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# The Effect of Sonication Duration and Concentration of Ascorbic Acid on the Yield of Reduce Graphene Oxide (rGO) from Isolation Results Bituminous Coal in East Kalimantan

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***Abstract:*** *The primary objective of this research is to determine the yield of reduced graphene oxide (rGO) derived from graphene oxide (GO) through the application of ascorbic acid (AA) employing a mechanochemical approach (sonication+AA). The graphene oxide was extracted from bituminous coal in East Kalimantan through sonication treatment lasting 3 hours at a pH of 4. Subsequently, it underwent conversion to rGO by the addition of AA (10%, 20%, and 30%) and varying sonication durations of 10 minutes, 30 minutes, and 50 minutes. The optimal concentration of AA and sonication time were selected based on achieving the lowest yield of rGO. The optimal value was achieved at an AA concentration of 10% with a sonication time of 30 minutes. Subsequently, the reduced graphene oxide (rGO) was subjected to characterization through Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy with Energy Dispersive X-ray Analysis (SEM-EDX). The transmittance percentages of the C-OH, C=O, and C-O-C peaks exhibited a significant increase. EDX characterization revealed a 49.4% rise in the carbon content, escalating from 47.9% in graphene oxide (GO) to 71.6% in rGO, and a concurrent 55.79% decrease in the oxygen content, declining from 32.92% in GO to 21.13% in rGO. The C/O ratio rose up from 1.45 (GO) to 3.38 (rGO). It has a density value of 1.45 g/cm3 and pH value of 3-4.*

***Keywords:****Ascorbic Acid, Bituminous, Graphene Oxide, Reduced Graphene Oxide,Sonication.*

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## I. INTRODUCTION

Indonesia is classified as a nation possessing substantial supplies and deposits, accounting for approximately 3.25% of the global total coal reserves. In 2022, Indonesia's coal production reached 687 million tonnes, catering to both domestic and international demands (Setiawan et al., 2023). The classification of coal from the lowest to the highest is based on the formation process, encompassing young lignite coal, sub-bituminous coal, bituminous coal, and anthracite coal (Yuliyani et al., 2021). Bituminous coal is composed of layered graphite and inorganic compounds such as $SiO_2$, $Al_2O_3$, $CaSO_4$, $Al_2Si_2O_5(OH)_4$, $FeCO_3$, $Fe_2O_3$, $FeS_2$ and other metal oxides (Garcia et al., 2020). The main advantage of bituminous coal lies in its high carbon content with low moisture content and volatility (Huda et al., 2017).

Bituminous coal has the capability to undergo thermal and chemical processes to transform into graphene from graphene oxide. Attaining pure graphene directly from graphene oxide poses challenges due to the persistence of residual functional groups. Consequently, the resulting product is termed reduced graphene oxide (Amri, 2019). Reduced graphene oxide has high conductivity, large surface area, and it has been proven that composites using reduced graphene oxide can modify the way electrodes work to be more optimal (Li et al., 2020). According to research conducted by Manoj & Ponni in 2013, regarding changes in organic functional groups using organic acids namely citric acid and acetic acid and treated with the addition of Ethylenediaminetetraacetic acid (EDTA). The results of Fourier Transform Infra-Red (FT-IR) characterization show that the use of organic acids can remove oxides in the coal structure (Manoj & Narayanan, 2013).The process of generating reduced graphene oxide from graphene oxide, employing ascorbic acid as a reducing agent aided by sonication, is anticipated to yield graphene using environmentally friendly chemicals. Ascorbic acid is acidic and a strong reductant. The properties of ascorbic acid tend to change due to oxidation, although it remains stable when in pure crystalline form (Cahyadi, 2018).

The synthesis of reduced graphene oxide can be accomplished through the mechanochemical method of sonication. Sonication is a technique employed to alter the size of a material using ultrasonic waves. The impact of sonication leads to a progressively significant reduction in molecular weight as the duration of

ultrasonic waves increases (Anugraini et al., 2018). Based on previous research conducted by Mutrofin et al. in 2021 on the preparation of coal nanoparticles from washing with the sonication technique, it is known that sonication technique is able to reduce particle size, to facilitate the release of hydrogen bonds (Mutrofin et al., 2021). Mutrofin et al succeeded in obtaining graphene oxide from graphite by adjusting pH 4 with the addition of $HNO_3$ and HCl using the sonication technique.

Based on these problems, this research applies the principles of green chemistry to obtain the yield of reduced graphene oxide from graphene oxide isolated from bituminous coal using ascorbic acid with a mechanochemical method in the form of sonication, to obtain optimized ascorbic acid concentration and sonication time for the isolation of reduced graphene oxide, and to obtain information on the characterization of reduced graphene oxide by density test, filtrate pH value, Fourier Transform Infra Red (FT-IR), solubility test, and Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX).

## II. EXPERIMENTAL PROCEDURE

### 2.1 Tools and Materials

The necessary equipment includes beaker glass, measuring cup, volumetric flask, watch glass, 500 mL Erlenmeyer flask, funnel, stirring rod, dropper pipette, measuring pipette, 10 mL volumetric pipette, spatula, porcelain cup, 150 and 200 mesh sieve, porcelain grinder, digital balance, oven, Hawach 11-micron filter paper, desiccator, plastic wrap, aluminum foil, magnetic stirrer, hot plate, ultrasonicator cleaner (Delta D68H), pycnometer, pH indicator, test tube, FTIR spectrophotometer (Varian FTS 1000), and SEM-EDX (Thermo Scientific Varian Phenom Prox). The required materials are bituminous coal from PT. Anugerah Bara East Kalimantan, ascorbic acid with concentrations of 10%, 20%, 30%, NaOH p.a 0.1 M solution, HCl p.a 0.1 M solution, n-hexane, toluene, and aquadest.

### 2.2 Coal Sample Preparation

Bituminous coal samples obtained from PT. Anugerah Bara East Kalimantan, were ground and mashed using a porcelain grinder. Then the crushed coal was sieved using 150 mesh and 200 mesh sieves. As much as 150 g sample of 200 mesh coal was dried in an oven at 40℃ for 10 hours and placed in a desiccator. Then, the sample was washed using aquadest (1:5) and stirred using a magnetic stirrer with a speed scale of 6-7 for 3 hours. Then, the sample was filtered using 11-micronHawach filter paper until the filtrate was clear of impurities (clear) and transferred into a porcelain cup. Then, the sample was dried in an oven at 40℃ for 10 hours and allowed to stand in a desiccator.

### 2.3 Isolation of Graphene Oxide

The coal sample, post-preparation, was weighed at 5 g and placed into a 500 mL erlenmeyer flask. Subsequently, 250 mL of distilled water was added. The initial pH of the coal sample was assessed using pH indicator paper. 0.1 M HCl was incrementally added until a pH of 4 was achieved. Three hours of sonication was carried out with an interval of 30 minutes every 1 hour. Then filtering was carried out using filter paper. The resulting yield was put into a porcelain cup and dried in an oven at 40℃ for 10 hours.

### 2.4 Synthesis of Reduced Graphene Oxide Variation of Ascorbic Acid Concentration

The yield of graphene oxide, amounting to 1 g, was placed into a 250 mL erlenmeyer flask. Subsequently, 50 mL of distilled water was added. Sonication was then conducted for a duration of 2 hours, with a 30-minute interval every 1 hour. Then, ascorbic acid was added with a concentration variation of 10%; 20%; and 30% as much as 10 mL and continued sonication for 30 minutes. The yield was weighed and the lowest yield was characterized by FTIR.

### 2.5 Synthesis of Reduced Graphene Oxide Variation of Sonication Time Length

The yield of graphene oxide, totaling 1 g, was measured and placed into a 250 mL erlenmeyer flask. Subsequently, 50 mL of distilled water was added. Following this, sonication was conducted for a duration of 2 hours, with a 30-minute interval every 1 hour. Subsequently, ascorbic acid with varying optimum concentrations was added, amounting to 10 mL. The sonication time was then adjusted to 10 minutes, 30 minutes, and 50 minutes, to determine the optimal duration. The results obtained in the form of yield were weighed and characterized by density, pH value, solubility test, FTIR, and SEM-EDX.

## III. RESULTS AND DISCUSSIONS

### 3.1 Synthesis of Reduced Graphene Oxide (rGO)

The synthesis of rGO was duplicated. Data pertaining to the yield of reduced graphene oxide, corresponding to variations in ascorbic acid (AA) concentration, were collected as in **Table 1**.

**Table 1. Yield of Reduced Graphene Oxide Variation of AA Concentration**

| Trial Number | AA Concentration | | |
|---|---|---|---|
| | 10% | 20% | 30% |
| 1 | 92,08% | 101,04% | 106,53% |
| 2 | 92,76% | 102,93% | 107,62% |
| Average | 92,42% | 101,98% | 107,07% |

The yields were 92.42% at a 10% ascorbic acid concentration, 101.98% at a 20% ascorbic acid concentration, and 107.07% at a 30% ascorbic acid concentration. Considering the yield data, the 10% ascorbic acid concentration, which yielded the lowest result, was chosen for further testing using FTIR.The choice of the lowest yield is predicated on the assumption that the reduction of impurities or reduction of oxide groups present in graphene oxide has been effectively achieved using ascorbic acid as a reducing agent. Ascorbic acid possesses numerous hydroxyl groups capable of binding to epoxy groups on graphene oxide.The utilization of ascorbic acid in the synthesis of reduced graphene oxide as a reducing agent demonstrates outstanding reduction capability. This is evident in the enhanced % transmittance observed in the hydroxyl, carboxylate, epoxy, and inorganic mineral groups of the FTIR spectra, as compared to the FTIR spectra of graphene oxide. Phenolic compounds in ascorbic acid can perform well in providing electrons, therefore more $Fe^{3+}$ is reduced (Sjahriza&Herlambang, 2021).

The selected ascorbic acid concentration is used tosynthesize of reduced graphene oxides by varying the sonication time, namely 10 minutes; 30 minutes; and 50 minutes. The yield data of reduced graphene oxide based on the variation of sonication time is obtained in **Table 2**.

**Table 2.Yield of Reduced Graphene Oxide Variation of Sonication Time**

| | Sonication Time | | |
|---|---|---|---|
| | 10 minutes | 20 minutes | 30 minutes |
| 1 | 95,78% | 92,08% | 93,79% |
| 2 | 95,71% | 92,76% | 95,14% |
| Average | 95,745% | 92,42% | 94,465% |

The yields were 95.745% at 10 minutes of sonication time, 92.42% at 30 minutes of sonication time, and 94.465% at 50 minutes of sonication time. Based on the yield data, the lowest yield was selected at a sonication time of 30 minutes, as reduced graphene oxide with optimal conditions.The sonication technique offers the advantage of low energy consumption and yields efficient results (Syamsuri et al., 2017). A factor that can influence the particle size produced is the sonication process time (Dwiastuti et al., 2016). The sonication technique aims to reduce particle size and facilitate the release of hydrogen bonds into $H_2O$ and carbocations (Mutrofin et al., 2021).

### 3.2 Density Test Using a Pycnometer

Density values are frequently employed to characterize the type of material, in accordance with the Archimedes principle that is applicable to liquid fluids (Alim et al., 2017). The working principle in determining density involves the comparison between the mass of an empty pycnometer that does not contain air at a certain temperature and volume and the mass of the pycnometer containing the sample at the same temperature and volume (Muhriansyah et al., 2016). The density value is calculated using the following formula.

$$\frac{W2 - W1}{(W4 - W1) - (W3 - W2)}$$

(Montgomery, 2014)

Description:

W1 = Weight of empty pycnometer
W2 = Weight of pycnometer containing dry sample
W3 = Weight of pycnometer containing sample and water
W4 = Weight of pycnometer filled with water

Density measurements were carried out on graphene oxide and reduced graphene oxide samples under optimal conditions, namely 10% ascorbic acid concentration with a sonication time of 30 minutes. The density

value of graphene oxide was found to be 1.09 g/cm$^3$, these results are close to the theoretical graphene oxide density value of 0.981 g/cm$^3$. And obtained a reduced graphene oxide density value of 1.45 g/cm$^3$, the density value is close to the theoretical reduced graphene oxide density value of 1.5 to 1.9 g/cm$^3$ (Torrisi et al., 2022). The data is tabulated in **Table 3.**

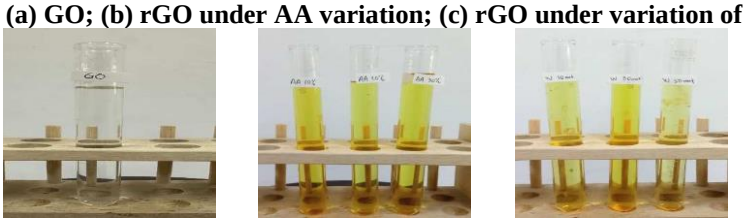
**Table 3.Density Values of GO and rGO**

| | Density value of test sample | Theoretical density value (Torrisi et al., 2022) |
|---|---|---|
| Graphene Oxide (GO) | 1.09 g/cm$^3$ | 0.981 g/cm$^3$ |
| Reduced Graphene Oxide(rGO) | 1.45 g/cm$^3$ | 1.5-1.9 g/cm$^3$ |

### 3.3 Test pH Value of Filtrate

The pH indicator is a parameter utilized to evaluate the acidity of a solution by measuring its pH value. Solutions with a pH value below 7 are considered acidic, while those with a pH value above 7 are considered alkaline. A solution with a pH value of 7 is classified as neutral (Wibowo, 2020). This research measures the pH value of the filtrate to determine the acidity level to form graphene oxide (GO) is known pH 4 and reduced graphene oxide (rGO)is known pH 4-3. A comparison of the appearance of the filtrate between graphene oxide and reduced graphene oxide was conducted and it is displayed in **Figure 1**.

**Figure 1.Filtrate (a) GO; (b) rGO under AA variation; (c) rGO under variation of sonication time**



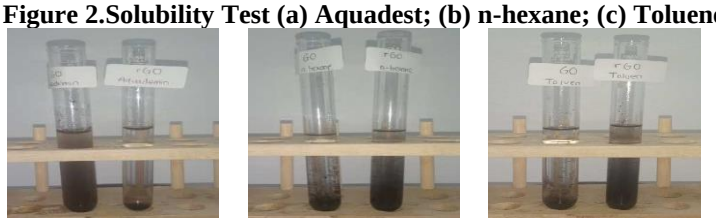
(a) (b) (c)

It is evident that the filtrate from graphene oxide is highly clear. This substantiates the successful reduction of impurities or metal oxide groups present in the coal after washing, achieved through sonication techniques. These techniques facilitate the release of hydrogen bonds into $H_2O$ and carbocations. The reduced graphene oxide filtrate looks yellow in color, proving that ascorbic acid successfully reduces the impurities or oxide groups contained in the graphene oxide. Ascorbic acid can be oxidized and form oxidation compounds, such as dehydroascorbate which gives the solution a yellow color (Zempleni et al., 2013).

### 3.4 Solubility Test Using Protic and Aprotic Solvents

Solubility tests are carried out using protic solvents or aqueous solvents in the form of aquadest and aprotic solvents or organic solvents in the form of n-hexane and toluene. Protic solvents hasOH ions, so they easily interact with polar functional groups. Aprotic solvents cannot donate OH ions, so they are non-polar which can dissolve non-polar compounds (Noviyanty et al., 2019). Solubility tests were conducted on graphene oxide and reduced graphene oxide at optimal conditions and the performance can be seen in **Figure 2.**

**Figure 2.Solubility Test (a) Aquadest; (b) n-hexane; (c) Toluene**



(a) (b) (c)

In solubility tests using aquadestor protic solvents, it is observed that graphene oxide exhibits partial solubility, whereas reduced graphene oxide is insoluble. The hydrophilic nature and solubility of graphene oxide in polar solvents stem from the presence of carbonyl and carboxyl groups. Reduced graphene oxide is insoluble in protic solvents due to the reduction of OH groups and has a tendency to be non-polar.According to solubility tests employing aprotic solvents, it is noted that graphene oxide is insoluble, while reduced graphene oxide displays partial solubility. The challenges in dissolving graphene oxide in organic solvents arise from strong hydrogen bonds that lead to adherence and robust interaction among neighboring layers(Zankana et al., 2023). Meanwhile, reduced graphene oxide can partially dissolve proving the reduction of hydroxyl groups, so that it can dissolve in aprotic solvents. The subjective observation is in **Table 4**.

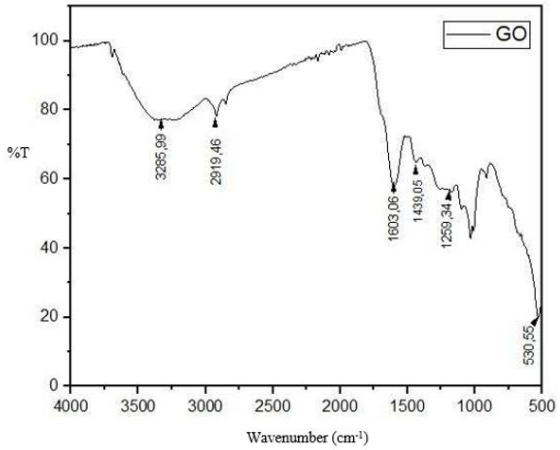
**Table 4. Solubility Test of GO and rGO**

| Solvent Type | Solubility Test | |
|---|---|---|
| | GO | rGO |
| Akuademin | Partially Soluble | Insoluble |
| n-hexane | Insoluble | Partially soluble |
| Toluen | Insoluble | Partially soluble |

### 3.5 Fourier Transform Infrared Analyisis

Infrared characterization was conducted on reduced graphene oxide and graphene oxide at the optimal concentration of ascorbic acid and the optimal sonication time. It was performed at a wavenumber of 4000-400 $cm^{-1}$ and a resolution of 4 $cm^{-1}$. Based on the IR spectra of the resulting graphene oxide, an absorption band is obtained at a wavenumber of 1603.06 $cm^{-1}$ due to the presence of C = O bonds from carboxylic groups, as in **Figure 3**.

**Figure 3. IR Spectra of Graphene Oxide**



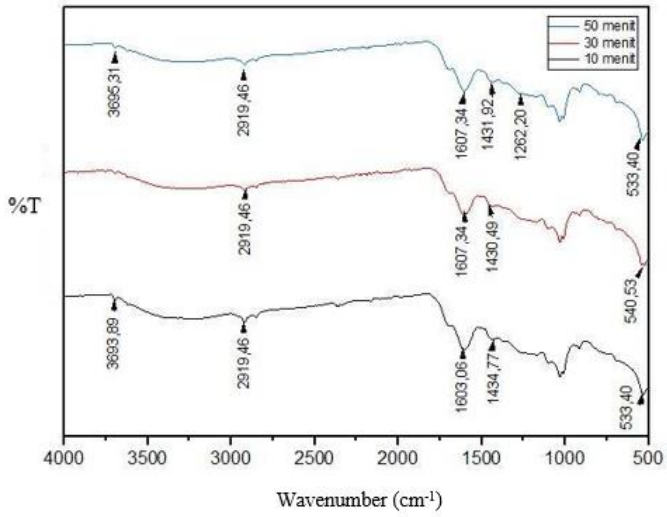

There is an O-H absorption band from the hydroxyl group at a wave number of 3285.99 $cm^{-1}$. The absorption band found at wave number 1259.34 $cm^{-1}$ is the C-O bond of the epoxy group. There is a C=C bond from the aromatic domain at wave number 1439.05 $cm^{-1}$. Absorption was found at wavenumber 530.55 $cm^{-1}$ from inorganic mineral groups. The absorption bands detected indicate the presence of graphene oxide.According to research conducted by Ciplak et al (2015) in the IR spectra of graphene oxide, an O-H absorption band was found at a wavenumber of 3410 $cm^{-1}$, the presence of carboxyl groups in the C = O absorption band with a wave number of 1721 $cm^{-1}$, in the C-O absorption band at a wave number of 1087 $cm^{-1}$, there is an aromatic C = C bond at a wave number of 1404 $cm^{-1}$ , and absorption found at a wavenumber of 430 $cm^{-1}$ from inorganic mineral groups (Ciplak et al., 2015). **Table 5**. displayes the comparison of IR spectra of graphene oxide test results with the literature shows the presence of functional groups in the form of hydroxyl, carboxylic, epoxy groups, aromatic C = C groups and inorganic mineral groups.

**Table 5. Tabulation of Wavenumbers of Graphene Oxide Based on Literature andResults**

| NO | Wavenumbers $(cm)^{-1}$ | | Description |
|---|---|---|---|
| | Literature (Ciplak et al., 2015) | Graphene oxide | |
| 1 | 3410 | 3285,99 | O-H stretch of alcohol |
| 2 | 1721 | 1603,06 | C=O strain |
| 3 | 1404 | 1439,05 | C=C aromatic |
| 4 | 1087 | 1259,34 | C-O-C strain |
| 5 | 430 | 530,55 | Inorganic minerals |

Moreover, the reduced graphene oxide was characterized at the optimal ascorbic acid concentration of 10%, with variations in sonication time set at 10 minutes, 30 minutes, and 50 minutes, and the spectra is in **Figure 4**.

**Figure 4. IR Spectra of Reduced Graphene Oxide with Variation of Sonication Time**




Drawing insights from the IR spectra data of reduced graphene oxide, it is observed that variations in sonication time result in an increase in the % transmittance within the hydroxyl, carboxyl, epoxy, and inorganic mineral groups of graphene oxide. In accordance with the research conducted by Habte et al. (2019), the IR spectrum of reduced graphene oxide displays decreased transmittance in the O-H, C=O, and C-O-C absorption bands. At wavenumber 3400 cm-1, indicates the presence of O-H absorption bands, the presence of carboxyl groups at wave number 1622 $cm^{-1}$, and there is epoxy at wavenumber 1224 $cm^{-1}$ (Habte et al., 2019).Based on the tabulation of the wave number absorption of rGO from the test results and compared with the literature,**Table 6**, there are hydroxyl, carboxyl, and epoxy absorption bands.

**Table 6. Tabulation of Absorption of rGO Based on Literature and Test Results**

| Wavenumber ($cm^{-1}$) of Reduced Graphene Oxide | | | | Description |
|---|---|---|---|---|
| Literature (Habte et al., 2019) | 10 min | 30 min | 50 min | |
| 3400 | 3693,89 | - | 3695,31 | O-H stretch of alcohol |
| 1622 | 1603,06 | 1607,34 | 1607,34 | C=O strain |
| 1384 | 1434,77 | 1430,49 | 1431,92 | C=C aromatic |
| 1224 | 1032,58 | 1032,58 | 1032,58 | C-O-C strain |

Based on the tabulation of % transmittance of graphene oxide and reduced graphene oxide,**Table 7**, it can be seen that at wavenumbers 3700-3300 $cm^{-1}$ which shows the O-H functional group, there is a % transmittance in GO of 77.21%. The synthesis of rGO was carried out by using ascorbic acid at the optimal concentration of 10% and varying the duration of sonication time (10 minutes; 30 minutes; and 50 minutes). At 10 minutes, the % transmittance of rGO was obtained at 94.78%. There was not found O-H absorption in the IR spectra of rGO 30 minutes, proving that the use of ascorbic acid as a reducing agent is able to bind epoxy groups on graphene oxide with the help of sonication time for 30 minutes, resulting in the release of hydrogen bonds releasing $H_2O$ and carbocations. At 50 minutes of rGO, the % transmittance is 94.77%. There is an O-H absorption band at a sonication time of 50 minutes proving the occurrence of a reversible reaction.

**Table 7. Tabulation of % Transmittance of Graphene Oxide and Reduced Graphene Oxide**

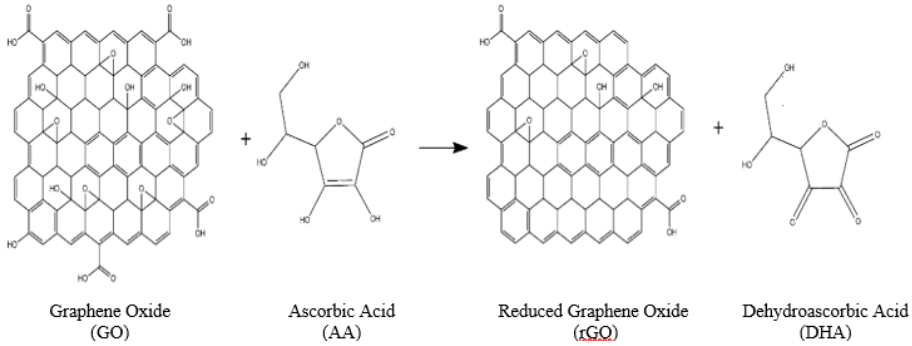
| Wavenumbers ($cm^{-1}$) | % Transmittance | | | |
|---|---|---|---|---|
| | GO | rGO 10 min | rGO 30 min | rGO50 min |
| 3700-3300(O-H) | 77,21 | 94,78 | - | 94,77 |
| 3100-2695(C-H) | 78,26 | 77,68 | 81,84 | 81,69 |
| 1678-1600(C=O, C=C aromatic) | 58,06 | 56,06 | 62,77 | 61,24 |
| 1400(C-C=C) | 65,09 | 63,76 | 69,31 | 68,29 |
| 1275-1020(C-O-C) | 42,78 | 34,68 | 41,21 | 36,80 |

| | | | | |
|---|---|---|---|---|
| 600-400(M-O) | 19,70 | 20,15 | 23,52 | 22,09 |

The aromatic C=C absorption band at wavenumbers 1678-1600 $cm^{-1}$ has a % transmittance on GO of 58.06%. On rGO sonication 30 minutes of 62.77%. The occurrence of an increase in % transmittance from GO to rGO, has the possibility of a reduction in C = O bonds at these wavenumbers, because the aromatic C = C area with C = O overlaps.

The wavenumber of 1400 $cm^{-1}$ shows the C-C conjugate group there is an increase in % transmittance from GO which is 65.09% to rGO 30 minutes which is 69.31%. There was C-O-C absorption band at wave numbers 1275-1020 $cm^{-1}$ there is a % transmittance on GO of 42.78%. In rGO sonication 10 minutes; 30 minutes; and 50 minutes sequentially obtained % transmittance of 34.68%; 41.21%, and 36.80%. The C-O-C group was optimally reduced in the 30minute sonication of rGO. However, at 10 min and 50 min of rGO sonication, there was an addition of C-O-C groups indicating a reversible reaction. At wavenumbers 600-400 $cm^{-1}$, there is an increase in the % transmittance of GO which indicates a decrease in metal oxide bonds to reduced graphene oxide. Based on the FTIR test, it can be determined that the optimally reduced graphene oxide with an ascorbic acid concentration of 10% with a sonication time of 30 minutes.The reaction mechanism of reducing graphene oxide using ascorbic acid is predicted and figured out as in **Figure 5**.

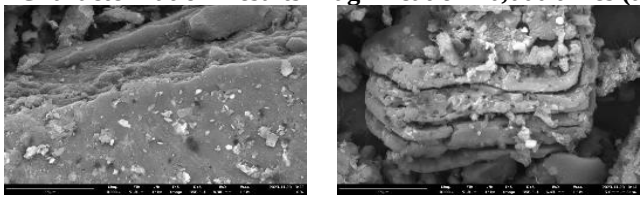
**Figure 5. Reduction Reaction Mechanism of Graphene Oxide Using Ascorbic Acid**




### 3.6 Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX) Analysis

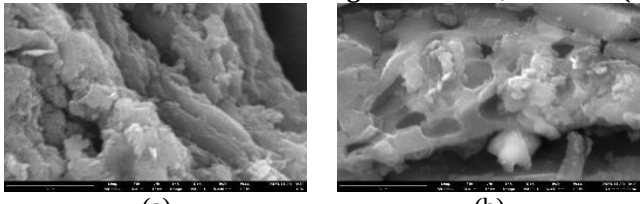
This research conducted SEM-EDX tests on graphene oxide and reduced graphene oxide under optimal conditions, namely 10% ascorbic acid concentration with a sonication time of 30 minutes. SEM-EDX tests were carried out at magnifications of 10,000 times and 50,000 times with 15kv specifications. The image in **Figure 6.** and **Figure 7.** is the result of the morphological analysis of graphene oxide and reduced graphene oxide.

**Figure 6. SEM Characterization Results Magnification 10,000 times (a) GO (b) rGO**



(a) (b)

**Figure 7. SEM Characterization Results Magnification 50,000 times (a) GO (b) rGO**

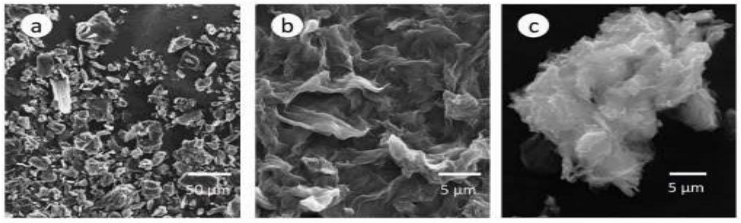


(a) (b)

The results of SEM morphological analysis of graphene oxide and reduced graphene oxide at 10,000 times magnification show that graphene oxide consists of one thick layer, while reduced graphene oxide consists

of layered sheets. The thickness in the morphological structure of graphene oxide is due to the presence of oxygen functional groups bound in its structure. This phenomenon illustrates that during the oxidation process, the graphite layers are exfoliated (Wahyuningsih et al., 2020).At 50,000 times magnification, it shows that graphene oxide is in the form of sharp and irregular flakes, while in reduced graphene oxide more pores resemble a hexagonal shape. The pores formed in reduced graphene oxide are caused by the sonication process, resulting in flaking (Honorisal et al., 2020).

**Figure 8. SEM Characterization Results Based on Literature (a) Graphite; (b) Graphene Oxide; (c) Reduced Graphene Oxide (Wahyuningsih et al., 2020)**




According to research conducted by Wahyuningsih et al (2020), the results of SEM morphological analysis on graphene oxide show layered sheets that give the impression of thickness, and the distance between the layers is clearly visible. The morphology of reduced graphite (rGO) shows thinner sheets when compared to graphene oxide, and it appears that the distance between the layers is smaller than before (Wahyuningsih et al., 2020). The results of the composition analysis on graphene oxide and reduced graphene oxide are as follows.

**Table 8. EDX Components of GO and rGO**

| Graphene Oxide | | | | | | Reduced Graphene Oxide | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Element | %Atom | | | Average | C/O | Element | %Atom | | | Average | C/O |
| | Spot 1 | Spot 2 | Spot 3 | | | | Spot 1 | Spot 2 | Spot 3 | | |
| C | 59,4 | 30,7 | 53,5 | 47,90 | 1,45 | C | 70,5 | 73,4 | 70,9 | 71,62 | 3,38 |
| O | 28,23 | 47,23 | 23,31 | 32,92 | | O | 20,8 | 21,5 | 20,9 | 21,1 | |
| Al | 1,22 | 10,9 | 1,15 | 4,43 | | Al | 0,09 | 0,95 | 0,19 | 0,41 | |
| Si | 0 | 8,48 | 0,61 | 3,03 | | Si | 0,04 | 0,24 | 0,09 | 0,12 | |

Based on **Table 8**.it can be seen that graphene oxide contains components such as carbon, nitrogen, oxygen, aluminum, silicon, sulfur, and calcium. Where the highest atomic % is in the elements carbon (47.9%) and oxygen (32.92%), other elements are impurities. Reduced graphene oxide has the highest atomic % in the elements carbon (71.6%) and oxygen (21.13%). Characterization using EDX shows an increase in the percentage of carbon content from GO (47.9%) to rGO (71.6%) by 49.47% and a decrease in the percentage of oxygen content from GO (32.92%) to rGO (21.13%) by 55.79%. There was an increase in the C/O ratio from graphene oxide (1.45) to reduced graphene oxide (3.38).An increase in the carbon element and a decrease in the oxygen element from graphene oxide to reduced graphene oxide proves that the use of ascorbic acid assisted by sonication techniques is able to reduce oxides in graphene oxide. According to research conducted by Alqarawi et al (2021), the % of atoms in reduced graphene oxide is obtained in the elements of carbon (81.5%) and oxygen (15.3%) (Alqarawi et al., 2021). The data on the % of carbon element atoms is close to the % of reduced graphene oxide carbon atoms that have been tested.

## IV. CONCLUSION

The synthesis of reduced graphene oxide (rGO) through the application of green chemistry principles, utilizing ascorbic acid assisted by the sonication technique, has been successfully executed. The optimal conditions were determined to be a 10% concentration of ascorbic acid with a sonication time of 30 minutes. The incorporation of ascorbic acid, assisted by the sonication technique, is aimed at reducing the oxide groups present in graphene oxide. Ascorbic acid, being rich in hydroxyl groups, facilitates binding to epoxy groups, and the sonication technique aids in the release of hydrogen bonds into H2O and carbocations. FTIR results show an increase in % transmittance in hydroxyl, carboxyl, and epoxy groups which proves the reduction of graphene oxide. Furthermore, EDX results show that the % of atoms in oxygen decreases and there is an increase in the carbon element, resulting in an increase in the C / O ratio from graphene oxide to reduced graphene oxide.

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