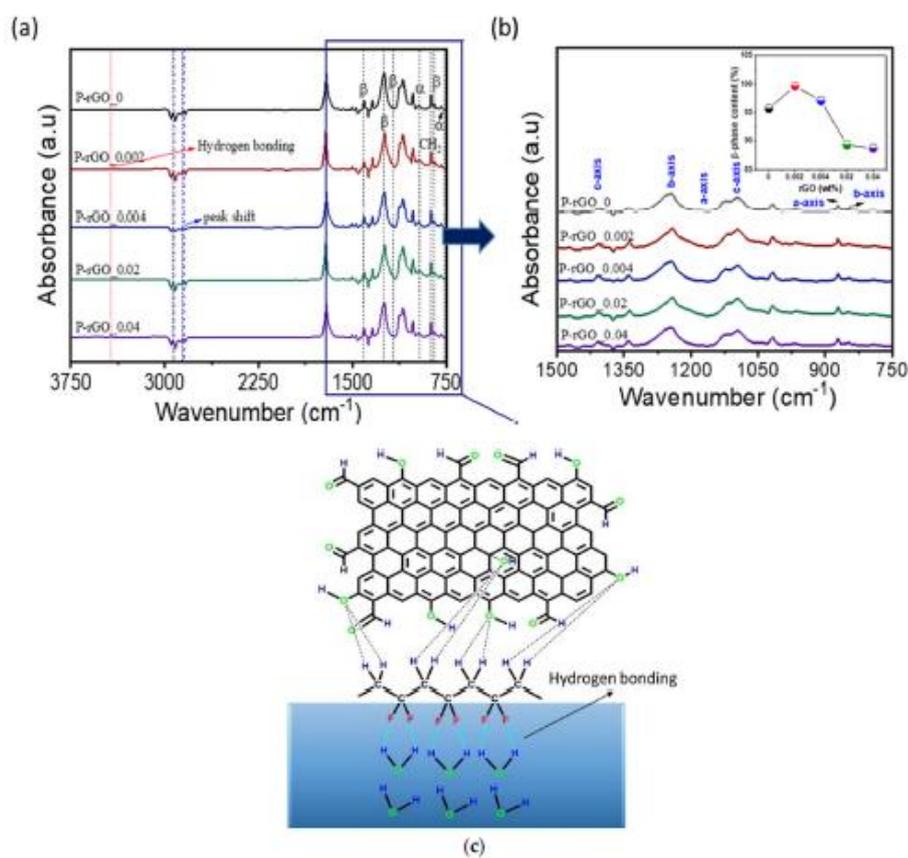


Figure 8. FTIR spectra of five thin films, (b) Peak details in the 750-1500 cm range in the FTIR spectrum and analysis of  $\beta$  phase content, (c) Illustration of hydrogen bonds between water molecules, polymer matrix, and rGO particles

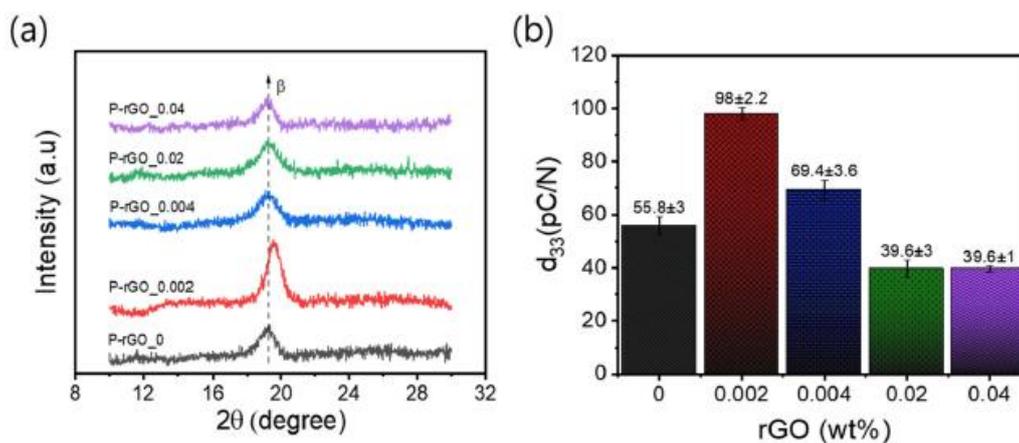


Figure 9. (a) XRD patterns for five thin films, (b) plot of piezoelectric modulus as a function of rGO content

(Yaseen and Park 2023)

In piezoelectric polymer matrix types, not only P(VDF-TrFE) is used as the polymer matrix in the formation of composite films from rGO and polymer matrix. There are other types of piezoelectric polymer matrices, such as PVDF (Poly(Vinylidene Fluoride)). Based on experiments conducted by (Tienne et al. 2022) It is known that the addition of rGO to the PVDF matrix significantly improves the crystallinity and thermal stability of PVDF thin films. The rGO material acts as a heterogeneous nucleation center that promotes the formation of the  $\beta$  phase, which is the phase that contributes most to the piezoelectric and dielectric properties of PVDF. The interface interaction between the oxygen groups on rGO and the PVDF chains increases structural regularity without the need for additional compatibilizers. In addition, the increase in thermal degradation temperature indicates that rGO is capable of slowing heat diffusion and strengthening the polymer network. This composite can be applied to functional thin films, particularly flexible energy devices and sensors. The study found that the addition of rGO to the PVDF matrix had a significant effect on the crystal structure, electrical properties, and thermal stability of thin films. PVDF, as a semi-crystalline polymer, has several crystalline phases, of which the  $\beta$  phase is the most desirable because it directly contributes to the piezoelectric and dielectric properties of the material. Figure 9 shows that the presence of rGO acts as a heterogeneous nucleation agent that promotes the transformation of the  $\alpha$  phase to the  $\beta$  phase, as indicated by an increase in the intensity of the characteristic peak of the  $\beta$  phase. This finding is reinforced by FTIR analysis in Figure 10, which shows an increase in the characteristic absorption band of the  $\beta$  phase, indicating a more regular dipole orientation in the PVDF matrix. From a morphological perspective, SEM images show that rGO–PVDF films have a more homogeneous and dense surface compared to pure PVDF, especially at low to medium rGO concentrations. The relatively uniform dispersion of rGO helps limit irregular crystal growth and improves the microstructural uniformity of the film. This increase in structural regularity has a direct impact on electrical properties, with test results showing an increase in dielectric constant and a decrease in dielectric loss in rGO–PVDF films. In addition, rGO forms limited conductive pathways that enhance electrical response without eliminating the basic insulating properties of PVDF.

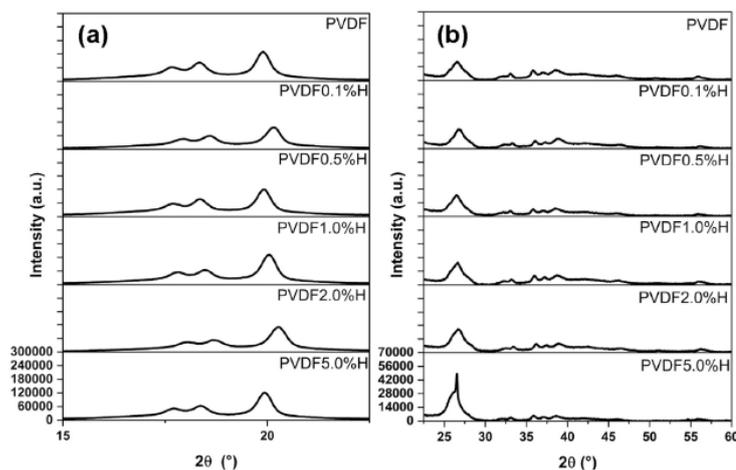


Figure 10. PVDF/rGO nanocomposite diffractogram results: (a) from  $15.0^{\circ}$  to  $22.5^{\circ}$  and (b) from  $22.5^{\circ}$  to  $60.0^{\circ}$

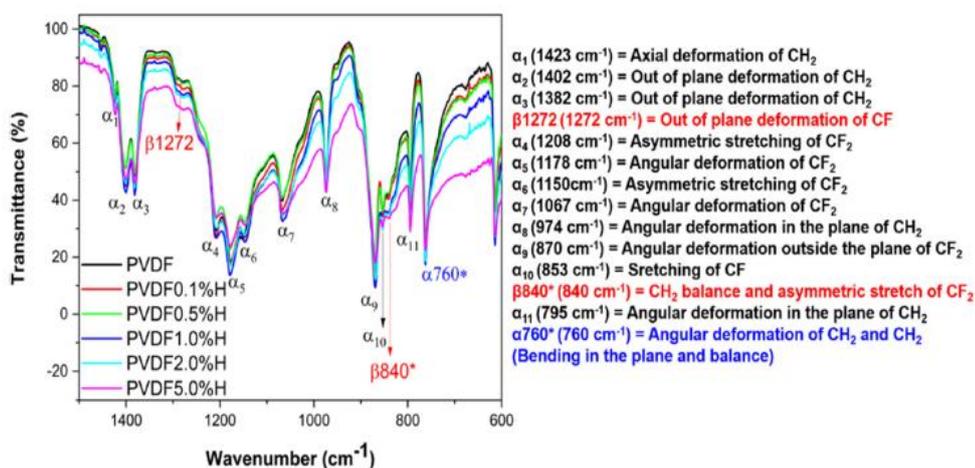


Figure 11. FTIR spectrum of extruded material with identification of the main crystalline phase

(Tienne et al. 2022)

#### 4. CONCLUSION

Based on the research that has been conducted, reduced graphene oxide (rGO) has been proven to be effective as a functional filler in polymer matrix-based thin films. The integration of rGO can improve the electrical, structural, and thermal properties of materials through the mechanisms of percolation network formation and interface strengthening. In the rGO–polyaniline system, rGO improves electrical conductivity and stability, while in rGO–PVDF and rGO–P(VDF-TrFE), rGO acts as a nucleating agent that promotes the formation of active crystalline phases and improves piezoelectric performance. Meanwhile, in rGO–chitosan composites, strong interfacial interactions result in improved electrochemical response relevant to sensor applications. Overall, the literature indicates that optimization of the synthesis method, rGO dispersion, and polymer matrix selection are the most important factors in the development of high-performance rGO–polymer matrix thin films.

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