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RESEARCH ARTICLE

Mixing of Graphite with X-ray Irradiated Water Towards the Exfoliation of Graphene Layers

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Abstract: ***Background*:** This study aims to study the mixing of graphite with water irradiated by X-ray (low energy gamma ray) towards the formation of graphene oxide (GO).

***Materials and Methods*:** The graphite is obtained from Zinc-Carbon (ZnC) battery wastes. This is a simple alternative technique in synthesizing GO based on X-ray irradiation without involving additional chemicals. X-ray irradiation is conducted upon 10 ml of distilled water using 20 kV of X-ray with irradiation time variation of 3 and 4 hours. The X-ray irradiation towards the distilled water causes radiolysis to occur in the water. The graphite solution consists of 0.6 grams of graphite in 100 ml of distilled water. The GO is formed by mixing the X-ray irradiated water with 5 drops of the graphite solution. The sample solutions obtained are shaken several times and left to settle for a night. The samples are then characterized using UV-Visible (UV-Vis) and Fourier transform infra-red (FTIR) spectroscopies, and tunneling electron microscopy (TEM), whereas scanning electron microscope and energy dispersive X-ray (SEM-EDX) characterization is done by coating the sample on glass slides.

***Results*:** The UV-Vis characterization results show a red shift of absorbance peaks from 234.5 nm to 244.5 nm as the time of irradiation is increased. These peaks indicate the formation of GO in the samples. The FTIR characterization results indicate that there are functional groups of OH, C = C, and C - O in the samples, which also show the existence of GO. The SEM images show the surface morphology of the sample, which resembles smooth-quadrilateral lump of clays, and the EDX result shows that the sample is composed of 2.86%, 54.02%, 11.62%, 2.2%, 26.23%, and 3.06% of carbon, oxygen, sodium, magnesium, silicon, and calcium atoms, respectively. The occurrence of carbon and oxygen atoms verifies further the formation of GO in the samples.

***Conclusion*:** Finally, the TEM result shows few-layers of GO materials supported by the electron diffraction pattern showing hexagonal structure of the GO.

Keywords: X-Ray irradiation, water radiolysis, graphene oxide, graphite of ZnC battery.

1. INTRODUCTION

Graphene Oxide (GO) [1,2] can be synthesized through various methods. These various methods are utilized to produce large-scale GO materials with simple and affordable equipment. The most commonly used method in producing GO or graphene material is the Hummer's method. In this method, various chemicals, *e.g.* $KMnO_4$, H_2SO_4, and $NaNO_3$ are used to produce the graphene material. However, it is also well known that the byproduct of this method may contain harmful chemicals to the environment, such as NO_2 and N_2O_4 gasses, and Na^+ and NO_3^- ions [3]. Hence, the Hummer's method has been modified [4] or improved [5, 6] since in order to become more environmental friendly [3]. Another green synthesis of GO is based on water electrolytic oxidation of graphite [7]. These advancements in the synthesis is propelled by the vast application of GO, such as bio-sensors [8], high energy batteries [9], hydrogen storage [10], antibacterial [11], and photocatalytic materials [12].

X-ray, which is a low energy gamma ray, is a form of electromagnetic radiation with very short wavelength, high energy, and high penetrating power. The ability of X-rays to

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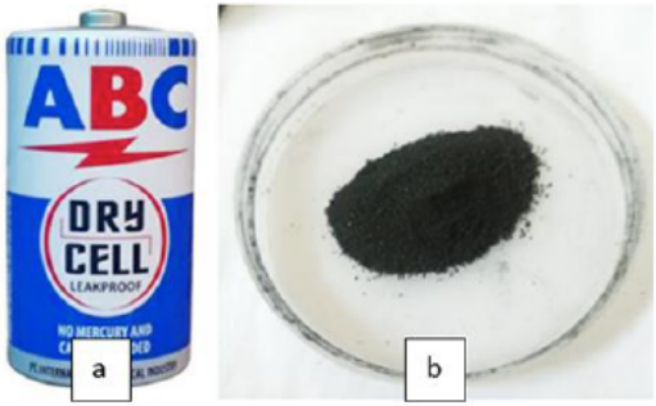

Fig. (1). a) A ZnC battery waste and **b**) graphite powder from the ZnC battery.

Fig. (2). The upper (**a**) and front (**b**) views of the X-ray generator. A GM counter is placed in front of the X-ray tube (**c**).

penetrate a material is used in the medical field for radiographic diagnosis [13] and radiotherapy [14]. On the other hand, gamma ray may also be used to produce graphene [15,16] or reduced-GO (rGO) [17] materials. In this case, a general two-step process is conducted to produce rGO or graphene, *i.e.*: 1) synthesizing GO using the Hummer's method, and 2) synthesizing the rGO or graphene by irradiating the GO *via* the gamma ray. The main role of the gamma ray is to induce reduction reactions, such that oxygen atoms can be depleted from the graphene layers *via* water radiolysis where water is the solvent. Water radiolysis [18] itself is a process where molecule dissociations occur in the presence of ionizing radiation producing radical ions of H^+ and OH^-. These radicals are very reactive and may recombine into superoxide (HO_2) and peroxide (H_2O_2), such that they may assist in oxidation and reduction reactions. Moreover, gamma-ray irradiation is considered environmental friendly because it does not cause pollution in the environment.

In this study, we report the formation of GO by indirect X-ray irradiation. This means that the X-ray irradiation is only conducted upon water and not on the graphite. The irradiated water is then mixed with the graphite solution in order to induce the exfoliation of graphite into GO purely by water radiolysis. Hence, in this study, instead of using the Hummer's method, we explore the possibility of using the oxidation ability of water radiolysis in obtaining GO without any chemicals heavily used in the Hummer's method. To the best knowledge of the authors, this study has not been conducted before.

The graphite material used in this study is the rods from zinc-carbon (ZnC) battery wastes [19]. ZnC batteries (see Fig. **1a**) are commonly used in various electronic devices, for example, radio, remotes, clocks, toys, and many more. The fact that ZnC batteries cannot be recharged becomes a problem in terms of their wastes. The recycling of these batteries is still being studied [20]. Therefore, this study is conducted in the spirit of recycling wastes; especially the rods of ZnC batteries into GO based on a simple and affordable X-ray irradiation method.

2. MATERIALS AND METHOD

The materials used in this study are i) powder of carbon rods [Fig. **1b**] from ZnC battery wastes and ii) distilled water. No other chemicals are used in this study. Moreover, the tools used in this study are (i) a blender, (ii) a measuring glass, (iii) beaker glasses, (iv) sample tubes, (v) a stopwatch, (vi) a digital scale, (vii) aluminium foils, (viii) an X-Ray generator (580 TEL-X-OMETER) (Figs. **2a** and **2b**), (ix) UV-Vis spectrophotometer (Shimadzu UV-2550), (x) XRD (Rigaku Miniflex 600), (xi) SEM (CoXem), (xii) FTIR (Nicolet Avatar), (xiii) Geiger-Muller (GM) counter [Fig. **2c**], and TEM.

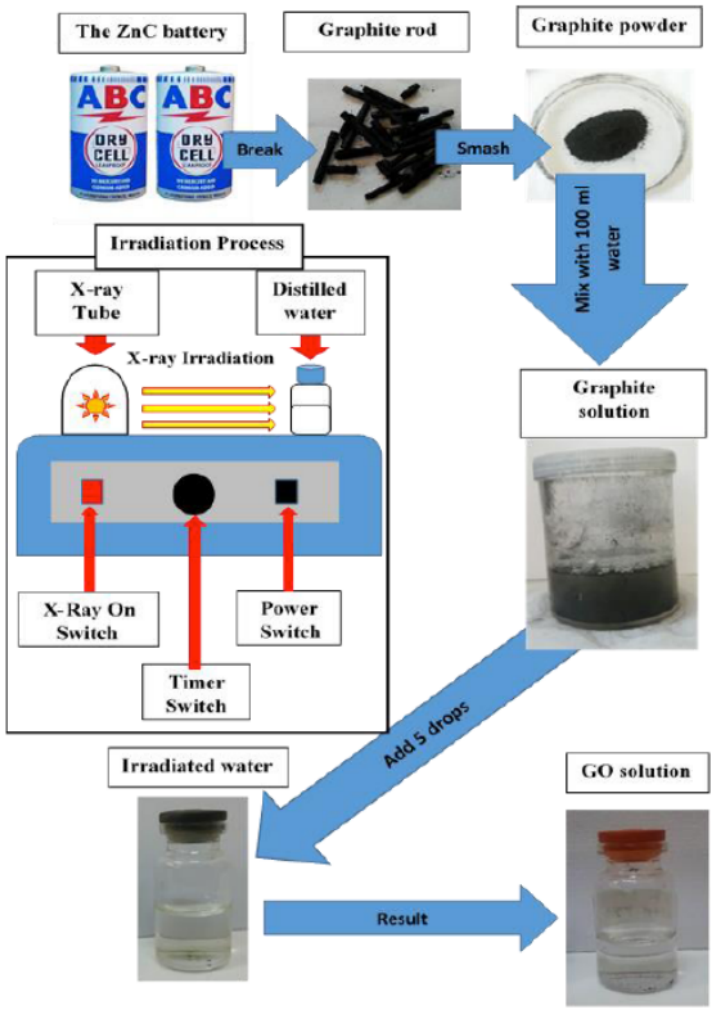

Fig. (3). The illustration of the experimental procedure.

Firstly, the carbon rods are grinded into graphite powder. Furthermore, the graphite solution is produced by mixing 0.6 grams of the powder with 100 ml of distilled water using a blender for 2 minutes. This is illustrated in the upper part of Fig. (**3**).

A preliminary observation is conducted to check the radiation produced by the X-ray generator. This is done by placing a GM counter in front and parallel to the X-ray tube and leaving some space between them (see Fig. **2c**). Various samples in sample bottles are then alternately put between the X-ray tube and the GM counter, *i.e.* without any sample (air), distilled water, and graphite solution. For each of the sample, the X-ray generator is turned on such that the GM counter counts the radiation produced by the generator in count per minute (cpm). In this case, we do not convert the results into other radiation units. Furthermore, we also conducted preliminary UV-Vis tests upon the distilled water before and after being irradiated by the X-ray.

The experimental procedure in this study is straightforward, *i.e.*: X-ray irradiation of distilled water in sample tubes with a voltage of 20 kV. The irradiation is conducted upon two (2) sample tubes each consisting 10 ml distilled water for time durations of 3 and 4 hours, respectively. This is again depicted graphically in Fig. (**3**) in the "Irradiation Process" box. In this case, we do not calculate the dose of the X-ray exposed to the samples. The parameter used in this study, as mentioned above, is only the duration time of the X-ray irradiation. Hence, the information of the X-ray dose is implicitly contained in the parameter used in this study. After the irradiation process, each of the irradiated water sample is added with five (5) drops of the graphite solution, shaken several times, and then left to equilibrate overnight. This may be observed in the lower part of Fig. (**3**).

Subsequently, the samples are characterized. UV-Vis and FTIR spectroscopies, and TEM are conducted in liquid-phase, whereas SEM-EDX is done in solid-phase. The solidified

Sample	Averaged cpm
Air	41.4
Distilled water	39.4
Graphite solution	17.6

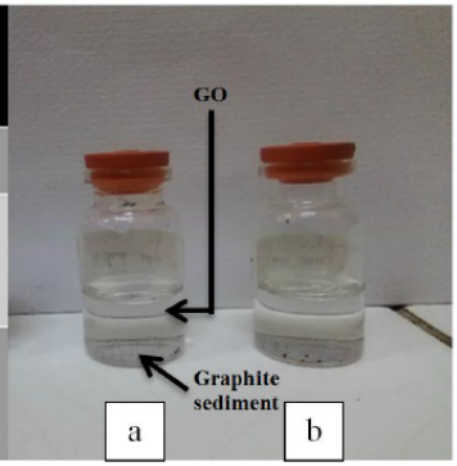

Fig. (4). The GO solutions obtained by mixing graphite solution with irradiated water for 3(**a**) and 4(**b**) hours.

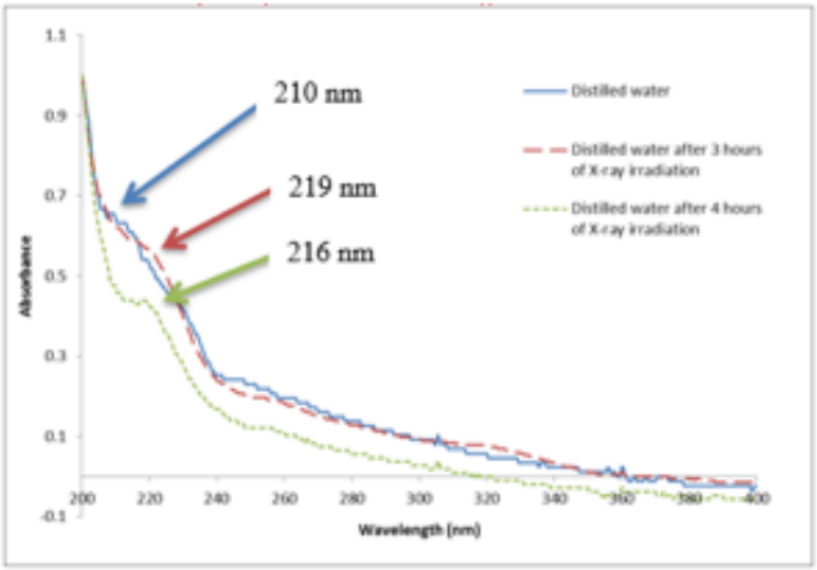

Fig. (5). UV-Vis characterization results for distilled water before and after X-ray irradiation.

sample is obtained by dipping glass slides upon the surface of the sample solutions. The glass slides are then heated inside an oven for 10 minutes with a temperature of 250 ^{o}C.

3. RESULTS AND DISCUSSION

Fig. (**4**) presents images of the samples (in bottle samples) obtained from this study, *i.e.*: mixing graphite with irradiated distilled water for 3 (Fig. **4a**) and 4 (Fig. **4b**) hours. The images show clear solution of GO with a small amount of graphite sediments on the bottom of the bottles. The separation between the liquid and solid phases is induced by gravity as it is left for a night. The liquid solution should contain exfoliated graphite caused by water radiolysis, whereas bulk or larger graphite materials settle in the bottom. There is no difference in the physical appearance for the samples with 3 and 4 hours of X-ray irradiation of the distilled water.

Moreover, the preliminary observation results concerning the X-ray generator performance may be observed in the table on the left of Fig. (**4**). The table shows measurement results of radiation count (in cpm) for air, distilled water, and graphite solution as X-ray radiation absorbents. The results show that both distilled water and graphite solution absorb X-ray radiation. Moreover, distilled water absorbs around 5% of the X-ray radiation (with respect to air) confirming that water radiolysis should occur in the distilled water. The physical appearance of the irradiated distilled water is similar to that of the distilled water without irradiation. A further comparison of the distilled water before and after X-ray irradiation may be observed based on the UV-Vis results in Fig. (**5**). It may be observed that a redshift occurs after the distilled water is irradiated by X-ray. This indicates that the X-ray irradiation changes the chemical formulation of the distilled water, *i.e.*: into radicals.

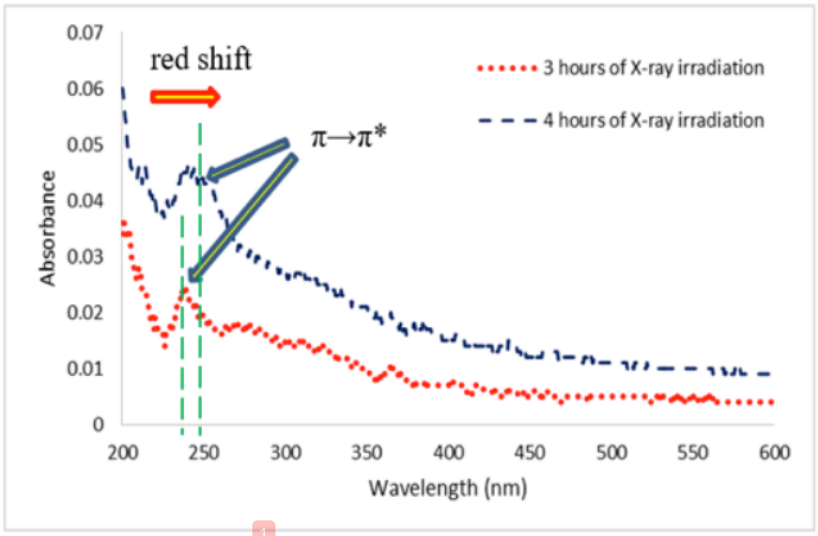

Fig. (6). UV-Vis characterization results for the GO solution with 3 and 4 hours of distilled water irradiation time.

UV-Vis characterization results of the GO samples can be observed in Fig. (**6**) with 3 (red-dotted line) and 4 (blue-dashed line) hours of X-ray irradiated distilled water. It can be observed from the UV-Vis results that there are pairs of peaks, *i.e.*: 234.5 and 270 nm, and also 244.5 and 300 nm for 3 and 4 hours of X-ray irradiated distilled water, respectively. The first (234.5 nm and 244.5 nm) and shouldering peaks (270 nm and 300 n m) represent electronic transitions of $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$, respectively. These pairs of peaks clearly show the existence of GO in the samples as obtained by Saxena *et al* (2011) and Lai *et al*. (2012). Moreover, a red-shift occurs from the peaks of 234.5 nm to 244.5 nm for 3 to 4 hours of X-ray irradiated distilled water, indicating a further reduction of the GO material [23]. The UV-Vis results are also consistent with the results obtained by Wisnuwijaya *et al*. (2017), especially for the GO material synthesized from the same material of ZnC battery carbon rods using a custom-made ultrasound generator. It may be observed in a study [19] that the UV-Vis profile of the graphite powder from the carbon rods does not produce any peaks, which is a further confirmation of graphene layers exfoliation by the occurrence of GO peaks in this study. It may also be observed that the GO peaks obtained in this study are better than the study by Wisnuwijaya *et al* [19], *i.e.*: 234.5 nm and 244.5 nm compared to 221 nm to 227.5 nm, respectively.

FTIR characterization results of the GO solutions are presented in Fig. (**7**) with variation of distilled water irradiation time for 3 (red-dotted line) and 4 (blue-dashed line) hours. For all variation of the water irradiation time, we can find OH (hydroxyl) groups, which are shown at a band around 3400 cm^{-1} indicated by a U-shaped indentation on the band. In addition, there are C = C and C - O functional groups at bands of 1630 cm^{-1}, and 1250 cm^{-1}, respectively, for all distilled water irradiation time. The aforementioned oxygen functional groups, *i.e*: C - O and OH, further strengthen the indication of GO in the samples. The C = C bonds are consistent with the $\pi \rightarrow \pi^*$ transitions of the UV-Vis results (Fig. **6**) again in accordance with the results obtained by Saxena *et al* (2011). The FTIR profiles obtained in this study are similar to that of the FTIR results obtained by Gurunathan *et al* (2015) with the existence of OH, C = C, and C - O functional groups. Furthermore, it may be observed in Fig. (**7**) that increasing the distilled water irradiation time (from 3 to 4 hours) tends to decrease the OH groups and increase the C - O groups. The reduction of the OH groups is expected as the oxygen content in the GO material is being further reduced. However, this is somewhat challenged by the increase of the C - O groups, which slows down the reduction process.

The SEM images of the solidified sample are presented in Fig. (**8**) with various magnifications. The surface morphology images with 500X (Fig. **8a**) and 1000X (Fig. **8b**) magnifications show the occurrence of quadrilateral-like materials, which are arranged in the form of small islands. With the addition of magnifications to 5000X (Fig. **8c**) and 10000X (Fig. **8d**) further detail of the materials are observed, *i.e.*: smooth surfaces, blunt edges, and overlapping each other resembling clay piles. These SEM images are quite similar to that obtained by Khrisna *et al* (2015), which shows the effect of gamma ray irradiation towards graphite material. However, in this case, the formation of the GO material is ultimately caused by the oxidation activities of the radicals in the samples.

The piles of materials that resemble smooth-quadrilateral clay consist mainly of carbon and oxygen atoms with percentages of 2.86% and 54.02%, respectively, according to the EDX characterization results presented in Fig. (**9**). It may be observed that the oxygen atoms dominate the EDX results showing the abundance of oxygen content in the sample in accordance with the FTIR results in Fig. (**7**) and the existence of GO material. However, the hydrogen element, which occurs in the FTIR results, is not detected in the EDX results. This may be caused by the depletion of the hydrogen atom in the preparation of the solidified sample. In addition, other elements are also found, namely silicon (26.23%), sodium (11.62%), magnesium (2.2%), and calcium (3.06%). These aforementioned metal elements may be present at the

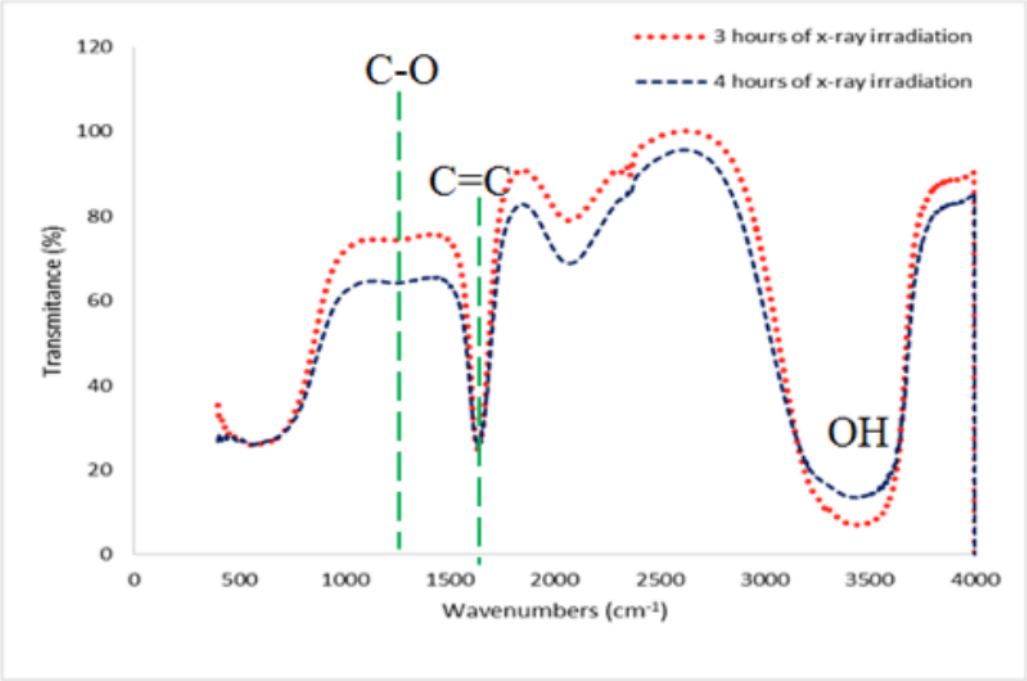

Fig. (7). FTIR characterization results for GO solutions.

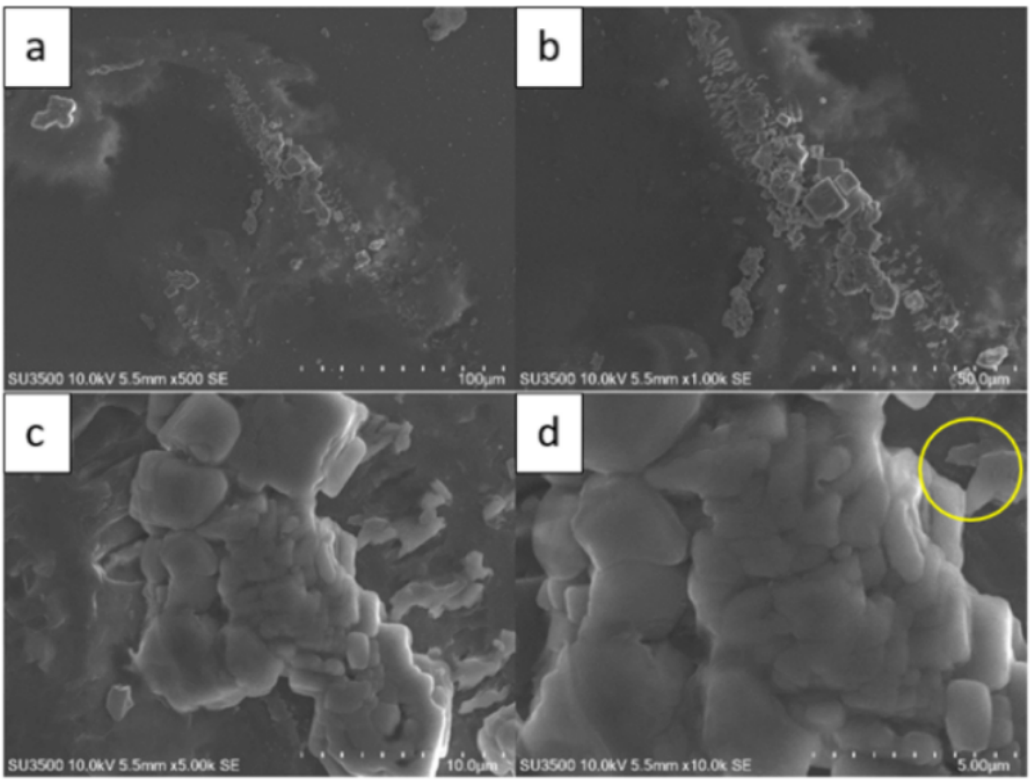

Fig. (8). The SEM images of the GO sample with magnifications of 500X (**a**), 1000X (b), 5000X (**c**), and 10000X (**d**).

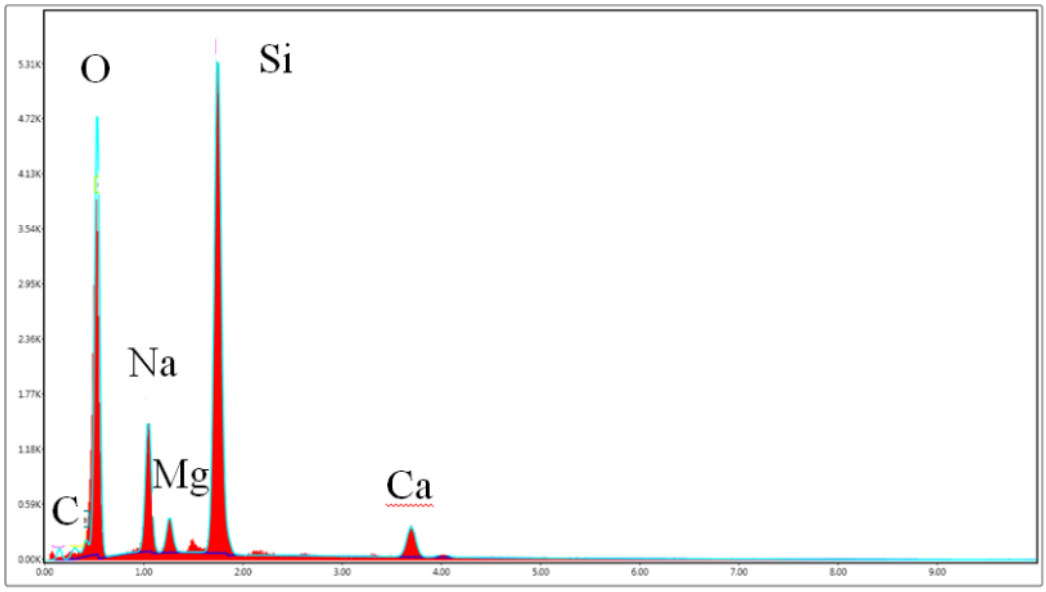

Fig. (9). The EDX results of the solidified GO samples.

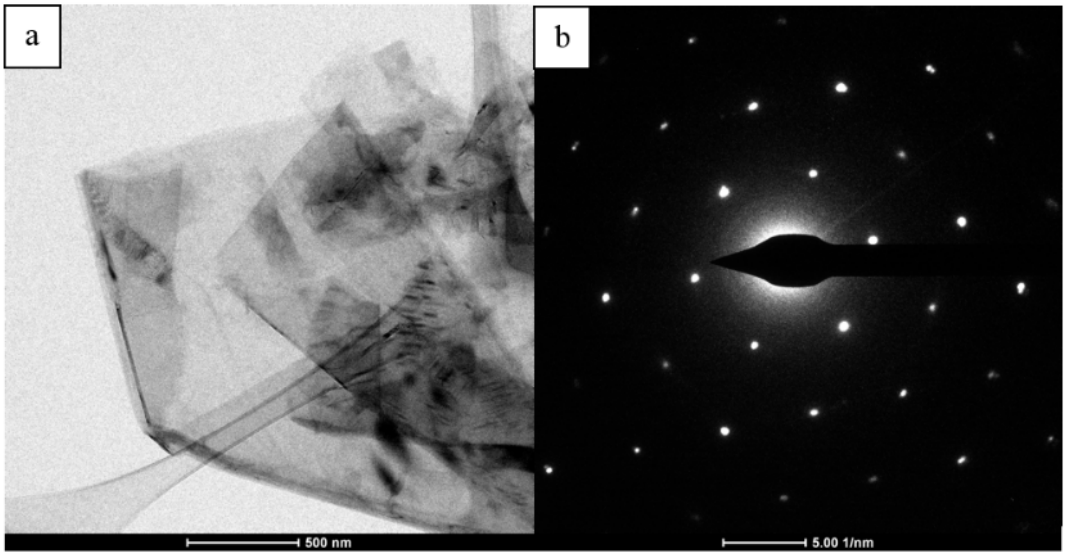

Fig. (10). The TEM results of the GO solution.

preparation stage of the sample for the SEM-EDX characterization. A high percentage of the silicon element may come from the glass slides used as a base for the GO samples.

Finally, the TEM image of the GO material may be observed in Fig. (**10a**). This image clearly shows a few layers of the GO material. These layers are stacked into 2 to 3 transparent layers. Furthermore, the Selected Area Electron Diffraction (SAED) of the GO material can be observed in Fig. (**10b**), which shows the hexagonal structure of the GO.

The X-ray exposed to distilled water causes radiolysis of water resulting in various radicals, *i.e.*: hydrated electron, hydrogen radical (H*), hydroxyl radical (HO*), HO_2*, OH^-, H_3O^+, H_2, and hydrogen peroxide (H_2O_2) [15,18]. Without the direct effect of the X-ray irradiation and any other chemicals, the intercalation of graphite upon the graphene layers should mainly be triggered by these radicals. Here, we propose a chemical reaction that might contribute to the formation of GO involving hydrogen peroxide as an oxidative agent, similar to H_2SO_4 and $KMnO_4$ in the Hummer's method [25], *viz.*:

$$\text{Graphite} + H_2O_2 \longrightarrow \text{GO} + H_2O. \qquad (1)$$

Furthermore, other oxidative agents, such as OH^- and OH* also interact with the graphite in between the graphene layers to exfoliate the graphite material. As in the salt-intercalation exfoliation [26], the positive ions may penetrate the spaces between the graphene layers to assist the exfoliation process.

CONCLUSIONS

The mixing of graphite solution and distilled water irradiated with X-ray has been conducted in order to produce GO. The UV-Vis characterization results show a red shift of the absorption peaks from the wavelength of 234.5 nm to 244.5 nm as the distilled water irradiation time is increased. The FTIR characterization results show the occurrence of OH, C = C, and C - O functional groups. The SEM images show the surface morphology of the sample that resembles smooth-quadrilateral materials piling like clay. Finally, the sample material is composed of carbon and oxygen atoms, which are characteristics of GO, and other elements, *viz.*: sodium, magnesium, silicon, and calcium. Hence, the UV-Vis, FTIR, SEM, and TEM results confirm the formation of GO material as graphene layers are exfoliated with the assistance of the radicals in the sample. However, a step-by-step procedure in the production process of the GO based on water radiolysis is not yet established in this study. Therefore, this may be explored in future studies.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not applicable.

HUMAN AND ANIMAL RIGHTS

No Animals/Humans were used for studies that are the basis of this research.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

Not applicable.

FUNDING

None.

CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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