XRD and Microscopic Images for Synthesis Graphite Nanoparticles by Oxidation Method

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Abstract

Graphite nanoparticles were successfully synthesized using mixture of H2O2/NH4OH with three steps of oxidation. The process of oxidations were analysis by XRD and optics microscopic images which shows clear change in particle size of graphite after every steps of oxidation. The method depend on treatments the graphite with H2O2 in two steps than complete the last steps by reacting with H2O2/NH4OH with equal quantities. The process did not reduces the several sheets for graphite but dispersion the aggregates of multi-sheets carbon when removed the Van Der Waals forces through the oxidation process.

Key Words: Graphite, XRD, Nanoparticle, H2O2/NH4OH.

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Introduction

The applications for huge fields of science commonly needed for specific properties of materials such high thermal and chemical stability with electrical and mechanical properties which represent critical parameters. Graphite maybe the best typical and real practical example for the development in manufacturing materials for a long time ago when influence directly in the macroscopic behavior of the materials [1]. Graphite can rolling to forming carbon nanotubes CNTs [2] and Nano sheets NS [3] or graphene G [4]. Graphite when losing many Van der Wales forces between the sheets, mostly. The new species commonly known as graphite or carbon Nano sheets, and graphite oxide, which characterized by more electrical, mechanical, thermal and chemical properties with excellent stability [5]. Usually graphite Nano sheets can be prepared by chemical vapor deposition or modified with radio-frequency and microwave plasma, arc-discharge, and chemical reduction of exfoliated graphite oxide. Graphite from many sources are commonly used as the starting material for preparing graphite oxide or Nano sheets due to inexpensive with purity. In this study, a modified method for production of graphite Nano sheets using three steps of simple The steps of oxidation were analyzed by X-ray analysis and optic macroscopic.

chemical treatment with H_2O_2 and NH_4OH which does not require expensive or complex equipment.

Experimental

The graphite was purchased from Fluke, and Hydrogen peroxide (30%) from Barcelona-Spain. Ammonium hydroxide. 37% was supplied from Sigma Aldrich. The process of oxidation was done by modifying piranha reagent as we reported in our previous work [6] which represent in diagram 1 with three steps. The first step includes dispersion 1 g of graphite in 15 ml of H_2O_2 with stirring for 3 hours at 10 °C than drying at 80 °C for 4 hours, which refer to it by [Graphite-(O) n].

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The second step includes produce [Graphite-(O)m] by the same process which mentions in step one when re-oxidation with H_2O_2. The second sample of graphite was added in 15 ml of H_2O_2 at 10 °C for 3 hours than adding 15 ml of NH_4OH to solution with stirring for 1 hours to produce [Graphite-(O)o]. The third step will be forming suspensions include precipitated material [Graphite-(O)o] and black solution (e) [Graphite-(O)x] which precipitate after more than 1 hours. The graphite after every step of oxidation were increasing the ratios of oxidation n< m< o << x respectively.

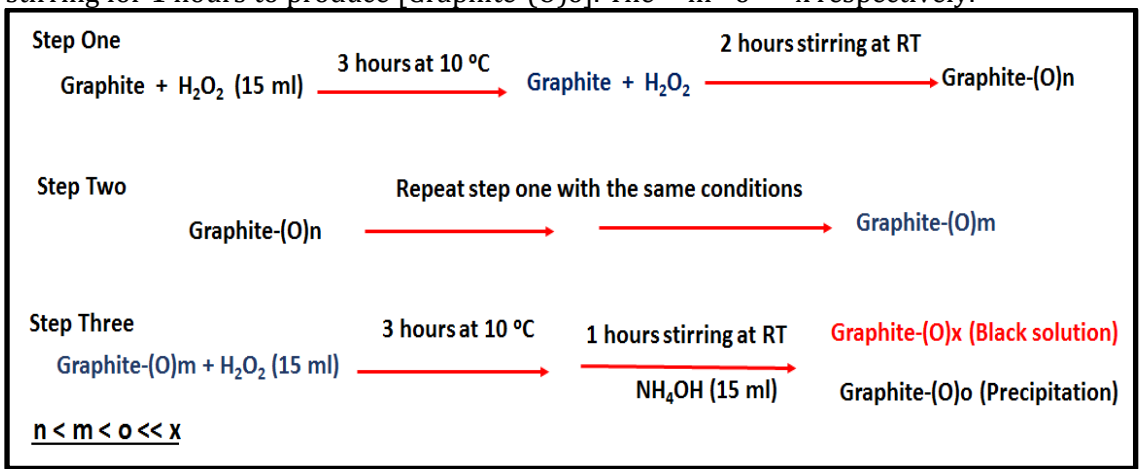

Figure 1. Skim diagram for the three steps of graphite oxidation process

Results and Discussion

The crystalline and morphology analysis for process of graphite oxidation were done by X-ray diffraction and microscopic optics. The (Riga Rotalflex) (RU-200B) apparatus were used to obtain the X-ray diffraction (XRD) patterns using Cu Kα radiation at 0.15405 nm with a Ni filter. The tube current and voltage was 100 mA and 40 kV respectively while scan rate with resolution was 5°/min and 0.02° for analysis from 2Θ=10° to 2Θ= 50°. The nature of distribution and composition for graphite species during oxidation were taken with images at 3 µm scope by lab microscopy CD.

Figure 2 shows the X-ray diffraction (XRD) patterns of graphite powder before and after treatment with three steps. The XRD patterns of graphite and graphite oxide were shown two characteristic peaks of graphite at 26.5° and 43° which refer to 002 and 100 patterns respectively [7]. The characteristic behavior for the two peaks were shifted towards less 2Θ which increase from (a) to (d) samples respectively due to increase the function groups after each step [7-8]. Many noises for all the scopes of XRD analysis, which increase with increase the effect of oxidation. The last behavior can be related to reduce the number of sheets after each step of oxidation, which increase the noise when represent the nature of structure after increase graphene sheets [9]. Figure 3 refers to XRD analysis for the step 3 when produce precipitation material (d) and black suspenders (e) which isolate precipitate after more than 1 hours.

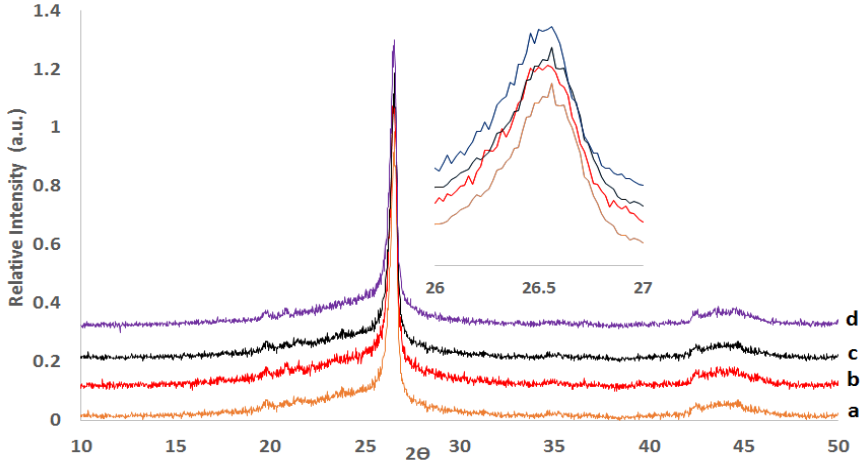

Figure 2. XRD patterns for oxidation process of graphite before oxidation a, and after oxidation with three steps b=[Graphite-(O)n], c=[Graphite-(O)m] and d=[graphite-(O)o].

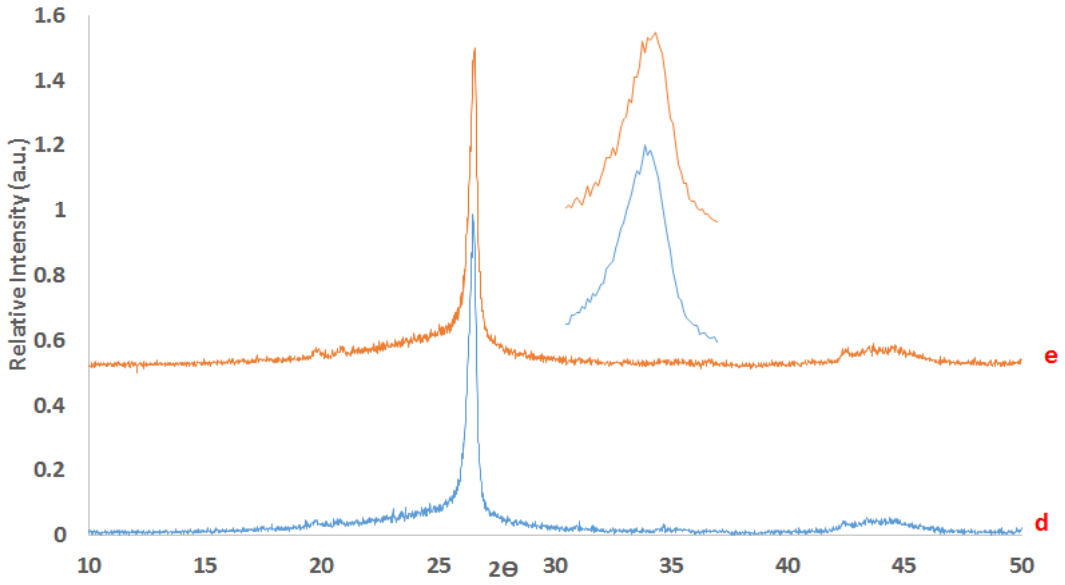

Figure 3. XRD patterns for graphite after three steps of oxidation d (precipitation) and for e (suspension).

Scherer's equation (d= (K*λ) /(β*cos θ) was depend to determine partial size (d) for materials [10] from the interaction of incident, monochromatic light (λ) with specific angle (θ) which depend on the nature of material. The value of (d) was calculated for the line broadening at half the maximum intensity (β = FWHM) with the shape factor (K) typically equal 0.9. The particles size was change from 27 nm to 25, 21, 19, and 17nm for graphite before oxidation, [Graphite-(O)n], [Graphite-(O)m], [graphite-(O)o] and [graphite-(O)x] respectively.

The steps of oxidation were characterized by optic microscope when show clear images for the conversion of graphite to less size to reduce the density after each step. Figure 4, refer to graphite before oxidation with many graphite particles that characterized by aggregates which arrange between 0.4-4 µm due to Van- Der Wales forces. Figure 5, 6, and 7 refer to three steps of oxidation b,c, and d respectively which clearly shows reduced the agglomerate after every step of oxidation that mostly related to substitute Van- Der Wales forces by function groups.

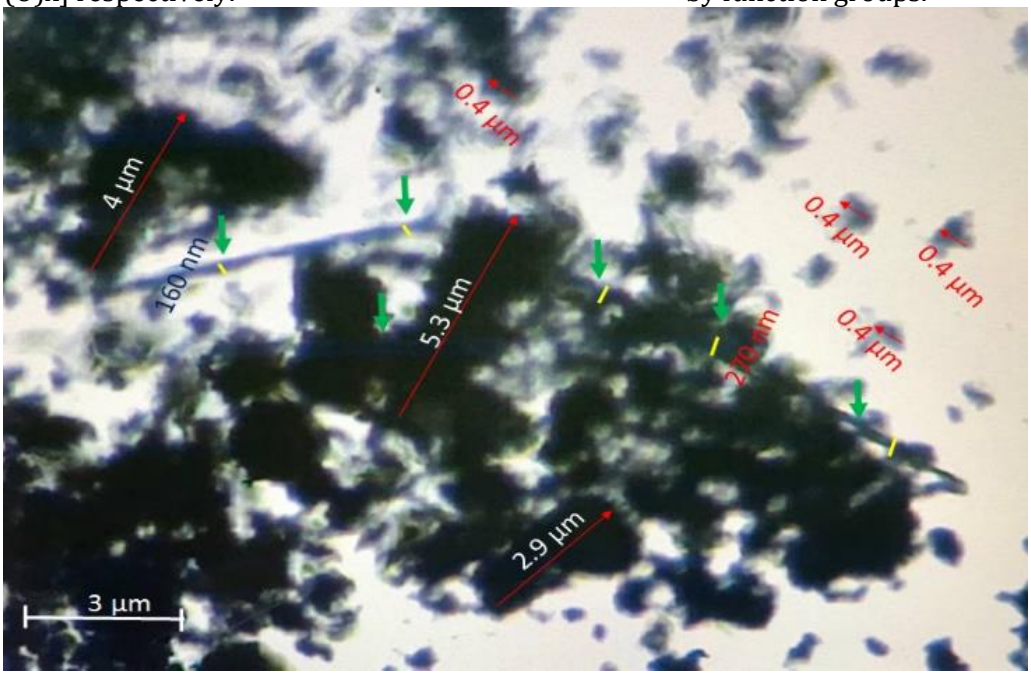

Figure 4. The optics images for graphite before oxidation

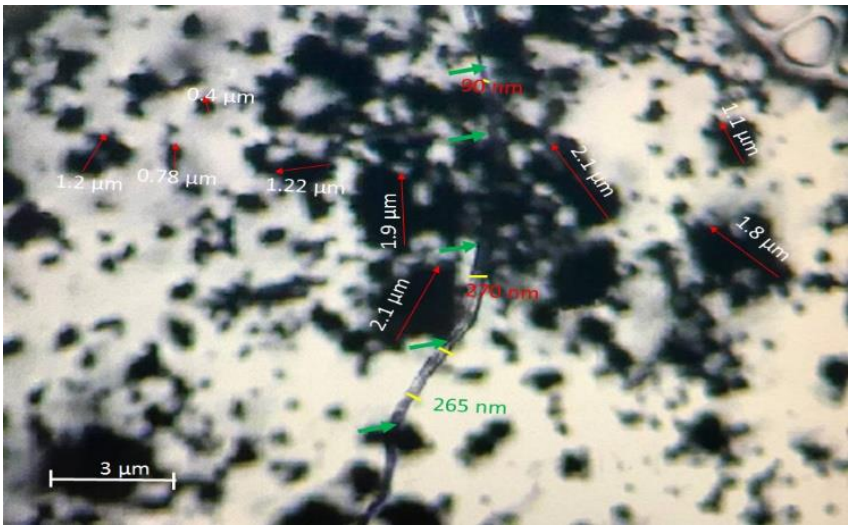

Figure 5. The optics images for graphite after oxidation in step 1 to produce [Graphite-(O)n]

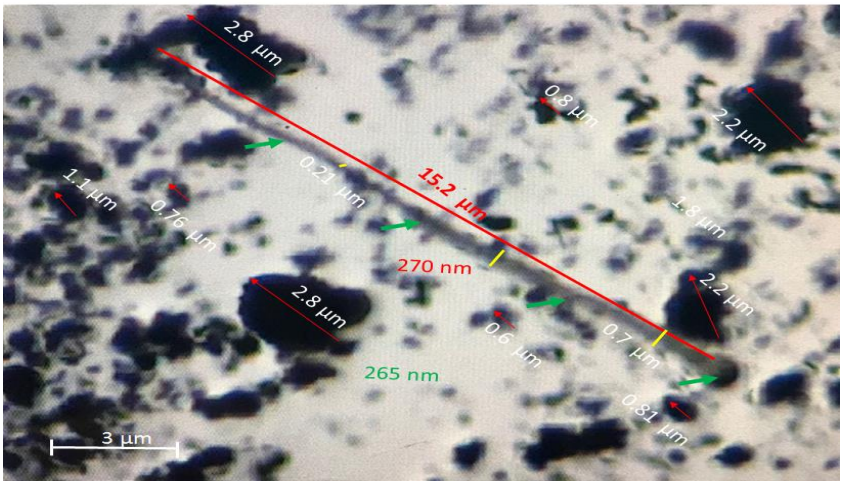

Figure 6. The optics images for graphite after oxidation in step 2 to produce [Graphite-(O)m]

The particle size of graphite were reduced to 50% after the first step when shows 0.4-2.1 µm in diameter. Treatment of graphite after two times by H_2O_2 shows another reduce reach to 16 % of particle size as compared with first step when appears many species with size less than 1.4 µm.

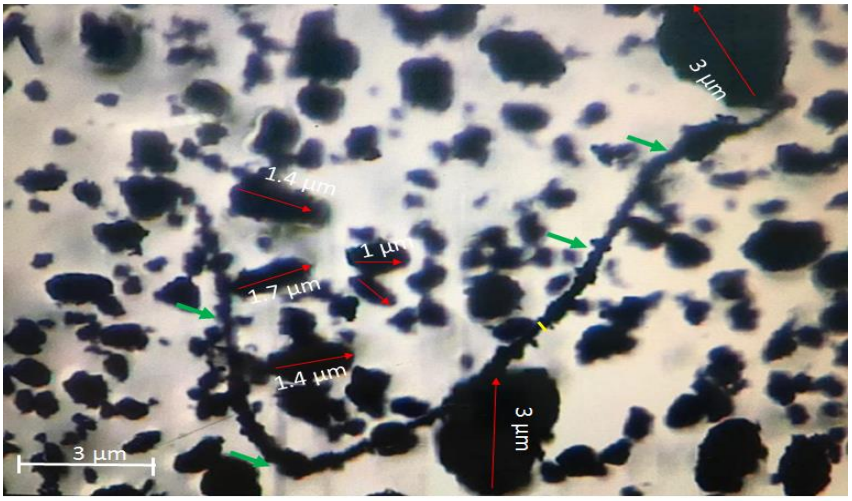

Figure 7. The optics images for graphite after oxidation in step 3 to produce [Graphite-(O)o]

The optic images which mention before shows filaments shapes which refers to carbon nanotubes with many walls may form during the process of synthesized graphite. The amazing note was the tubular structure appeared cleaner after each step of oxidation to reach for figure 8 when filament

shows high purities. This result was agreement with many reported literature that deals with the purification of carbon nanotubes by using oxidation reagent [11-12]. From the optic microscope images it can be seen that MWCNTs were converted from a bundle of carbon filaments form tow single filaments of MWCNTs due to remove Van-Der Walls forces as shown in figure 8.

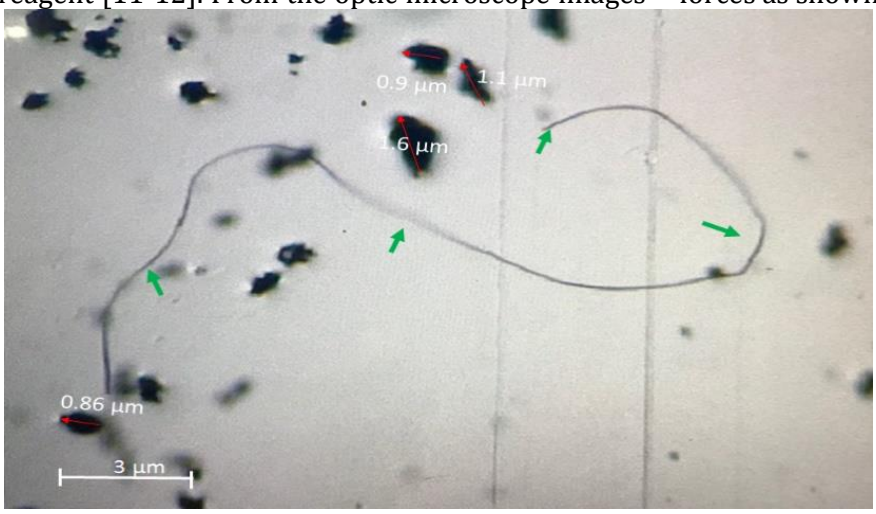

Figure 8. The optics images for graphite after oxidation in step 3 which precipitated after 1 h. to produce [Graphite-(O)x]

Conclusion

In this work we produce simple technical to synthesized graphite oxide or Nano sheets using the modified oxidation method in H_2O_2/NH_4OH solution. The crystalline graphite structure and particle size with the nature of the Nano sheets was analysis of, XRD and optic microscopy. The synthesized graphite Nano sheets which produce from this method can be depend for production of few-layer graphene sheets. The steps of oxidation were succeeded to reduce the graphite oxide particle size and agglomeration. The process of oxidation shows many carbon nanotubes, which mostly MWCNTs that may produce during the synthesized process of graphite. The MWCNTs were dispersed when oxidized by the oxidation process, thus it could be beneficial for many applications.

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