

A Novel Synthesis Route and Functional Group Analysis by FTIR Spectroscopy of Graphene Oxide from Indigenous Coal and Graphite

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Abstract

This study develops a novel synthesis route for producing Graphene Oxide (GO) using local Jambi coal and commercial graphite as carbon precursors through the Modified Hummers method. The objective of this research is to evaluate the success of the oxidation process and identify the oxygen-containing functional groups formed in the synthesized GO using Fourier Transform Infrared (FTIR) Spectroscopy. Three sample variations were prepared: coal-based GO (GOC), graphite-based GO (GOG), and coal-graphite mixture (GOGC). The FTIR results reveal the presence of characteristic GO functional groups, including –OH, C=C, C–O, C–OH, and C–O–C. The highest oxidation level was observed in the graphite-based sample, while the mixed sample exhibited a lower oxidation degree. These findings confirm that local coal can serve as a viable alternative carbon source for GO synthesis, offering added value to local resources and supporting the development of carbon-based advanced materials.

Keywords: Graphene oxide; Nanomaterial.

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INTRODUCTION

Coal is a carbon rich sedimentary resource formed from ancient organic materials that has undergone geological transformation over millions of years (Solikah and Bramastia, 2024). Indonesia is recognized as the third largest coal producer in the world according to the International Energy Agency (2023), with major deposits located in Sumatra and Kalimantan. Geological variations across regions result in differences in coal properties that affect its type, grade, and maturity level (Hakim, 2023). Based on calorific value, 62% of Indonesian coal is classified as medium quality, 27% as low quality, 9% as high quality and only 2% as very high quality [1]. Jambi province has around 19 million tons of medium calorific coal, which serves as a strategic resource for the development of advanced carbon based materials (Suryani et al., 2024). However, the use of coal as a fuel causes environmental degradation, including land damage and water pollution, especially when mining practices are less sustainable.

The demand for the utilization of coal that adds value through approaches to convert coal into Graphene Oxide (GO), a carbon based nanomaterial produced through strong chemical oxidation [4]. GO has a large surface area, reactive oxygen functional groups and easily modifiable chemical properties, making it suitable for applications in adsorption, catalysis, sensors, energy storage and composite materials. Previous research has shown that alternative carbon sources such as rice husk carbon [11], graphite for fuel cell precursors (Hanifah et al., 2021), and carbonized biomass [12] can be used in GO synthesis. These findings emphasize the importance of developing synthesis routes based on abundant local materials to reduce dependence on commercial graphite.

Graphene is a two dimensional material composed of carbon atoms arranged in a hexagonal lattice that has various physical phenomena. The stack of Graphene forms graphite [3]. GO is a carbon based nanomaterial obtained from the oxidation of graphite or other carbon sources using strong oxidants [4]. GO has a large surface area, high chemical reactivity and wide applications in the fields of energy, environment and materials. GO from various sources has also been used as an adsorbent for heavy metals, a precursor material for fuel cells and other functional carbon materials ([11], Hanifah et al., 2021). Carbonized coal can also be used as an alternative carbon source in graphene synthesis (Purwandari et al., 2024).

The variation of carbon sources affects the design of synthesis methods and drying processes to obtain stable GO [5]. Drying is an important step to maintain structure and prevent particle agglomeration. Oven drying remains the main choice because it is simple and efficient, and it can influence the oxygen-carbon ratio, interlayer distance, as well as the stability of GO morphology before characterization is conducted (Tene et al., 2021). Thus, selecting the appropriate drying method is a crucial factor in ensuring the final quality of graphene oxide produced from various carbon sources, including coal [14].

This study presents an innovative coal-based graphene oxide synthesis route to achieve optimal material quality [6]. Coal and commercial graphite is used as a substitute for conventional graphite via the Modified Hummers method. FTIR characterization is employed to analyze functional groups. This approach can overcome the limitations of conventional graphite and enhance the added value of local coal as a high-tech carbon material. Additionally, the development of this synthesis route is expected to produce GO with more consistent oxidation levels and a more stable structure compared to previous methods (Zhang et al., 2022).

METHODS

This research involves nanomaterial from Jambi coal and commercial graphite powder processed into Graphene Oxide. The study has 3 variations in the use of raw materials, namely 100% coal, 100% commercial graphite powder, and a mixture of coal and graphite powder.

Research Design

This study employs an experimental laboratory research design with a quantitative approach. The main focus of the research is the synthesis of graphene oxide (GO) derived from local coal and commercial graphite using the Modified Hummers method. The research includes a series of processes such as oxidation, purification, drying, and characterization using Fourier Transform Infrared Spectroscopy (FTIR). All experimental procedures were conducted in laboratory facilities equipped for wet chemical synthesis and advanced material characterization.

Research Target/Subject

The research subjects consist of graphene oxide synthesized from two types of carbon precursors: local coal and commercial graphite. The coal was initially dried, ground, and sieved to obtain a homogeneous fine powder, whereas graphite was used directly without pre-treatment. Sample selection was carried out using a purposive sampling technique based on the suitability of both materials as carbon sources for graphene oxide synthesis. The chemical reagents used in the oxidation and purification processes include KMnO_4 , H_2SO_4 , H_2O_2 , distilled water, and HCl .

Research Procedure

The research procedure begins with the preparation of the samples, where the coal is dried and ground into a fine powder before being used as a precursor. The synthesis of graphene oxide follows the Modified Hummers method, starting with the dispersion of coal or graphite powder into concentrated H_2SO_4 under cooling conditions. KMnO_4 is added gradually while maintaining a controlled temperature to ensure a stable oxidation process. After the oxidation reaches the desired stage, distilled water is added slowly to expand the reaction volume, followed by the addition of H_2O_2 to stop the reaction and reduce manganese residues. The resulting mixture is then purified through filtration and repeated washing with distilled water and dilute HCl until the pH approaches neutral. The purified graphene oxide is dried using an oven to produce a stable dried powder, which is then ground to achieve uniform particle size. The final stage of the procedure involves material characterization using FTIR to identify functional groups of the GO samples.

Instruments, and Data Collection Techniques

Data were collected using Fourier Transform Infrared Spectroscopy (FTIR) instruments. FTIR data were obtained within a range of $400\text{--}4000\text{ cm}^{-1}$ to identify functional groups formed.

Data analysis technique

The collected data were analyzed based on the characteristic features of graphene oxide. FTIR spectra were examined to identify functional groups such as O-H , C=C , C-O , epoxy, and carboxyl, which indicate successful oxidation. The results characterization methods were compared to assess the effect of carbon precursor type on the quality of the synthesized graphene oxide.

RESULTS AND DISCUSSION

The FTIR spectrum of the GOG 100% sample exhibits several characteristic absorption peaks of graphene oxide. A broad peak in the region of 3151 cm^{-1} indicates the stretching vibration of the -OH group, which originates from hydroxyl functionalities and adsorbed moisture on the material surface, consistent with findings [7]. This peak is one of the main indicators of GO formation resulting from oxidative disruption of the graphitic structure. A peak around 1596 cm^{-1} corresponds to the C=C stretching vibration of the aromatic carbon framework, suggesting that some sp^2 domains remain preserved after oxidation (Bete et al., 2019).

The absorption within 1356 cm^{-1} is associated with C-OH vibrations, indicating the presence of hydroxyl groups attached to the GO surface [11]. At lower wavenumbers, approximately 1215 cm^{-1} , the C-O stretching vibration appears, arising from epoxy or alkoxy groups, while the peak around 1014 cm^{-1} corresponds to C-O-C vibrations typical of strong

oxidation in graphene oxide, as described by Díaz et al. (2020). Overall, these peaks confirm that the GOG 100% sample underwent extensive oxidation, producing a substantial amount of oxygen-containing functional groups [6] stating that GO synthesized via modified Hummers oxidation typically displays strong -OH and C-O-C signature [6].

The FTIR spectrum of the GOB 100% sample shows several main peaks that confirm the presence of oxygen-containing groups characteristic of graphene oxide. The broad peak around 3336 cm^{-1} corresponds to the stretching vibration of the -OH group, generally arising from hydroxyl groups and adsorbed water on the GO surface [4]. The peak at 1580 cm^{-1} indicates aromatic C=C vibrations, suggesting that some sp^2 graphite domains remain intact after the oxidation process [6]. Furthermore, absorption at 1187 cm^{-1} indicates the presence of epoxy C-O groups, while the peak at 1032 cm^{-1} indicates the presence of C-O-C groups, both of which are characteristic of the oxidized GO structure [5,13]. The combination of these peaks confirms that the GOB variation 100% sample has successfully undergone oxidation while still retaining part of its graphite carbon structure.

The FTIR spectrum of the GOGB 50% sample shows a similar pattern but with noticeable differences that indicate a lower oxidation degree than GOG 100%. The broad absorption near 2676 cm^{-1} signifies the presence of -OH groups but with lower intensity, pointing to reduced hydroxyl formation, consistent with observations [14].

Table 1. Group content in graphene oxide of graphite

No.	Graphene Oxide (Graphite 100%)	Graohene Oxide (Coal 100%)	Graohene Oxide (Graphite and Coal 50%)	Functional Groups	Wavenumber (cm^{-1})	References
1.	3151	3336	2676	-OH (Hydroxyl Group)	2500 - 3300	[15]
2.	1595	1580	1556	C=C (Carbon Double Bond)	1500-2200	[16]
3.	1356	-	-	C-OH (Alcohol Group on Carbon Framework)	1350 - 1360	[9]
4.	1215	1187	1137	C-O (Single Carbon-Oxygen)	1050 - 1260	[9]
5.	1014	1032	-	C-O-C (Epoxy Group)	1022 - 1300	[17]
				C-O-C (Epoxy Group)	1014	[10]

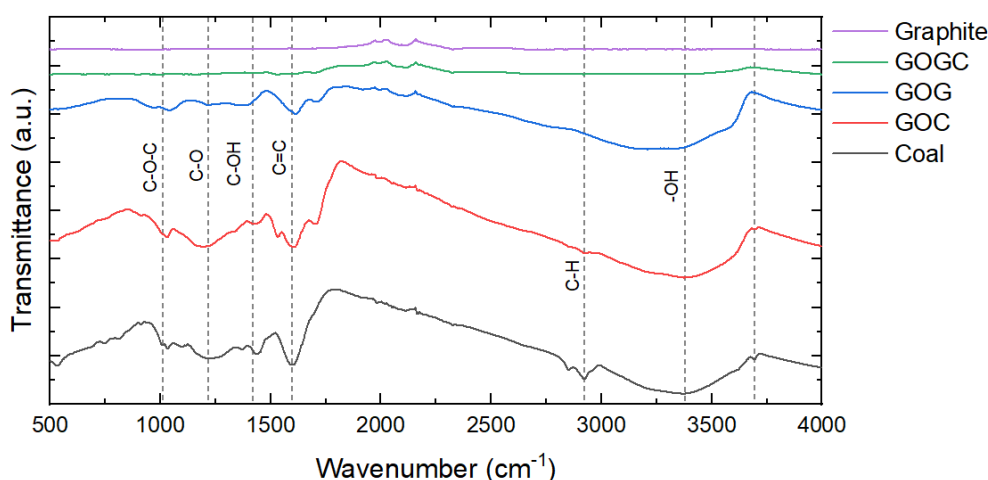


Figure 1. FTIR spectrum of Coal, Graphite, and Graphene Oxide. Three sample graphene oxide was synthesized based on Coal, Graphite and the mixture from of Coal and Graphite.

A peak near 1556 cm^{-1} represents the C=C stretching vibration, indicating that the aromatic carbon domains are still largely retained. GO produced under milder oxidation conditions tends to exhibit stronger C=C peaks due to the preservation of sp^2 -bonded regions

[8]. The band around 1215 cm^{-1} corresponds to C–O stretching from epoxy or alkoxy groups, although its intensity is noticeably lower than in the GOG 100% sample [13]. The dominance of bands in this range indicates that the C–O bond plays a major role in the chemical stability of GO, as these groups become the reactive centers that allow GO to interact with water and other polar molecules. The C–O band in the $1022\text{--}1300\text{ cm}^{-1}$ area confirms that the graphene sheets have undergone effective oxidation and exhibit the characteristic features of Graphene Oxide as reported in reference studies. The absence of a clear C–O–C peak further indicates a reduced extent of oxygen-bridge formation, consistent with findings by Fathy et al. (2024). These observations suggest that the GOGC variation 50% sample undergoes a lower degree of oxidation, likely due to different precursor composition or reduced oxidizing conditions. As stated by Purwandari et al. (2020), GO samples with lower oxidation levels typically display weaker C–O and –OH peaks, which aligns with the characteristics observed in this spectrum.

The appearance of a peak at 2921 cm^{-1} in the FTIR spectrum of the coal sample clearly confirms that the material still contains saturated aliphatic C–H groups, which are the main structural components in low to medium rank coal. This band is a characteristic functional group of coal and supports the interpretation that the organic phase in the sample is still dominated by complex hydrocarbon structures. The absorption band at 3694 cm^{-1} is the main characteristic of the O–H stretching vibration on the surface of kaolinite minerals, which are one of the important inorganic components in coal. This signal usually appears together with companion bands at 3651 and 3620 cm^{-1} , confirming the presence of structural hydroxyl groups within the kaolinite aluminosilicate layers, where kaolinite shows a strong peak at 3694 cm^{-1} as a distinctive feature of surface O–H groups. Another study examining the interaction of coal with clay minerals also showed a similar pattern, namely the presence of kaolinite characterized by a strong O–H band in the range of $3600\text{--}3700\text{ cm}^{-1}$, including a peak at 3694 cm^{-1} , thus confirming that this signal originates from the inorganic impurity phase in the coal, not from its carbon organic structure.

CONCLUSIONS

FTIR analysis confirmed the presence of key oxygen-containing functional groups—including –OH, C=C, C–O, C–OH, and C–O–C—across all synthesized GO samples, indicating that the oxidation process occurred successfully in each variation. The graphite-based GO exhibited the highest oxidation level, while the 50% coal–graphite mixture showed a comparatively lower degree of oxidation, which aligns with the structural differences inherent in each carbon precursor. Those results demonstrated clear and consistent formation of oxygen functional groups, reinforcing the potential of local coal as a viable and promising alternative carbon precursor for GO synthesis.

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AUTHOR CONTRIBUTIONS

In this study, L. Z. Maulana played a role in initiating the research concept framework that has been used. V. A. Nanda was responsible for synthesis and analyzing the data, while R. M. Anggraini carried out the validation process. M. F. Afrianto conducted data analysis, and F. Deswardani carried out investigative activities. F. B. Pujaningsih provided various required research resources, while Alrizal, W. B. Alfajri, and F. A. Faruqi curated the data. L. Z. Maulana drafted and wrote the initial manuscript, and V. A. Nanda assisted in the review, rewriting, and editing stages.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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DECLARATION OF ARTIFICIAL INTELLIGENCE USE

We hereby confirm that no artificial intelligence (AI) tools or methodologies were utilized at any stage of this study, including during data collection, analysis, visualization or manuscript preparation. All work presented in this study was conducted manually by the authors without the assistance of AI-based tools or systems.

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