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Surface Functionalization of Nano Graphene Oxide by Amino Acid

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***Abstract*---**In this study, nano graphene oxide (NGO) was synthesized by modified Hummer's method, the functionalization surface of nano graphene oxide (NGO) by amino acid (methionine, cysteine) by covalent functionalization of NGO with amino acid group using microwave 20% power (140 W) and characterization by Fourier transform infrared spectrophotometer (FT-IR), X-ray diffraction spectroscopy (XRD), and field emission scanning electronic microscopy (FESEM). The calculated particle size of prepared compounds GO, GOM, and GOC is dependent on the pattern X-ray diffraction spectroscopy (XRD), XRD using (Debye – Scherer) and (Williamson – Hall) equation GO (16 nm), GOM (16.83nm), and GOC (19.nm). The FESEM exhibited for graphene oxide surface has many layers of light grey and has kinked regions and wrinkled edges, while re-stacked layers are light colored. The surfaces of GOM and GOC have sharp edges, crumpled and rippled.

***Keywords*---**Nano Graphene oxide, microwave, amino acid, (XRD) X-ray diffraction.

Introduction

Graphene oxide (GO) was exhibited high specific surface area properties because of congaing epoxy, hydroxyl, and carboxyl groups biomedical imaging, biological sensors, and medication delivery are examples of uses, and high biocompatibility with human cell [1,2]. Recently, some amino acids, peptides and proteins can be highly adsorbed ith graphene oxide on the surface electrostatic interactions, hydrophobic interactions and hydrogen bonds [3, 4]. Amino acids are very important to life, contains at least one amino group and one carboxyl group, their side chains vary between different amino acids. Amino acids have multiple functions and are the basis of protein [5]. In the present study functionalize of

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graphene oxide by amino acid using microwave in the preparation and measure crystalline size of compounds GO, GOM, GOC.

Methodology
Materials

Hydrochloric acid, sulfuric acid, tetrahydrofuran (THF), dimethylformamide (DMF) from BDH, graphite, hydrogen peroxide, potassium permanganate, sodium nitrate, methionine, cysteine from Fluka,

Instruments

Fourier transforms infra-red (FTIR-8400S, Shimadzu),X-ray diffraction (XRD, LabX-XRD-6000, Shimadzu), Field emission scanning electronic microscopy (FESEM, 5KV, Zeiss).

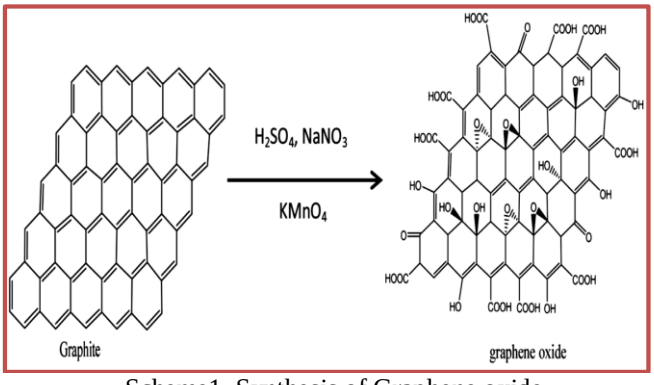
Synthesis of Graphene oxide

To begin, the graphite flakes (1 g) and $NaNO_3$ (0.5 g) were cooled to 0-5C^0 in an ice bath with a small amount of H_2SO_4 (23 mL). Then 3 g of $KMnO_4$ was gradually added to the mixture. For 30 minutes, the mixture was heated to 35°C and magnetically stirred. After that, 5 mL of 30% H_2O_2 was added to the mixture, which changed the color to yellow. The precipitate was washed with distilled water. The Hummer's methods were used to synthesize the graphene oxide. [6] as shown in Scheme 1

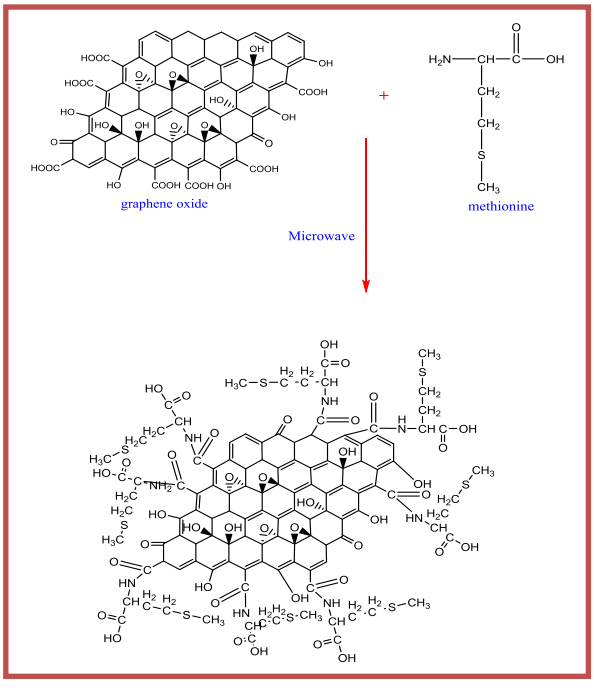
Synthesis of GOM and GOC

Compounds

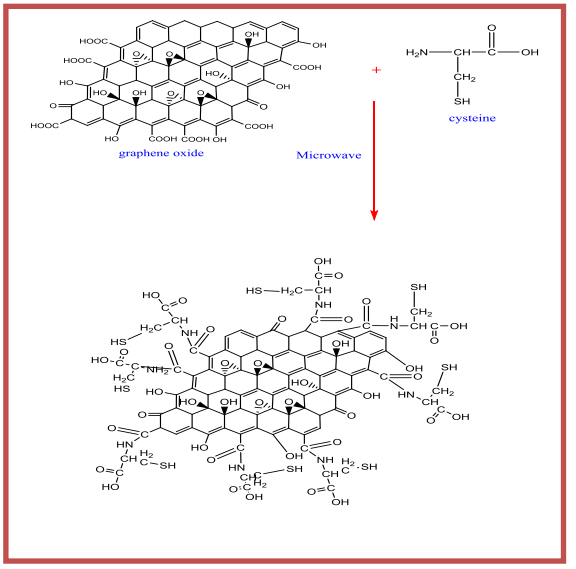
0.5 g of GO was ultra-sonicated for 30 minutes in 50 ml DMF in a beaker, followed by 1.5 g and 10 mmole of Methionine, which was also ultra-sonicated for 30 minutes. In a microwave, the suspension was heated for 30 minutes at 20% power (140 W). After that, the suspension was washed with 200 mL anhydrous ethanol several times, followed by distilled water and ethanol. To get the black powder product, the acquired GOM was dried at 70°C for 6 hours (GOM) Scheme 2. GOC was synthesized using the same process, but with a different cysteine weight (1.21 g) [7], as indicated in Scheme 3.



Scheme1. Synthesis of Graphene oxide



Scheme2. Synthesis of GOM



Scheme3. Synthesis of GOC

Results and Discussion

Figures (1-3) show spectra FT-IR of the (GO, GOM, and GOC) powders, respectively. The peak at 3386.39 cm-1 (O-H) stretching vibrations can be observed in the (GO) spectrum., 1724.36 cm-1was the strong (C=O) stretching , the peak of C=C aromatic ring stretching and (C-OH)bending were 1622.8 cm-1 and 1378.85 cm-1, respectively, and the peak at 1029.8 cm-1 corresponds to the (C-O) epoxy group's characteristic [12]. The peak at 3434.6 cm-1 The stretching of (OH) groups can be shown in the spectra of GOM and GOC figures 2 and 3. The asymmetric symmetric of (-NH2) stretching vibrations are ascribed to the appearance of absorption bands at 3334 and 3222 cm-1, respectively. Peaks 2956 and 2823 cm-1 are attributed to the asymmetric and symmetric stretching vibrations of (C-H) bands .Furthermore, the peak at 1639.2 cm-1 is due and to aromatic ring (C=C)stretching vibration, whereas The two peaks at 1559.17 and 1122.37 cm-1, respectively, are attributed to a combination of (N-H) bend, C=O of amide, and C-N stretching vibration, while the peak at 797.421 cm-1 is due to aromatic ring C=C bending. [13,14].

The technique of X-Ray diffraction (XRD) is commonly used to characterize crystalline materials. Table-1 and figure (4-6) pattern of graphene oxide (GO, GOM and GOC) exhibiting 2θ with d- spacing, this indicates that the methionine and cysteine were successfully attached to the GO surface during the modification [12, 15]. The size crystallite was determined by (Debye – Scherer) equation [7] for the new synthesized compounds using The X-ray Diffraction patterns.

$$D=K\lambda / \beta \cos \theta \text{ ---------- (1)}$$

Where D is the crystallite size, (λ) is wavelength (nm) of X-ray, (HWHM) is the half-width at half maximum, the hape factor (K) is usually taken to be 0.9, (θ) is x-ray

angle, the measure crystallite dimension according to equation (1) the calculated size of GO is (16 nm) but for the GP (6.10 nm), another equation (Williamson – Hall) to calculate the size of crystallite [17] :

$$\beta \times \cos\theta = [\ (k\ \lambda)/(D\ )\ ] + [\ 4£\ x\ \mathrm{Sin}\ \theta] \text{ ----(2)}$$

Figure (7 and 8) describe the crystallite size measurement of GOM and GOC by (Williamson – Hall) equation. Where (£) the the particle micro strain, the estimated crystallite size from graphic between ($\sin\theta$) on x-axis, ($\beta \times \cos\theta$) on the y-axis, calculated (D) by intercept for (K λ / D), the size of crystallite measuring using by (Williamson – Hall) equation) and micro strain for GOM and GOC shown in table (2).

The Field Emission Scanning Electron Microscopy (FESEM) is a technical images surface morphology. Figure 9 exhibiting graphene oxide which has a smooth, low thickness sheet and flat, graphene oxide surface has many layers of light grey and has kinked regions and wrinkled edges. Colour; also contain some cavities, crumpled flakes and stacks associated with each other. Figure 10 and 11 in GOM and GOC surface shows multiple layers, part of which are dark grey colour, while re-stacked layers are light coloured, surface of GOM and GOC have sharp edges crumpled and rippled [5.17].

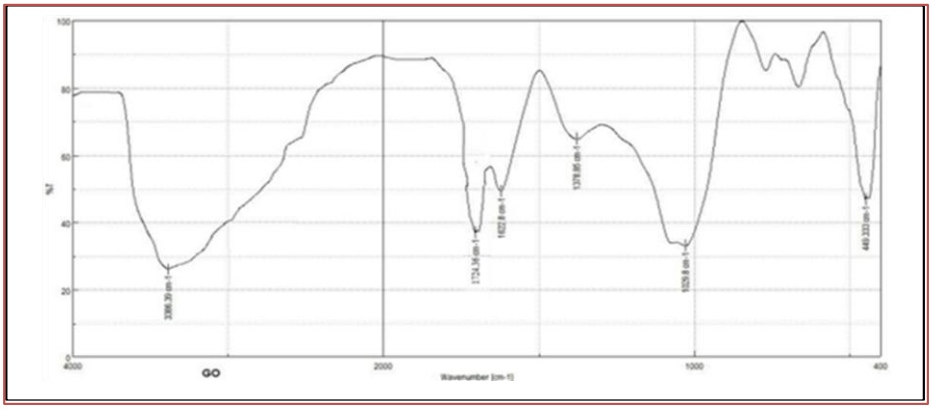


Fig1. FTIR for NGO

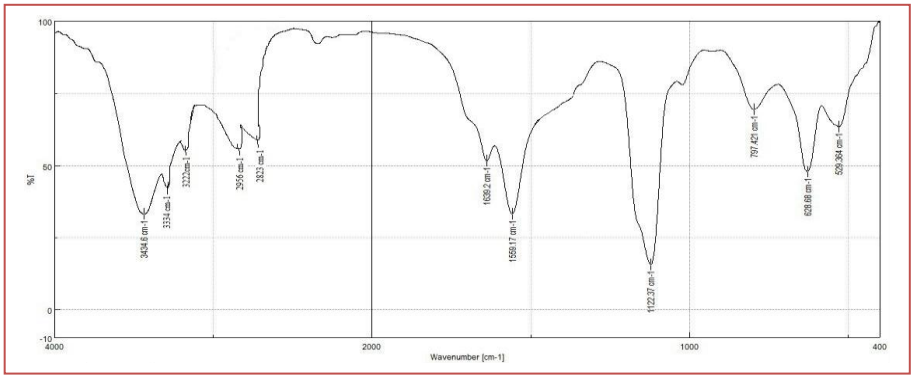


Fig2. FTIR for GOM

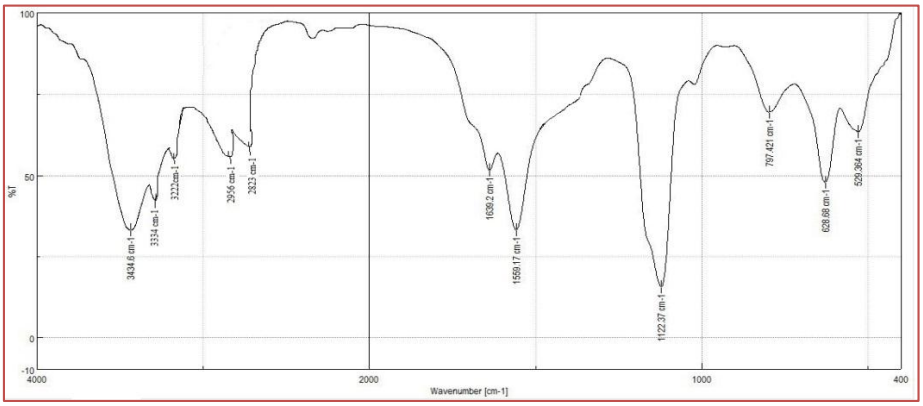


Fig3. FTIR for GOC

Table1. Values of 2θ and d- spacing for (GO) and (GP) compound

Compound	2θ (degree)	d-spacing (A^0)
GO	10.97°	8.06
GOM	34.02	2.63
	38.80	2.63
	48.86	1.86
	49.51	1.83
	54.73	1.67
	55.35	1.65
GOC	34.05	2.63
	38.80	2.32
	54.72	1.67
	55.35	1.65
	58.43	1.57
	62.15	1.49
	65.24	1.43

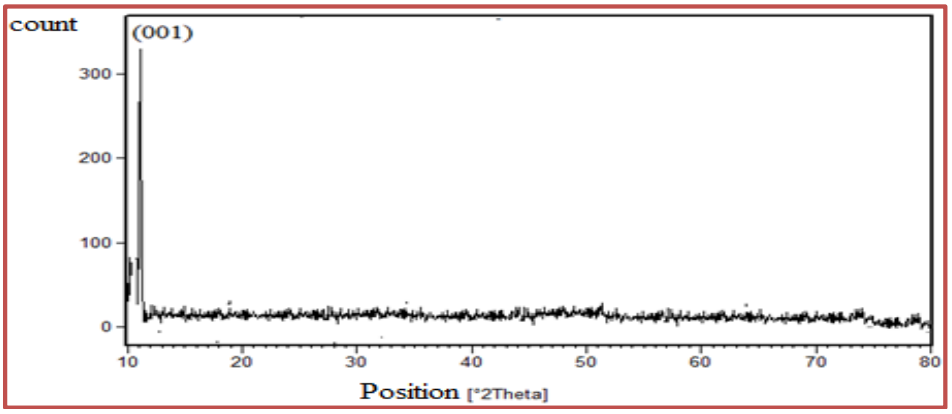


Fig4. XRD –Diffraction of (GO)

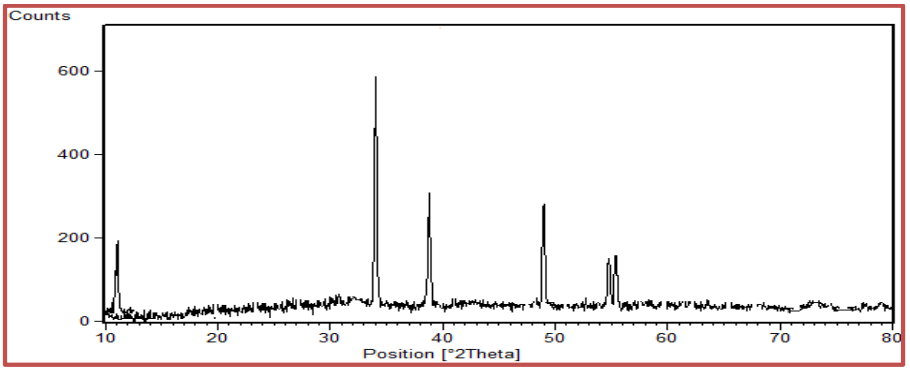


Fig5. XRD -Diffraction of (GOM)

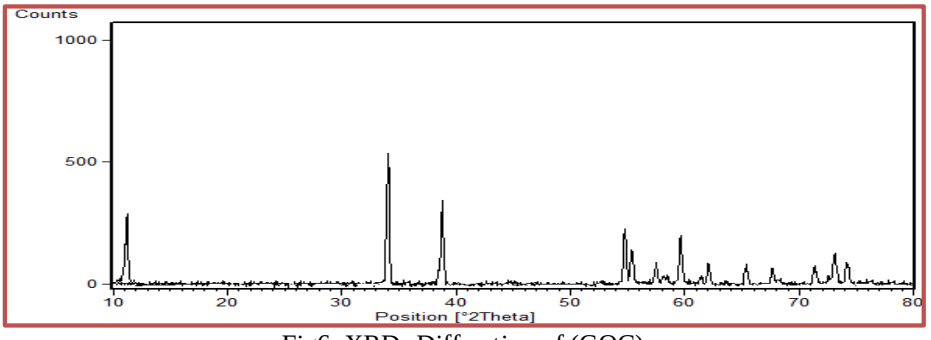


Fig6. XRD -Diffraction of (GOC)

Table2

Crystallite sizes and micro strain for GOM and GOC

Compound	Crystallite size	Micro strain
GOM	16.83	-0.00239
GOC	19.52	-0.00182

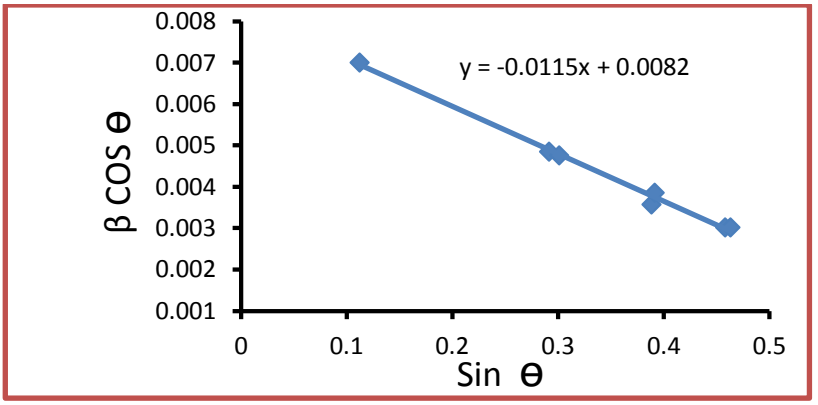


Fig7. (Williamson-hall) equation for GOM

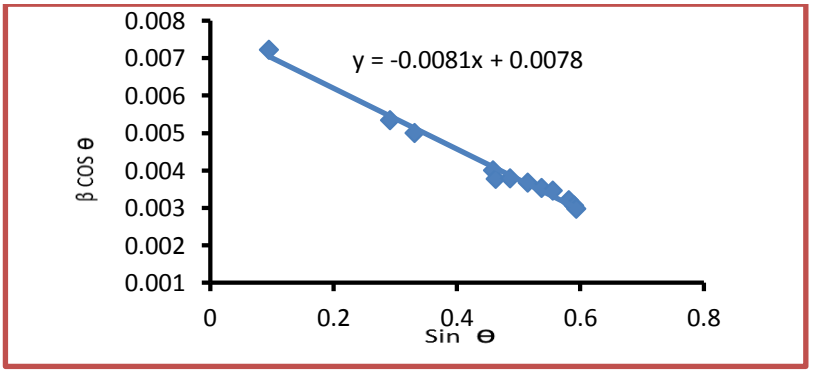


Fig8. (Williamson-hall) equation for GOC

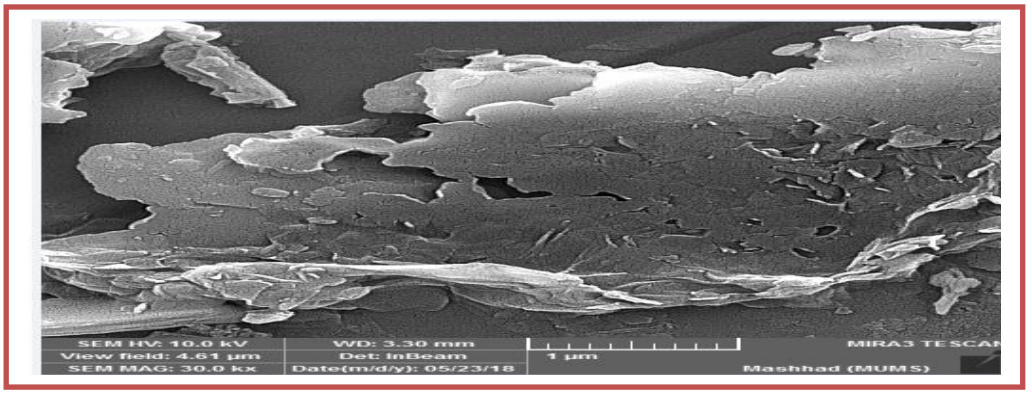


Fig9. FESEM for NGO

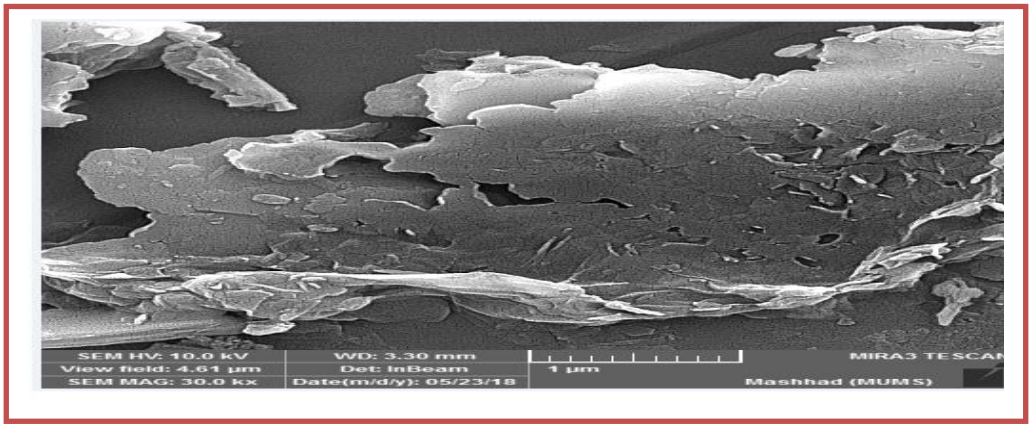


Fig10. FESEM for GOM

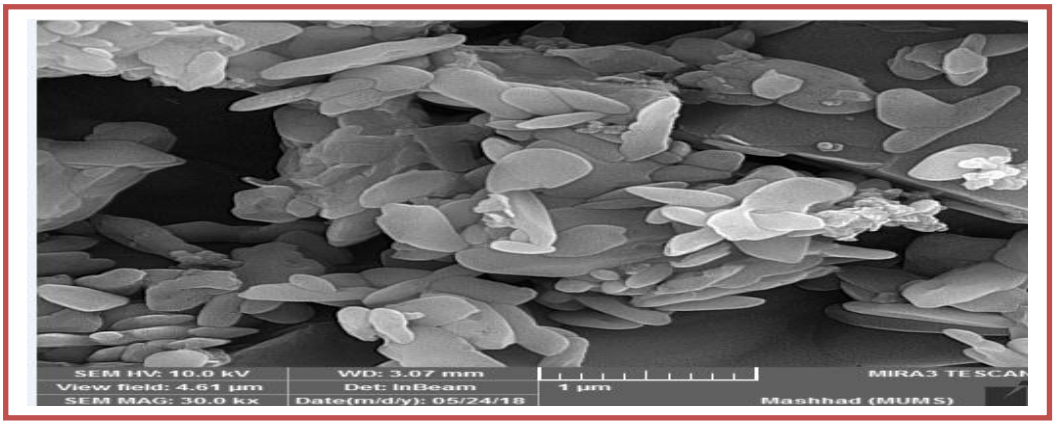


Fig11. FESEM for GOC

Conclusion

the reaction of graphene oxide with methionine (GOM) and cysteine with graphene oxide (GOC) by Microwave, the calculated crystalline size for the graphene oxide GO is (16nm), GOM (16.8nm) and GOC(19.52) depend to pattern of XRD – Diffraction by using (Debye – Scherer) and (Williamson – Hall) equation.

Acknowledgments

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