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# The Study of Functionalization Effect (poly aniline (PAni) and thiocarbohydrazide (TCH)) on Electrical Properties of Graphene Oxide Nanoparticles

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# <u>Abstract</u>

Graphene oxide which one of carbon nano structures: its single layer (SLGO) or a few layers (FLGO, no more than ten layers) of graphite, these sheets contain many functional groups, epoxy, carboxy, carbonyl and hydroxyl groups. Graphene oxide (FLGO), grapheme oxide-poly aniline and grapheme oxide-thiocarbohydrazide have been prepared and characterized by infra red spectra, X-ray diffraction and atomic force microscope. The addition of these materials by chemical reaction to the grapheme oxide nano particles have an important role in the electrical properties of this nanomaterial which is insulator, so graphene oxide was chosen in this research for studying the electrical properties of functionalized graphene oxide. Through LCR measurements we noticed increasing in the conductivity.

**Key words:** - Graphene oxide, nanoparticles, electrical properties, poly aniline, thiocarbo – hydrazide.

دراسة تأثير اضافة (البولي انلين و ثايو كاربو هيدرازايد) في الخواص الكهربائية لدقائق اوكسيد الجرافين النانوية

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# الخلاصة

يعد اوكسيد الجرافين احد تراكيب الكربون النانوية هو عبارة عن صفيحة واحدة (SLGO) او عدة صفائح من الجرافيت بحيث لا تتجاوز عشر صفائح بعضها فوق بعض ويسمى (FLGO) وتحتوي هذه الصفائح على مجاميع وظيفية مختلفة هي مجاميع الايبوكسي، الكربوكسيا، الكربونيل و الكحول. تم تحضير اوكسيد الجرافين النانوي بشكله متعدد الطبقات مجاميع الايبوكسي، الكربوكسيا، الكربونيل و الكحول. تم تحضير اوكسيد الجرافين النانوي بشكله متعدد الطبقات مجاميع الايبوكسي، الكربوكسيل، الكربونيل و الكحول. تم تحضير اوكسيد الجرافين النانوي بشكله متعدد الطبقات مجاميع الايبوكسي، الكربوكسيل، الكربونيل و الكحول. تم تحضير اوكسيد الجرافين النانوي المركبات النانوية المحضرة بالاضافة الى تحضير اوكسيد المرافين النانوي بشكله متعدد الطبقات المحضرة باستخدام طيف الأشعة تحت الحمراء، حيود الأشعة السينية و التصوير ألمجهري باستخدام مجهر القوى الذرية. ان اضافة هذه المجاميع الوظيفية الموجودة على الاوكسيد يلعب دورا مهما في تغيير ان اضافة هذه المجاميع الوظيفية الموجودة على الاوكسيد يلعب دورا مهما في تغيير الخواص الكهربائية المعروفة لهذا المركب النانوي والذي يمتاز بانه غير موصدل للاتيار الكهربائي; لذلك فقد تم اختياره في الخواص الكهربائية المعروفة لهذا المركب النانوي والذي يمتاز بانه غير موصدل للتيار الكهربائي; لذلك فقد تم اختياره في الخواص الكهربائية المعروفة لهذا المركب النانوي والذي يمتاز بانه غير موصدل للتيار الكهربائي; لذلك فقد تم اختياره في الخواص الكهربائية المعروفة لهذا المركب النانوي والذي يمتاز بانه غير موصدل للتيار الكهربائي; لذلك فقد تم اختياره في الخواص الكهربائية المعروفة لهذا المركب النانوي والذي يمتاز بانه غير موصدل للتيار الكهربائي; لذلك فقد تم اختياره في الخواص الكهربائية المعروفة لهذا المركب النانوي والذي يمتاز بانه غير موصدل للتيار الكهربائي; لذلك فقد م اختياره في الخواص الخواص الكهربائي; لذلك فقد تم اختياره في الخواص الكهربائية المعروفة لمحاميع (البولي انلين وثايو كاربو هيدراز ايد) على الخواص الكهربائية ولاحظنا من هذا البحث لدر اسدة تلاير اضرافة هذه المجاميع (البولي اناين وثايو كاربو هيدراز ايد) على الخواص الكهربائية ولاحظنا مال خلال قياسات كلورا الكهربائية لاوكسيا

الكلمات المفتاحية : أوكسيد الجرافين، الدقائق النانوية، الخواص الكهربائية، البولى انلين، ثايوكاربو هيدر از ايد.

# **Introduction**

The talent to design and prepare specific nano materials will make new future promise of nanotechnology. Designed Nano carbon materials were increasingly studied due to their excellent properties and numerous applications [1, 2]. Graphene oxide oxide which one of the important of carbon nano structures that extensively studied due to their high surface area / volume ratio and having many different functional group able to react with compounds to develop new nano materials with superior and different properties. There are many methods are known for the production of graphite oxide (GO). They all were based on the oxidation of graphite with oxidants in the presence of strong acids. HUMMER and OFFEMAN published 1958 Chlordioxid free method for the production of graphite oxide. [3]. Graphite is here oxidized with potassium permanganate (KMnO<sub>4</sub>) in a mixture of concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and sodium nitrate (NaNO<sub>3</sub>). The actual oxidant is Dimangan heptaoxid (Mn<sub>2</sub>O<sub>7</sub>), which was formed from KMnO4 in the presence of H<sub>2</sub>SO<sub>4</sub>. [4].

$$KMnO_4 + 3 H_2SO_4 \longrightarrow K^+ + MnO_3^+ + H_3O^+ + 3 HSO_4^-$$
$$MnO_3^+ + MnO_4^- \longrightarrow Mn_2O_7$$

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Chemical equation (1): pattern forming Dimangan heptaoxid of potassium permanganate and sulfuric acid. [4].BOEHM and SCHOLZ [5] characterized the obtained graphite oxides from the different production methods with respect to their chemical composition, specific surface and the layer separation [5]. Similar to graphite, GO has a staggered layer structure. Depending on the manufacturing method, the layer distance of graphite of 3.4 Å expands 6.0 - 13.2 Å [6, 7]. The exact chemical structure of GO is still controversial and the subject of intense discussions. The complexity of the structure and the amorphous character of the material complicate the accurate structure determination [4].

In the course of the investigation, there were several structural proposals, which are shown schematically in Figure (1).



Figure (1): Structural models for graphite oxide (GO) by HOFMANN,

Ruess, Scholz, and BOEHM and LERF and Klinowski [8].

Graphene oxide (GO) is characterized by the presence of functional groups that are amenable to further modification. For use as nano filler for plastics especially the covalent attachment of polymers to the graphene oxide surface of great interest. [8] Like improved dispersibility in organic solvents [9-11] increased compatibility of the filler with the polymer matrix is achieved [9-10]. There can be two approaches for surface modification of graphene follow with polymers, the so-called grafting-from and grafting-to method, as shown in Scheme 2. When grafting from polymerization is initiated starting from the graph [11].

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While the grafting process to an end-functionalized polymer is attached to the graph via a chemical reaction [12].





Therefore, provides often as an alternative surface modification, the grafting-to method that can be done in two different ways: (i) either the graph is provided with a corresponding functional group to react with a specific polymer may or (ii) the polymer is modified in order to react with the oxygen-containing groups of GO [8].

# **Experimental**

# 1 - Chemicals:

Carbon disulphide and Hydrogen peroxide were obtained from Sharlau ' potassium permanganate obtained from poison, Graphite from Merck, while aniline, Ammonium persulphate, hydrazine hydrate 80% and Sodium nitrate were obtained from HI-MEDIA, Sulphuric acid and Hydrochloric acid were available from Riedel-de Haen.



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#### 2. Instrumentation:

All infra red spectra of nanomaterials were recorded in (65 FT-IR Perkin Elmer Spectrophotometer). The prepared materials are characterized by x-ray diffraction using (Shemadzu – XR – 6000) device with Nickel - Cooper filter for the x-ray radiation (Cu K $\alpha$ ,  $\lambda$  = 1.5406 Å). Atomic Force Microscope (AFM) type PHYWE was used to study the surface of the nanoparticles. LCR Electronic Test Device is used to measure the Inductance (L), Capacitance (C), and Resistance (R) of a component. The electric properties are measured by using LCR (HEWLETT.PACKARD) device.

#### 3. Preparation of organic materials:

# **3.1** Preparation of Thiocarbohydrazide (TCH) :[14, 15]

5 ml carbon disulphide (CS<sub>2</sub>) was added in a 100 ml round bottom flask in the ice bath. 20 ml from hydrazine hydrate was added drop wise with stirring. This mixture was refluxed for 30 minutes, until yellow-white precipitate was formed. The yellow-white precipitate was washed in ethanol, recrystallized in distill water yet white crystals were formed, dried it in  $70^{\circ}$ C for 4 hours.



Chemical equation (2): Preparation of TCH

# 3.2 Synthesis of Polyaniline (PAni):-

A 50 ml beaker contains 3 ml of distillated aniline was placed in ice bath at 0 °C for 10 minutes then, 20 ml of 1M HCl was added drop wise and 20 ml of (2 g of Ammonium per sulphate (APS) dissolved in 20 ml of 1M HCl) was added drop wise with kept the temperature at 0 °C. After that the solution above stirring for 2 hours in an ice bath, then the solution was kept in the refrigerator overnight. The yield filtered and washed with distillated water 4 times and with



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ammonium hydroxide 1M, 20 ml with stirring for 30 minutes. Then filtered and washed with distillated water until the PH was neutral. Finally, the precipitate was washed with 15 ml of benzene with stirring for 15 min and dried at 80 °C for 6 hours.

#### 4. Syntheses of nanomaterials:

# 4.1. Synthesis of Graphene Oxide (GO):

Hummer method was used [16, 17] to oxidize the graphite's for the synthesis of GO as follow: Graphite 1gm, sodium nitrate1.5 gm and of sulfuric acid46 ml were mixed and strongly stirred at 0°C for 15 minutes in a 500 ml reaction flask immersed in ice bath. Then potassium permanganate 6gm was added slowly to the above solution and cooled for 30minutes. After this, the suspended solution was stirred continuously for 1 hour at 35°C, and water92 ml was added slowly to the suspension for 10 minutes. leave the solution on stirring for1hr. Subsequently, the suspension was diluted by warm water 280ml and stirring it for 4 hours. After then leaves the solution at room temperature, treated with  $H_2O_215$  ml (30%) to reduce residual permanganate to soluble manganese ions. Finally, the resulting suspension was filtered, washed with distilled water, and dried in a vacuum oven at 70°C for 24 hours to obtain GO



Scheme (2): Preparation of Graphene oxide (GO)



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#### 4.2 Thiocarbohydrazide (GO-TCH) [18]:-

0.5 g of graphene oxide was mixed with 1.0 gm of thiocarbohydrazide (TCH) in 25 ml Pyrex beaker. The Mixture was grinds and put it's in a sand bath with a temperature of 160 °C. This mixture was stirred by spatula until it will be melted. The new substance was washed with hot deionized water to remove the part which doesn't react TCH and dried at 70 °C for 4 hours.



Scheme (3): Preparation of Graphene Oxide Functionalized with TCH.

# 4.3 Synthesis of Graphene Oxide Functionalized with Polyaniline (GO-PAni) :-

1 g of Graphene oxide was sonicated with distillated aniline for 20 min and filtered and the collected was placed in a small beaker in ice bath at 0 °C. 20 ml of 1M HCl was added drop wise and 10 ml of (1 g of Ammonium per sulphate (APS) dissolved in 10 ml of 1M HCl) was added drop wise with kept the temperature at 0 °C. After that the solution above is stirring for two hours. In an ice bath, then the solution was kept in the refrigerator overnight.

The yield filtered and washed with distillated water 4 times and with 20 ml ammonium hydroxide 1M with stirring for 30 minutes, then filtered and washed with distillated water until the PH was neutral. Finally, the precipitate was washed with 15 ml of benzene with stirring for 15 minutes and dried at 80 °C for 6 hours.

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# **Results and Discussion**

The nano materials have been synthesized and characterized by (FTIR), (XRD) and (AFM). They are found to be air stable; in addition to good yield percentage.

3.1. FTIR Spectra:

3.1.1 FTIR of organic materials:

3.1.1.1 Thiocarbohydrazide (TCH):-

The IR spectrum of thiocarbohydrazide showed peaks at 1531,755 and 1490 cm<sup>-1</sup> which are associated for N-H wagging, bending and C-N is stretching vibration, respectively. The bands of the characteristic & (C=S) stretching were observed in the IR spectrum at 1286 and 933 cm<sup>-1</sup>. The band in 3305 is due to N-H stretching vibration, and the bands at 3274 and 3204 cm<sup>-1</sup> are due to NH<sub>2</sub> stretching vibrations. 1639 and 1142 cm<sup>-1</sup> bands are assigned to the NH<sub>2</sub> bending and wagging vibrations[16, 17].



Figure (2): FTIR Spectrum of TCH

3.1.1.3 Poly aniline (PANI):-

The peaks at 1586 and 1491 cm<sup>-1</sup> corresponding to C=C quinonoid and benzenoid deformation vibrations. 1293 and 1143 cm<sup>-1</sup> are assigned to the C-N of secondary aromatic amine stretching deformation and C=N stretching of (-N=quinoid=N-), respectively. While the band at 824 cm<sup>-1</sup> is attributed to C-H of aromatic ring. The stretching vibration of N-H shows a broad peak at 3388 cm<sup>-1</sup>[19].

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Figure (3): FTIR Spectrum Poly aniline (PAni)

3.1.2. FTIR of nano narticles and nano functionalized nanoparticles:-

3.1.2.1. FTIR of Graphene Oxide:-

The FTIR spectrum of GO Figure 4 shows a broad peak at 3406 cm<sup>-1</sup> of -OH in the high frequency area. The absorption peaks at 2942 cm<sup>-1</sup> and 2845 cm<sup>-1</sup> are represent the symmetric and anti-symmetric stretching vibrations of CH<sub>2</sub>. The peak of stretching C=O appears at 1718 cm<sup>-1</sup>. While the peak centered at 1622 cm<sup>-1</sup> is assigned to C=C bonds associated with skeletal vibrations of unoxidized graphite domains. 1381 cm<sup>-1</sup> is represented the vibrations c-o of carboxylic acid Finally, the absorption peaks at 1200 cm<sup>-1</sup> and 1128 cm<sup>-1</sup> are correspond to the stretching vibrations of C-O of epoxy and alkoxy groups [21-23].



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1587

188

1287

# Instrument model=WQF-510 resolution=4 scan times=10 2014/5/27 18:25 go.ASF Figure (4): FTIR Spectrum of Graphene Oxide

2487 2187 Wavenumbers / (cm-1)

3.1.2.2 FTIR of Functionalized Graphene Oxide with TCH:

278

3383

The infrared spectra of this compound shows decreasing the characteristic bands of graphene oxide such as a band 3447, 1328, 1019and 1720 cm<sup>-1</sup> which are assigned to the -O-H, C-O-H (OH bends) C-O-C of epoxy and C=O of (residual of carbonyl groups), and shows many new characteristic bands which attributed to the reaction of graphene oxide with thiocarbohydrazide. These diagnostic bands are; the double band at 3186, 3268 cm<sup>-1</sup> assigned to NH<sub>2</sub> stretching vibration, 1615 cm<sup>-1</sup> and 1497 cm<sup>-1</sup> assigned to NH<sub>2</sub> bend and C-N stretching vibration respectively, weak band at1552 cm<sup>-1</sup> assigned to N-N cm<sup>-1</sup> stretching vibration.







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3.1.2.3 Graphene oxide- poly aniline composite (GO- PANI):-

All character bands of PANI chains are observed in GO- PANI composite but the PANI bands are slightly shifted to lower frequency this case indicating the stacking and hydrogen bonding between GO nanosheets and the PANI backbone and hydrogen bonding between GO nanosheets and the PANI backbone. These peaks are 1574 and 1488 cm<sup>-1</sup> corresponding to C=C quinonoid and benzenoid stretching vibrations. 1297 and 1135 cm<sup>-1</sup> are assigned to the C-N of 2<sup>0</sup> aromatic amine stretching deformation and C=N stretching of (-N=quinoid=N-), respectively. 823 cm<sup>-1</sup> band is attributed to C-H aromatic ring. The stretching vibration of N-H shows a broad peak at 3196 cm<sup>-1</sup>. All these bands are clearly indicates the presence and formation of GO-PANI [24].





#### 3.2. X-Ray Diffraction Characterization (XRD)

3.2.1-X-Ray Diffraction of Graphene oxide :

X-ray diffraction pattern (figure 7) of GO powder shows a large interlayer spacing equal to  $8.12A^{\circ}$  at the position  $10.8^{\circ} 2\theta$  and other two bands at  $(2\theta = 23.95^{\circ})$  and peak at  $(2\theta = 43.79^{\circ})$ ) attributed to the intermediate layer.



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Figure (7): XRD of Graphene oxide

# 3.2.2 XRD pattern of poly aniline (PAni):-

The x-ray diffraction pattern of PAni powder exhibit a narrow peaks than that found in nanoparticles. XRD of pure PAni shown in (figure 8). The mainly peaks appeared at 19.8, 20.9 and 25.2° 20. The peak centered at may be ascribed to the repetition of benzenoid and quinoid rings in PAni chains and the peak at  $2\theta = \sim 25^{\circ}$  may be caused by the periodicity perpendicular to the polymer chain, while the peak at  $2\theta = \sim 20^{\circ}$  also represents the typical distance between the ring planes of benzene rings in nearby chains or the close-contact inter-chain distance [19].



Figure (8): XRD of poly aniline



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3.2.3 XRD of Graphene Oxide functionalized poly aniline (GO-PAni):-

Three new broad peaks of graphene oxide functionalized with poly aniline figure (9) centered at  $2\theta = 8.3^{\circ}$  and broad intense peak at 25.6° and around 43.1° correspond to (001), (002) and (100). Crystal planes are almost the same as that of pure poly aniline as indicate by Kumar, which are also the characteristic Bragg diffraction peaks of the polymer. Interestingly, for the GO-functionalized with poly aniline, we observed a weak and broad peak appearing nearly at  $2\theta = 8.3$ , which is lower than that of graphene oxide. This could imply that the interplanar spacing of the graphene oxide functionalized with poly aniline and that the graphene oxide was fully exfoliated by treatment with poly aniline. Therefore, the XRD patterns confirm also the formation of poly aniline grafting on the surfaces of the graphene oxide. These results provide further insight and clear evidence for the formation of functionalized graphene with poly aniline from graphene oxide during the process [25].



Figure (9): XRD of Graphene Oxide Functionalized with Polyaniline

# 3.2.4 XRD of Thiocarbohydrazide TCH:-

The x-ray diffraction pattern of thiocarbohydrazide exhibit major three peaks at 29.5, 15.84 and 72.98 and show an inner layer spacing approximately 3.02, 5.58 and 1.29 respectively without impurity peaks. This is indicating formation of pure TCH.



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Figure (10): XRD of Thiocarbohydrazide

3.2.5 XRD of Graphene Oxide Functionalized Thiocarbohydrazide (GO-TCH):-

The XRD pattern figure (11) shows new three peaks at  $2 \square \square \square 26.5$ , 25.5 and 24.7 these bands corresponding to the chemically converted graphene oxide into GO-TCH and the chemically reduction of GO was occurred during the functionalization process that mean TCH behave as reducing agent. Additionally d-spacing has been decreased. When comparing with GO and TCH, so this indicating TCH has been reacted and grafted in the edges and the surface of GO.





It is important technique to study the topography of the materials in nanoscale and to know the morphology and surfaces for the nanoparticles and polymer nanocomposite.

AFM investigation of Pure and functional GO figure 12 was represented the graphene oxide



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with different functional Groups. All AFM measurements was shown the graphene oxide sheets with thickness about 4-5 nm. With the exception the sheets which doped with poly aniline. These values were indicated that the prepared sheets in three layer one above another. These sheets were possess a wide area and help to keeping a very large amount of charges.



Figure (12): AFM image of (A) graphene oxide, (B) graphene oxide functionalized with thiocarbohydrazide and (C) graphene oxide functionalized with poly aniline

3.4 Electrical properties of pure nanomaterials and its composite: -

3.4.1 The real and imaginary permittivity of pure nanomaterials and its composite:

Figures (13-15) represent the variation of permittivity with respect to frequency, for the graphene oxide alone or with functional group composites systems.

All figures (13-15) were depict the variation of the real part of dielectric permittivity ( $\epsilon$ ') with frequency for all types of pure nanosheets.

At low frequencies (400-1000 Hz) permittivity, attained higher values, in all cases, then



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diminish rapidly with increasing of frequencies (4000-100000 Hz). This is reasonable sense – in the low frequency region the alternation of the field is slow, subsequently providing sufficient time to permanent and induced dipoles enlargements according to the applied filed lading to enhanced polarization. The pure nanosheets exhibited high values. On the other hand, pronounced. Enhanced values ( $\epsilon'$ ), especially at low frequency, can be attributed to increased conductivity, and / or interfacial polarization, and / or electrode polarization. Electrode polarization is related to the buildup of spice charges at the specimen / electrode interfaces and is characterized by very high values of both real and imaginary part of dielectric permittivity [26,27]. Interfacial Polarization (IP) is a result from the accumulation of unbounded charges of the interfaces of the constituents from long dipoles. This gives indirect evidences of the achieved distribution of nano inclusions. So, the higher value of ( $\epsilon'$ ) could be attributed to enhanced conductivity and Interfacial Polarization (IP) [28].



Figure (13): The relation between real, imaginary & ', & " permittivity with the frequency change values of pure graphene oxide (GO)









Figure (15): The relation between real, imaginary & ', & " permittivity with the frequency change values of graphene oxide-poly aniline (GO-PAni)

3.4.2 The conductivity of nanomaterials and its composites

Figures (16, 17) of conductivity were show the variation of results for the AC conductivity (AC) at different frequencies. The conductivity appears to be frequency dependent. This is considered as strong indication for charge migration via the whopping mechanism [29, 30]. Graphene oxide functionalized with polyaniline exhibited relatively higher conductivity values,



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when comparing with the other systems. Finally, it should be stated that in all studied specimens, conductivity was altered abruptly, implying that the transition from insulating to conductive behavior has been achieved by graphene oxide functionalized poly aniline and thiocarbohydrazide in the examined composites. These two composites were possessed a highly  $\varepsilon'$  and ( $G_{AC}$ ) with low  $\varepsilon''$ . As shown in (table 2).







Figure (17): The relation between conductivity  $\sigma_{AC}$  with the frequency change values of graphene oxide-thiocarbohydrazide (GO-TCH) and graphene oxide-poly aniline (GO-PAni)

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# **Conclusion**

As known graphene oxide is an insulator material and the challenges lie in the use of such materials and how to develop and increase the electrical characteristics. Because of graphene oxide has a high area / volume ratio and contain many different functional groups, we used it to study how we could increase the conductivity and charge saving. Through this study we noticed that the addition of polyaniline and thiocarbohydrazide increases the conductivity and real permittivity but reduce the imaginary permittivity, therefore it is possible to use this nanomaterials as fillers to prepare nano-composites which it can be used in the field of energy storage. Also we noticed graphene oxide– poly aniline (GO-PAni) has upper conductivity than graphene oxide (GO) and graphene oxide-thiocarbohydrazide (GO-TCH). The conjugated double bonds in poly aniline aromatic rings allow the (GO-PAni) molecule to be conductive and absorb the energy.

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