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Abstract

Purpose: This research, some derivatives were Synthesis and Characterization of Some 1,2,4-triazol-4-amino Schiff Bases of Decorated Nano Reduced Graphene Oxide with 2-naphthol-4-sulfonic acid .

Methodology: In by the hummer method preparing the graphene oxide (GO) and then reducing it to the reduced graphene oxide (RGO), Then prepare the corresponding and decorated derivative with 2-naphthol-4-sulfonic acid, Using the electric method (ArNRGO) in two steps, first one preparing the disonium salts and the second, electrical decoration, Prepare the corresponding Triazole derivative (ArNRGOTZ) by thermal fusion for the Ar-NRGO Aromatics Derivative with Thiocarbozide (TC), Also, ArNRGOTZS (3-1) nanoschiff bases prepared by heat mixing between ArNRGOTZ with 4-Nitrobnzaldehyde, 4-Chlorobenzaldehyde and 4-Methoxybenzaldehyde,

Findings: The data characterization by IR, XRD and SEM infrared spectroscopy with stability evaluation from TGA, DSC and DTG measurements.

Unique Contribution to Theory ,Practice, and Policy: Formation of 1,2,4-triazol-4-amino Schiff Bases of Decorated Nano Reduced Graphene Oxide with 2-naphthol-4-sulfonic acid as non-toxic compounds To benefit from them in the future as chemical compounds that transport drugs inside the living cell.

Keywords: *Graphene Oxide, 1,2,4-Triazole, 1,2,4-Triazol-4-Amino Schiff Bases*

INTRODUCTION

Graphene (nanoparticles), which are plane sheets, the thickness of these plates is the diameter of one carbon atom[1], and the hybridization process between carbon atoms is in the formula sp^2. The process of sp^2hybridization is the fusion of electrons present in two neighboring carbon atoms and the formation of a bond between them. Natural graphite is the basic material for preparing graphene oxide (GO) and the most common source for it[2] . The reason is that natural graphite contains many local defects in its structure that may serve as starting points for chemical reactions [3]. Nanofibers are of great importance in industrial applications [4]. Nanocomposites are created by incorporating nanoparticles into the manufacturing process of materials leading to enhancement, in their properties [5].

Triazoles are heterocyclic aromatic compounds of molecular formula $C_2H_3N_3$, It was named by this name for the first time by the scientist Potts[6] and its name includes the unsaturated position in its 1H and 4H and the number of tri nitrogen atoms and it is of two types depending on the bonding of nitrogen atoms in it: the identical triazoles, which are compounds in which the nitrogen atoms are not all related to each other, Where the two carbon atoms separate them and are present in two isomer forms due to the presence of the state of Totomerism[7,8]. Equatoriality can be observed in the steric form of triazoles contiguous triazoles,[9] which are compounds in which the three nitrogen atoms are connected. Among the applications of -4,2,1 triazole compounds, carboxylic acid compensators were used in the preparation of triazole, as they were used to obtain chelated polymers that were used as anti-cancer treatment,[10] anti-inflammatory[11] and harmful insecticide[12]. Some of its derivatives also showed effectiveness against Seven types are cancer of the blood, lung, colon, skin, ovaries, kidneys, and central nervous system, as well as sometimes used as anti-bacterial and anti-fungal agents[13,14], as well as stimulating brain cells to produce hemoglobin, as well as used as anesthesia, sedatives for cancer and anti-seizures[15]

METHODOLOGY

(Preparation of Nanoparticle Graphene) The Hummer method was laboring to oxidize graphite for synthesizing graphene oxide (GO) in the following manner Graphite: A 1 gm of sodium nitrate and 1.5 gm of sulfuric corrosive was acquainted with a 500 ml response jar. The response jar was set in a 0˚C ice shower for 15 minutes with consistent tumult. The above blend was additionally cooled for 30 minutes with 6 g of potassium permanganate. In this manner, the 4 mL suspension was weakened with 46 mL of water thus until the arrangement arrived at a volume of ten minutes, and the temperature was raised to 98 ˚C. Graphite suspensoid arrangement on warming was continually blended for two hours at 35˚C. The process was left with blending for 20 minutes. Along these lines, the suspension was debilitated with warm water 140ml and blended for 10 minutes. Starting there .

Forward, the course of action was held at room temperature, and treated with H_2O_2 15 ml (30%) to diminish remaining permanganate to dissolvable manganese particles. At long last, the resulting suspension was isolated by centrifugation, washed with 10% HCL and refined water, and dried in a vacuum oven at 70°C for 24 hours to get(GO)[16]. (100mg)(Graphene oxide) was dissipated in (1mL)HCl 37%. Then, at that point (1 mL) of hydrazine hydrate(80%) was added, and the blend was warmed at 95 ∘ C for 2 h. Then RGO was accumulated by filtration. The moved thing was wash away with water a couple of times to wipe out the excess hydrazine and was dried in a vacuum oven at 100 ∘ C for 12 h. [17]

(Preparation of Reduced Graphene Oxide-Aryl) * RGO-Ar. (1-Amino-2-naphthol-4-sulfonic acid) 0.7 0g. put in a container containing acidic arrangement its proportions of 1:1 water and HCl(37%) and in another breaker dissolve 0.001mol from $NaNO_2$ With a quantity of distilled water it was gradually added to the first step solution while maintaining the temperature of less than 5∘C in the dark remains on stirring for half an hour.[18] On the same solution connected to an electric cell with platinum electrodes[24] for both anode and cathode (10 V,1A) add a homogeneous solution of graphene

0.1g. after 24h separated by the centrifuge washed three times with deionized water and dried. scheme (1)

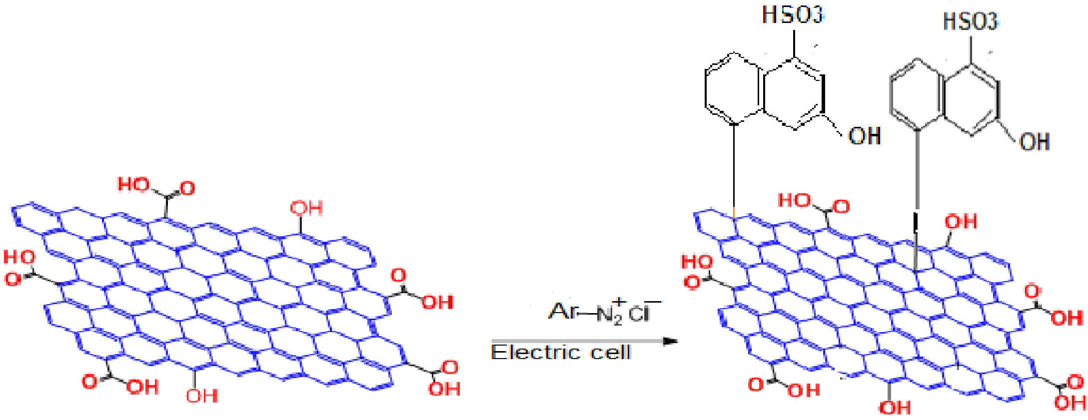

Figure 1: Scheme (1)

(Preparation of Triazole Derivatives (ArNRGOTZ)) Triazole derivatives (ArNRGOTZ) were prepared by thermal fusion of 0.1 g of (Ar-NRGO) with 0.3 g of thiocarbohydrazide (TCH) in a heated flask, heat the mixture in an oil bath until the color and nature changed[20] as shown in Scheme(2)

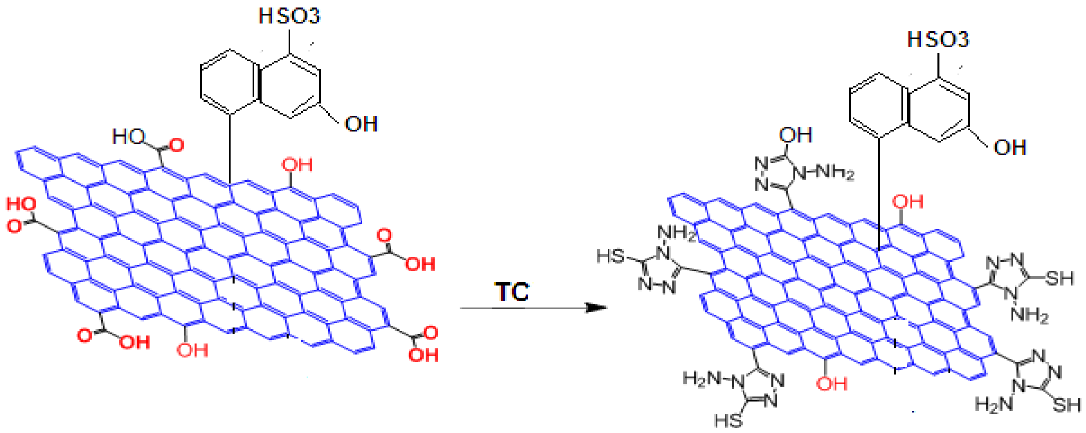

Figure 2: Scheme (2)

(Preparation of nano Schiff bases (ArNRGOTZS) by thermal fusion) The nano Schiff bases (ArNRGOTZS) were prepared by thermal fusion of 0.1 g (ArNRGOTZ) with (0.3 g of 4-Nitrobnzaldehyde or 0.3 g of 4-Chlorobenzaldehyde or 0.3 g of 4-Methoxybenzaldehyde) in a flask. Heat the mixture in an oil bath,(5-10) minutes until the color and nature of the melt change [21] as shown in Scheme(3)

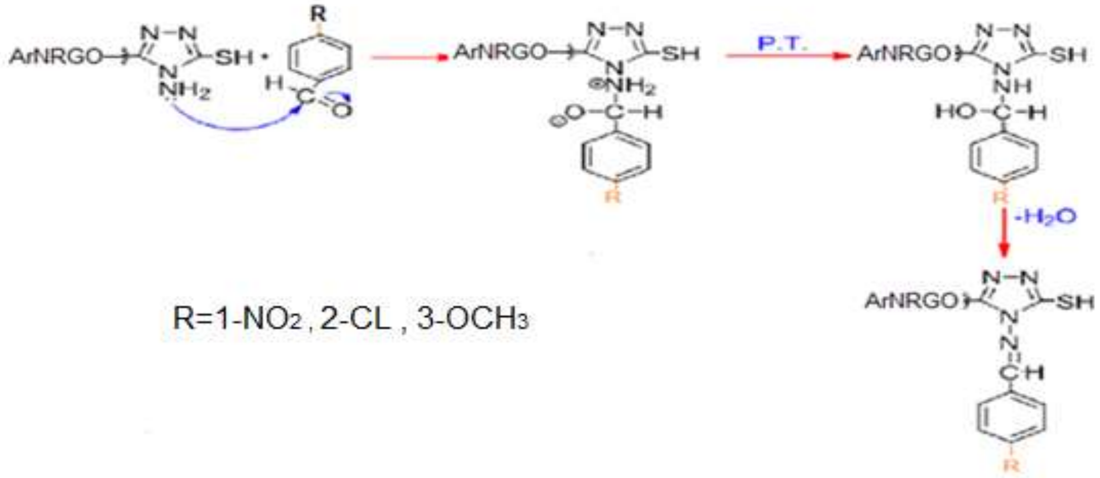

Figure 3: Scheme (3)

RESULTS AND DISCUSSION

The functional groups of GO were analyzed using FT-IR, as seen in Fig. (1). The FT-IR range shows major areas of strength for an expansive band addressing the extending vibrations of alcoholic and carboxylic Goodness at 3434 cm-1. Peaks at 3434, 1221, 953, and 1053 cm-1 correspond to asy, sy ν (OH) stretching, C-O-C extending for epoxy groups, and C-O extending for alkoxy groups. The pinnacle focused at 1622 cm $^{-1}$ is relegated to ν C=C bonds which are related with skeletal vibrations of unoxidized graphite spaces and 1730 cm $^{-1}$ shows the presence ν C=O of carboxylic corrosive gatherings on GO sheets. Fig (2) a X-beam, the peak centered at 1622 cm -1 is assigned to ν C=C bonds which are associated with skeletal vibrations of unoxidized graphite domains and 1730 cm $^{-1}$ indicates the presence ν C=O of carboxylic acid groups on GO sheets. Fig (2) an X-ray, which provided the pattern of out a powder sample of a substance X. The peak was observed at 11.84° and 0.74 nm represents the d-spacing. 25 which is more than that of Graphite 3D5 cubic unit cell (0.33 nm. Fig (3) spot the present functional groups i.e., carboxylic corrosive, carbonyl, hydroxyl, and epoxy on the basal and edges planes of (GO) sheets[22], and the expansion in d-separating subsequently affirms this oxidation of graphite pieces 9 It is realized that the level of oxidation is corresponding to the interlayer separating (d) of GO 85, The Debye−Scherrer 36 equation was utilized to determine the thickness of GO sheets[23,26,,27]. Where k is the Scherrer-constant is reliant of the state of the gem and the size appropriation (shape factor). β is the precise stacked width at half greatest pinnacle force in radian[24,25], 2θ of (λ = 0.1541 nm) of Cu Kα radiation source, and θ hkl of Bragg point in degree. The diffractograms were record in the scope of 10-80°. The thickness was lay out to be 5.34 nm. The quantity of layers stacked n can be esteemed by:

$$n = t/d$$

As for the interlayer spacing, denoted as 'd,' revealed the presence of seven stacked graphene oxide (GO) sheets perpendicular to each other.

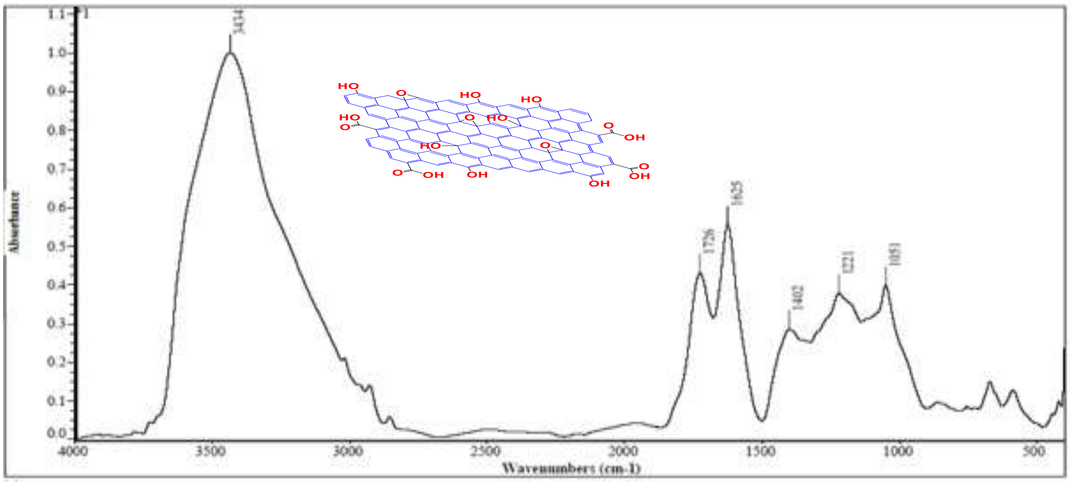

Figure 4: FT-IR for (GO)

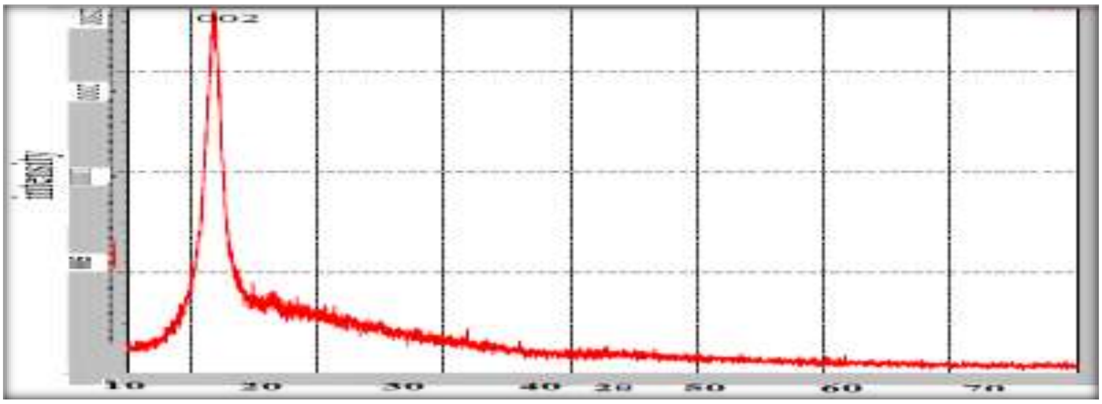

Figure5: XRD of (GO)

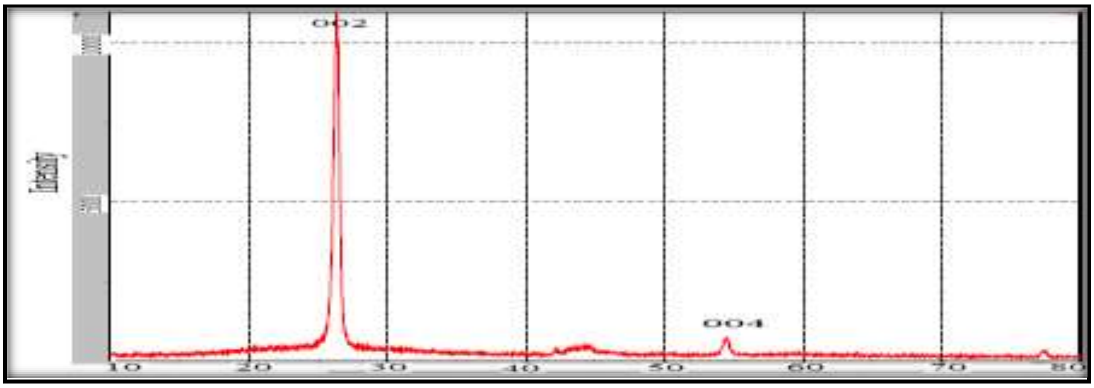

Figure 6: XRD Patterns of Graphit

The morphology of the GO (Fig.4 a,b) was investigated by scanning electron microscopy (SEM). It was observed that particle size reduction on GO.

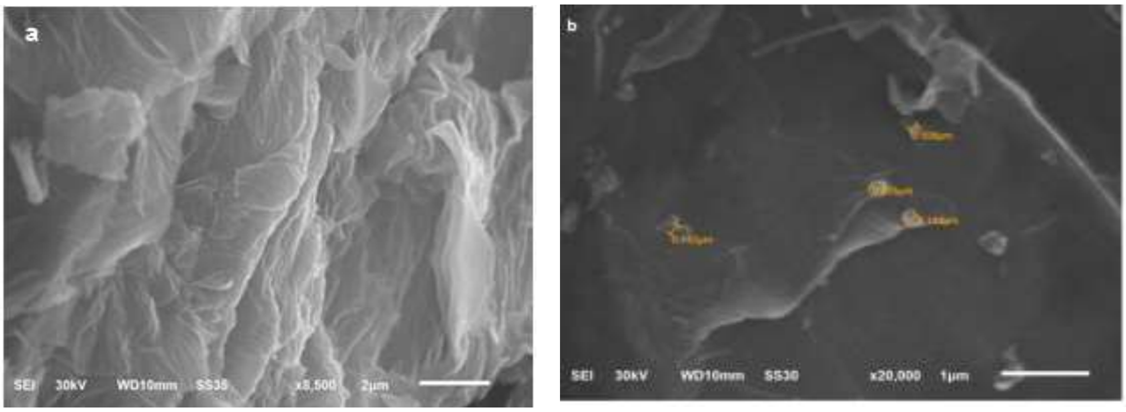

Figure 7: (a,b)SEM Image of GO

The reduced graphene oxide (RGO) FTIR spectrum is shown in Fig (5). XRD-patterns of (RGO) Fig. (6) shows a wide diffraction top showed up at 2θ 24.63° connoting unfortunate requesting of the sheets along the stacking course, which recommends the example was incorporated basically of a couple of layers of(RGO), lined up with (002) level surface of RGO individually, with an interlayer d-separating of 0.36 nm which is lesser than GO because of the cancellation of most oxygen useful gatherings. The interlayer d-spacing of RGO was a little greater than graphite (GT) may be recognized to the left over some of the oxygen functional group, mentioning to the partial reduction of(GTO) nanoplates by hydrazine, the presence of this peak and the disappearance of the diffraction of GO (2 theta 11.8°) suggesting that the RGO nanosheets were formed[21,22]. The thickness or height and the number of layers stacked (n) were as well valued and found to be 1.89 nm and 5 respectively. This number of stacked layers referred to graphene nanoplatlets which have unique attributes. The morphology of the RGO Fig (7) It shows the efficiency of oxidation and peeling vision of the large interface between the plates which is estimated to be more than 200 n.m with folds7.98n.m and 51.87n.m with the presence of oxidation on the plates (inter-depth 50-100 n.m)

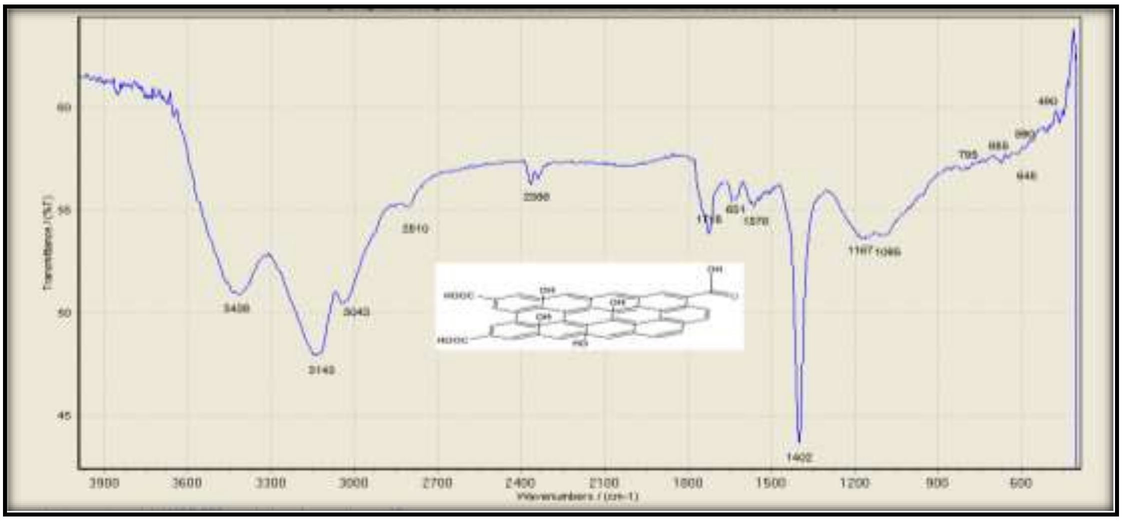

Figure 8: FT-IR of (RGO)

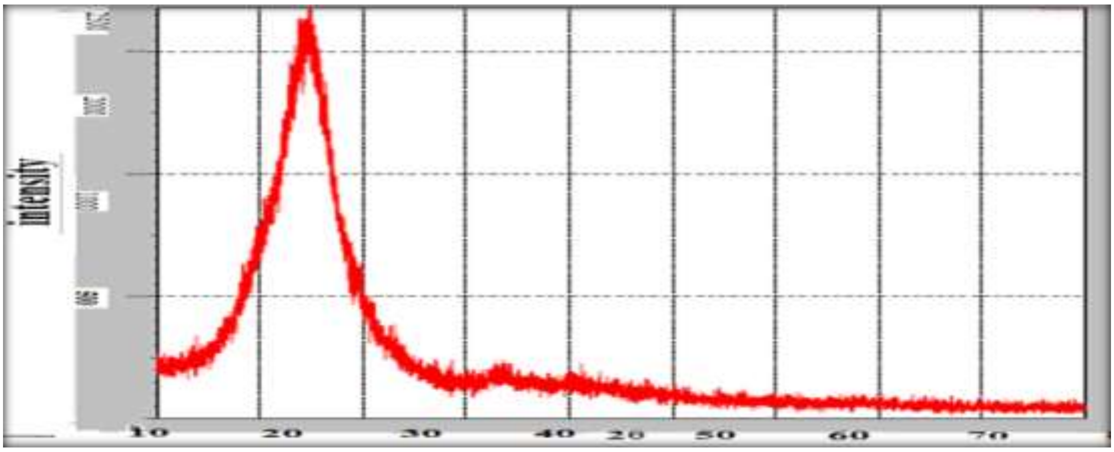

Figure 9: (RGO)s XRD

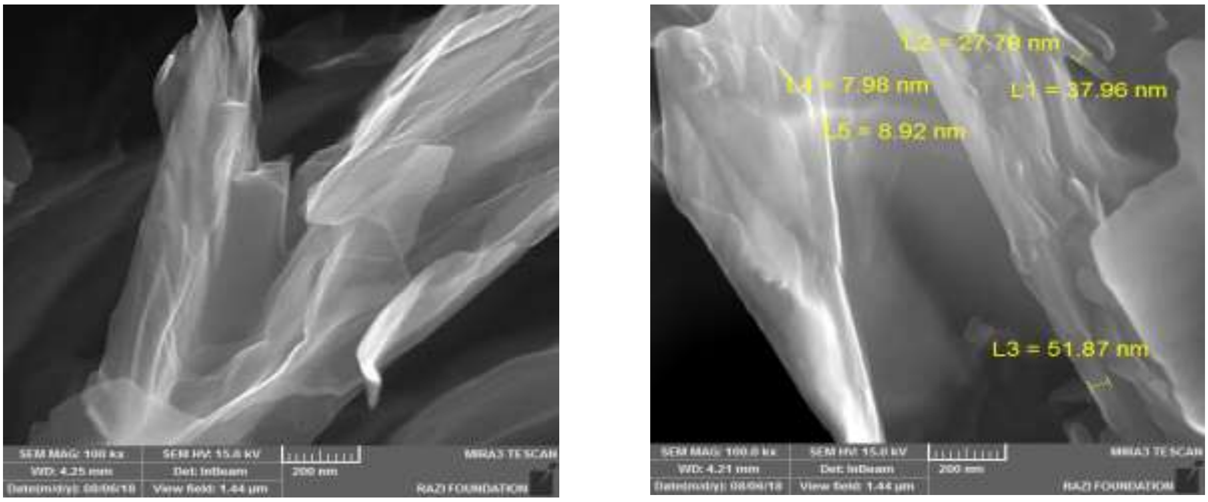

Figure 10: (RGO)s SEM

Fig (11) shows FT-IR spectrum of (ArNRGO), Fig (9) XRD patterns of (ArNRGO), and Fig(13) shows the SEM image of (ArNRGO), and (ArNRGOTZ) compound shows the FT-IR spectrum in Fig (14), Fig (15) XRD patterns and SEM image in Fig(16).

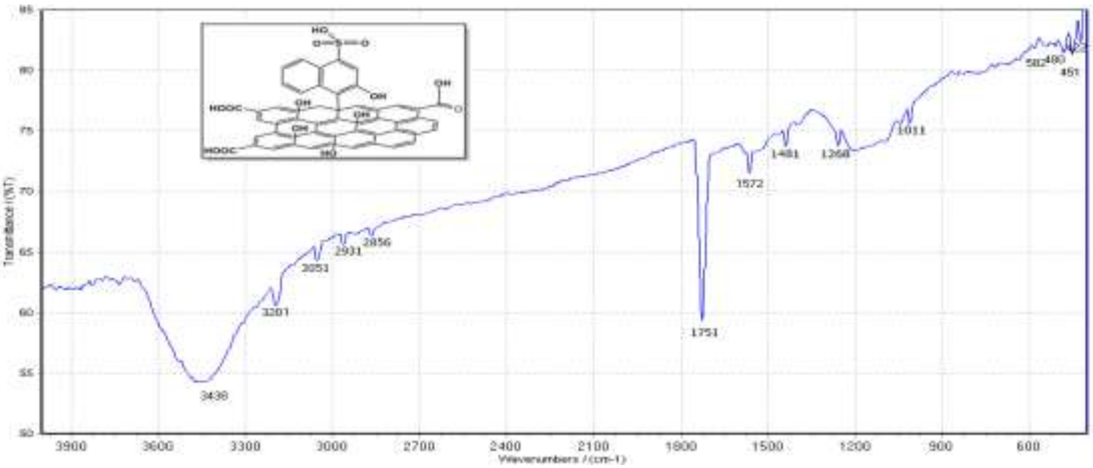

Figure 11: (ArNRGO)s FT-IR Spectrum

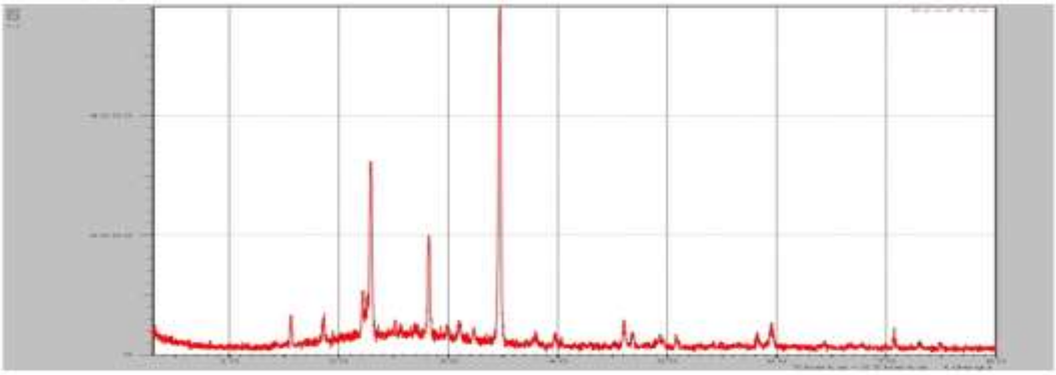

Figure 12: (ArNRGO)s XRD

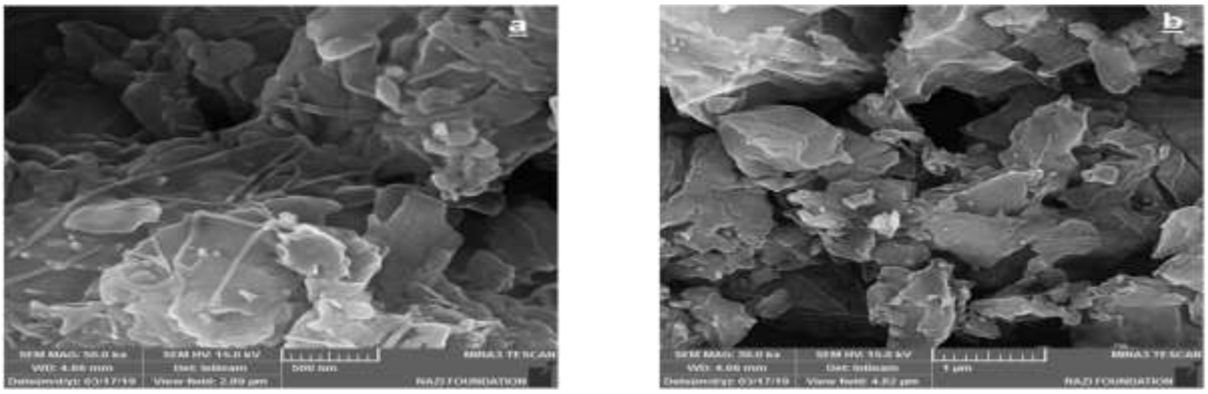

Figure 13: SEM Image of (ArNRGOTZ)

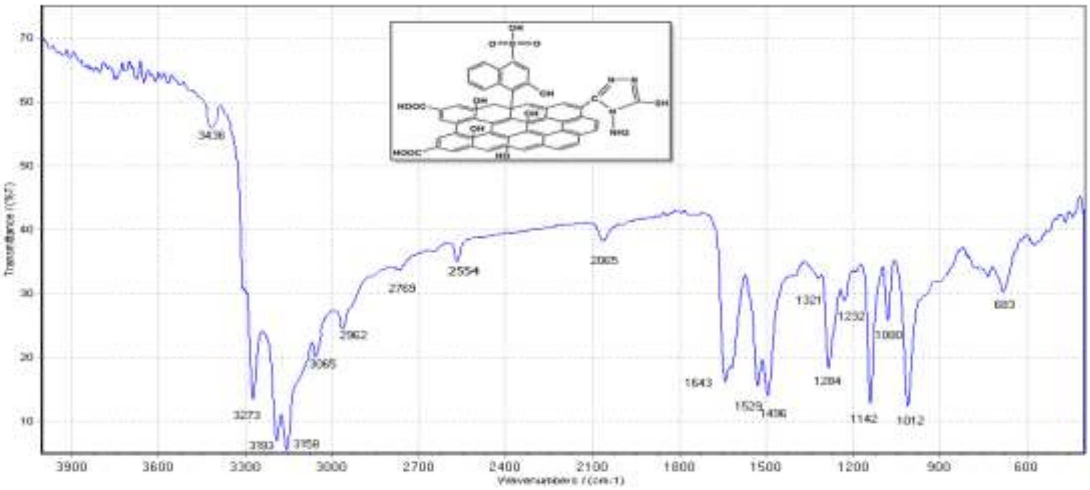

Figure 14: FT-IR Spectrum of (ArNRGOTZ)

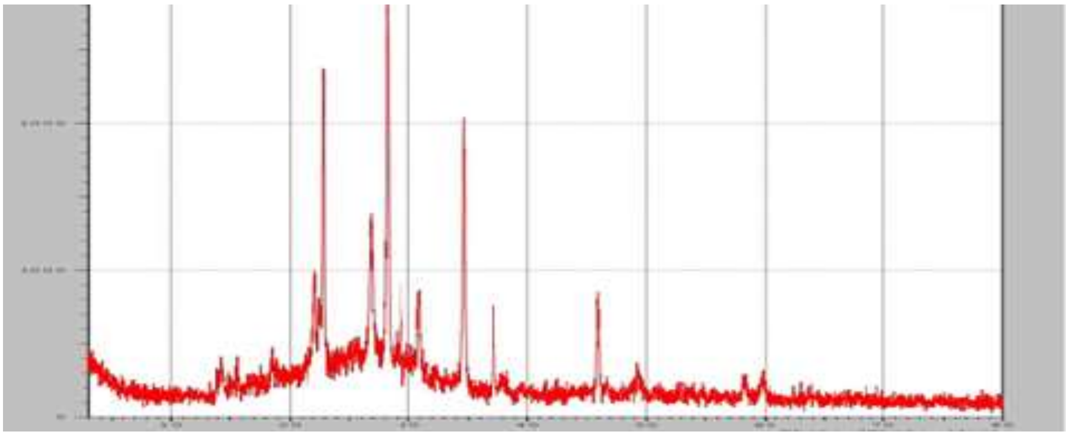

Figure 15: XRD Pattrens of (ArNRGOTZ)

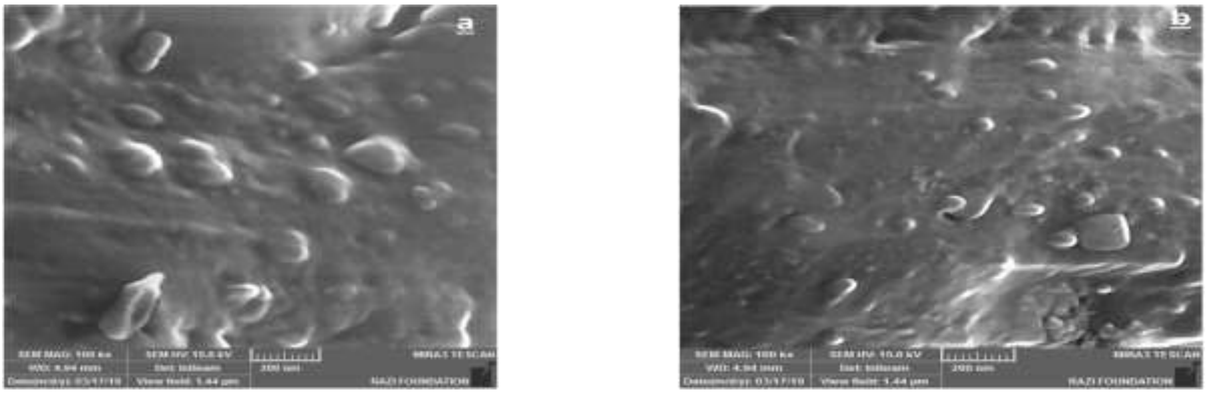

Figure 16: SEM Image of (ArNRGOTZ)

The(TGA, DTG ,DSC) measurements for (ArNRGOTZ) compound in Figure (14) had a weight loss of 5.33% at 130.6°C and the continued loss at 202.4°C, increased by 4.62% at 226.9°C, while DSC showed peaks at (214.3, 171.2) °C as an exothermic reaction.

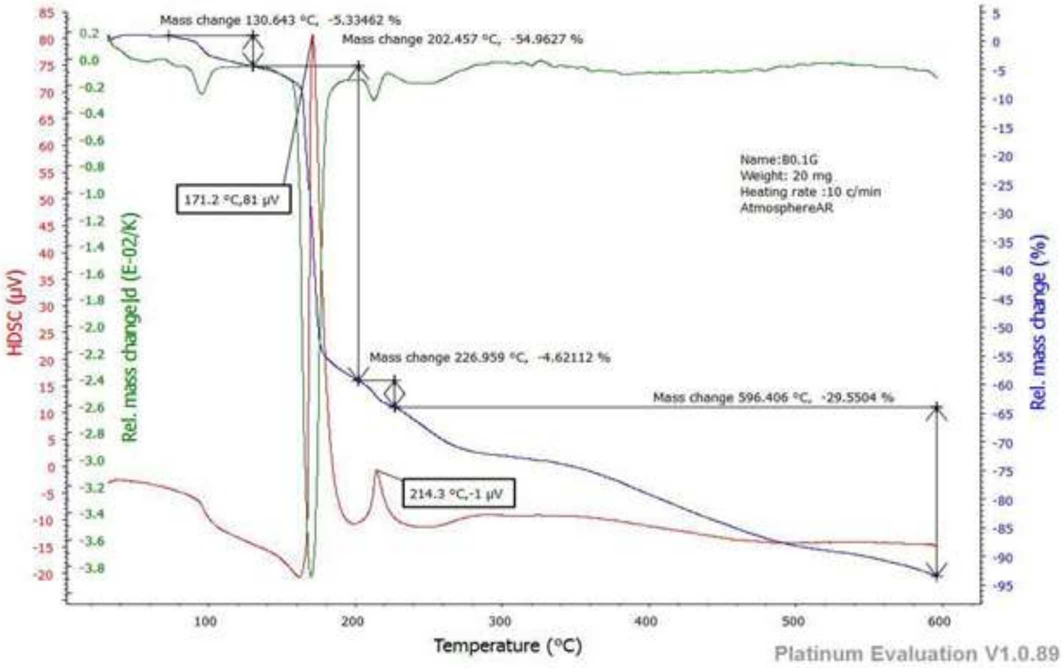

Figure 17: The TGA, DTG and DSC Curves of (ArNRGOTZ)

Fig (18,19,20) shows the FT-IR spectrum of (ArNRGOTZS$_{1-3}$), Fig(21,22,23) XRD patterns of(ArNRGOTZS$_{1-3}$) and Figure(24,25,26) shows the SEM image of(ArNRGOTZS$_{1-3}$)

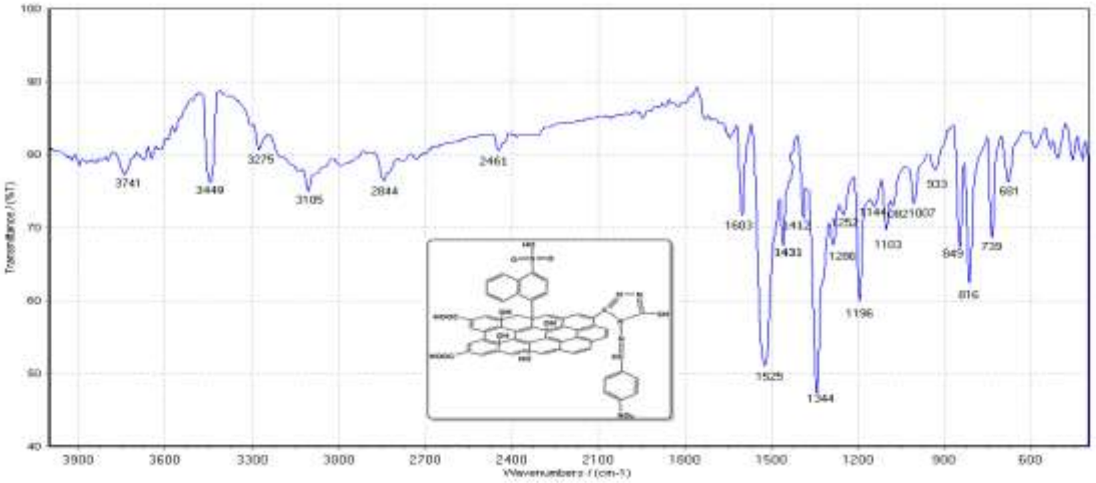

Figure 18: (ArNRGOTZS$_1$)s FT-IR Spectrum

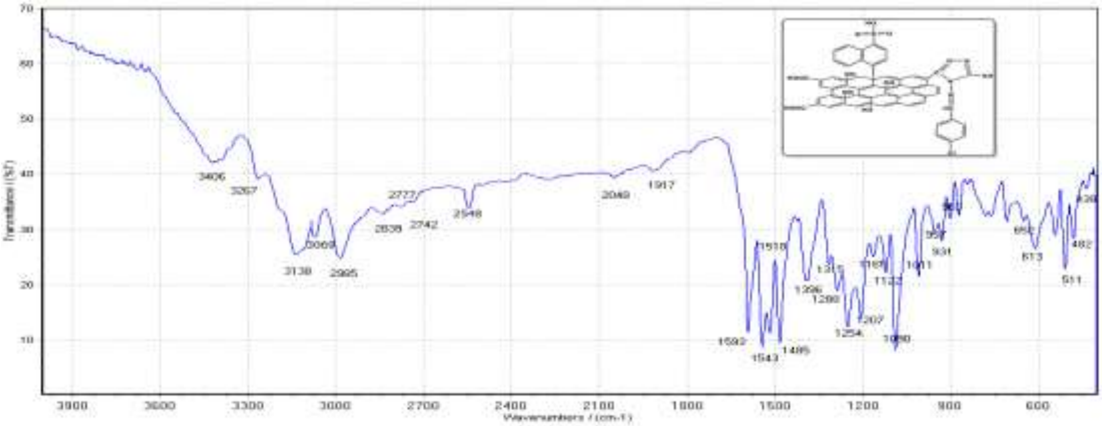

Figure 19: (ArNRGOTZS$_2$)s FT-IR Spectrum

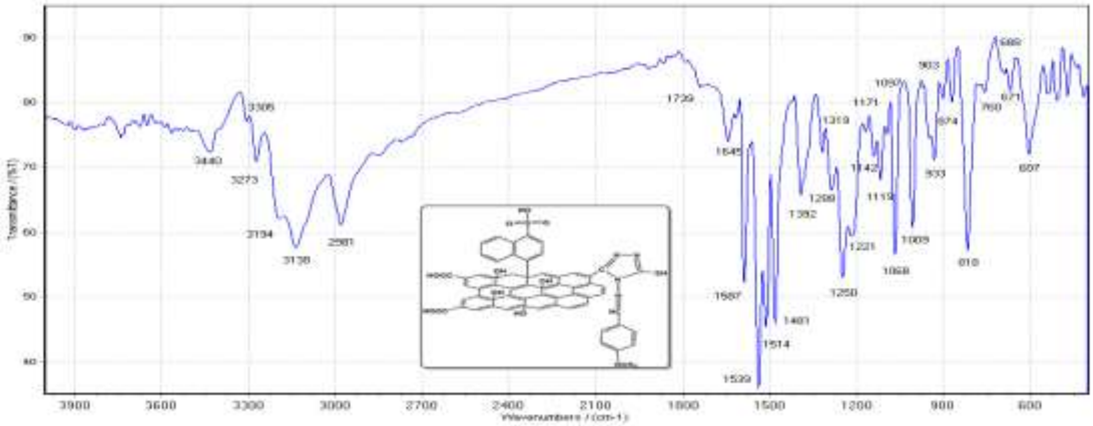

Figure 20: (ArNRGOTZS$_3$)s FT-IR Spectrum

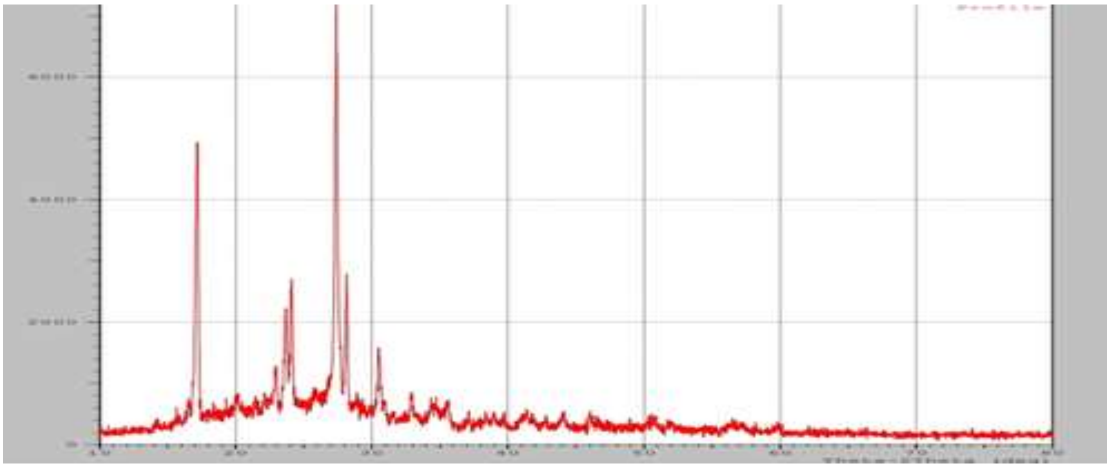

Figure 21: XRD Pattrens of (ArNRGOTZS$_1$)

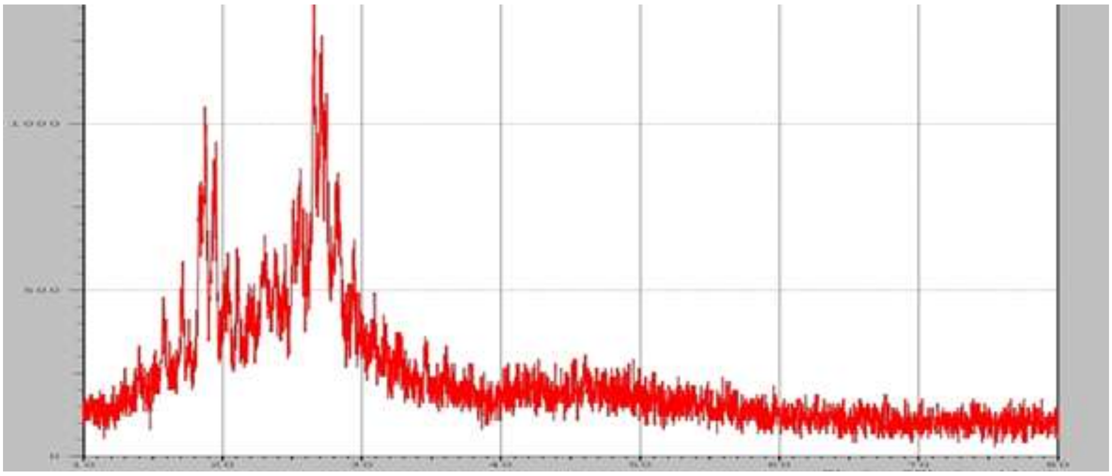

Figure 22: XRD Pattrens of (ArNRGOTZS$_2$)

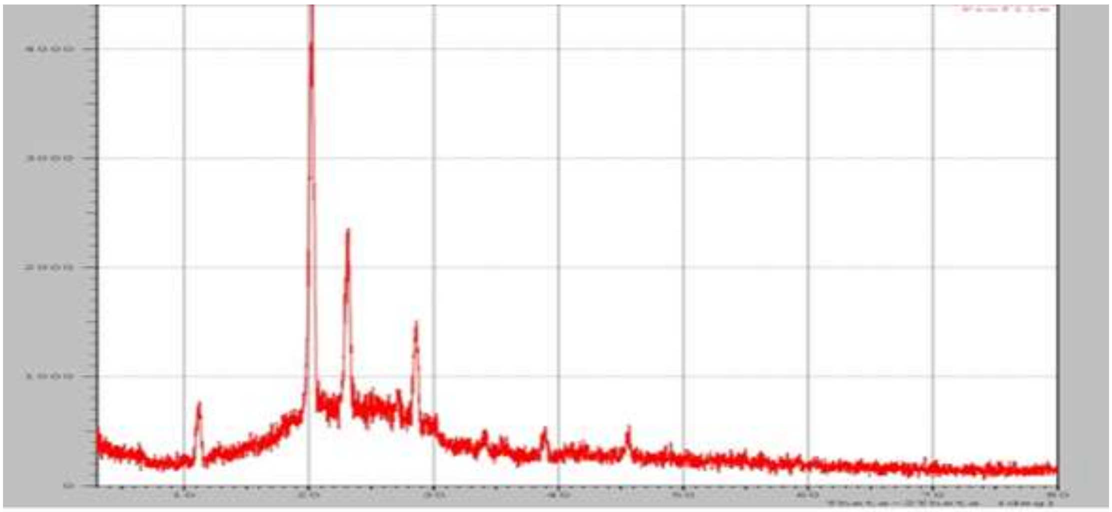

Figure 23: XRD Pattrens of (ArNRGOTZS$_3$)

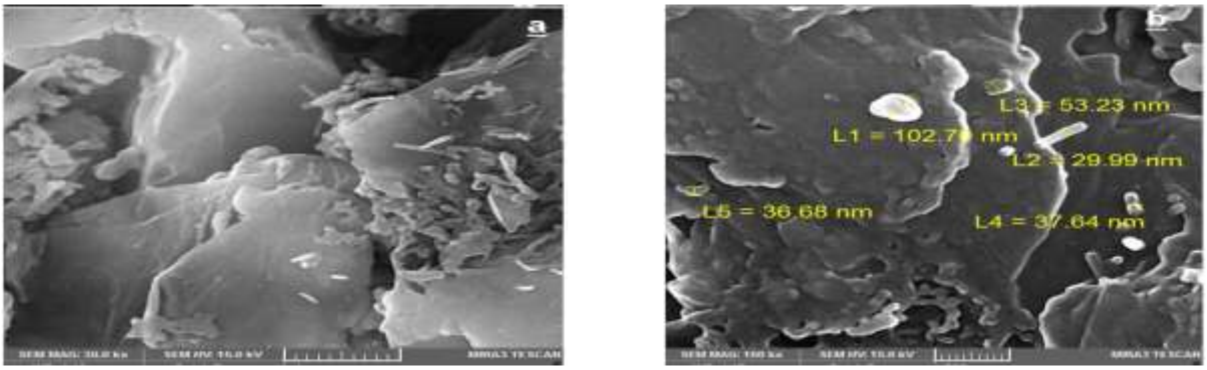

Figure (24) :SEM Image of (ArNRGOTZS$_1$)

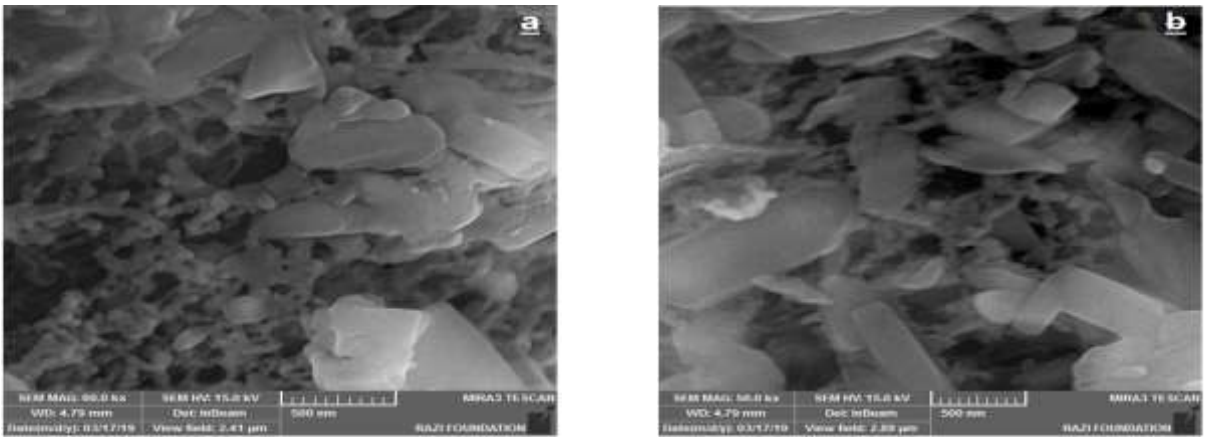

Figure 25: SEM Image of (ArNRGOTZS$_2$)

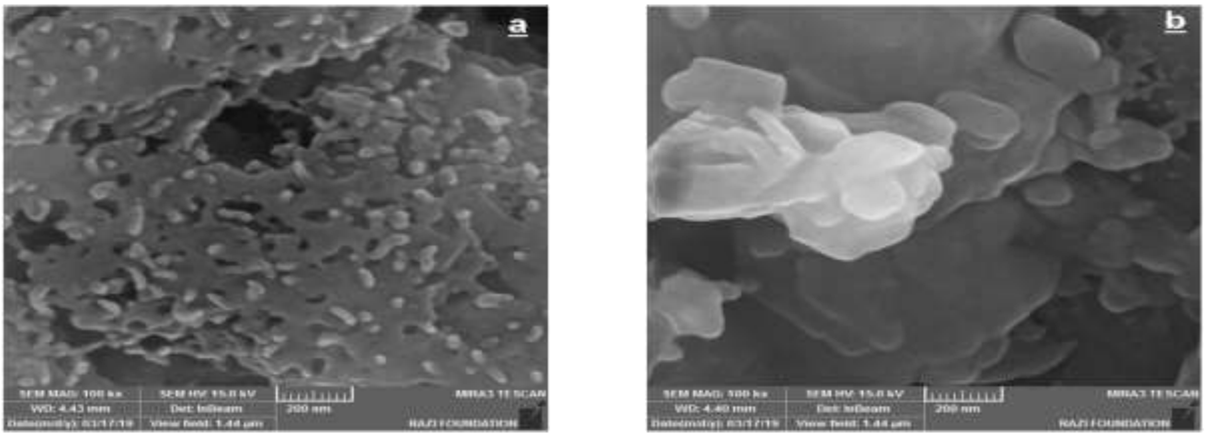

Figure 26: SEM Image of (ArNRGOTZS$_3$)

(ArNRGOTZS1-3)s measurements (TGA, DTG, and DSC) shown in Fig (24,25,26), respectively.

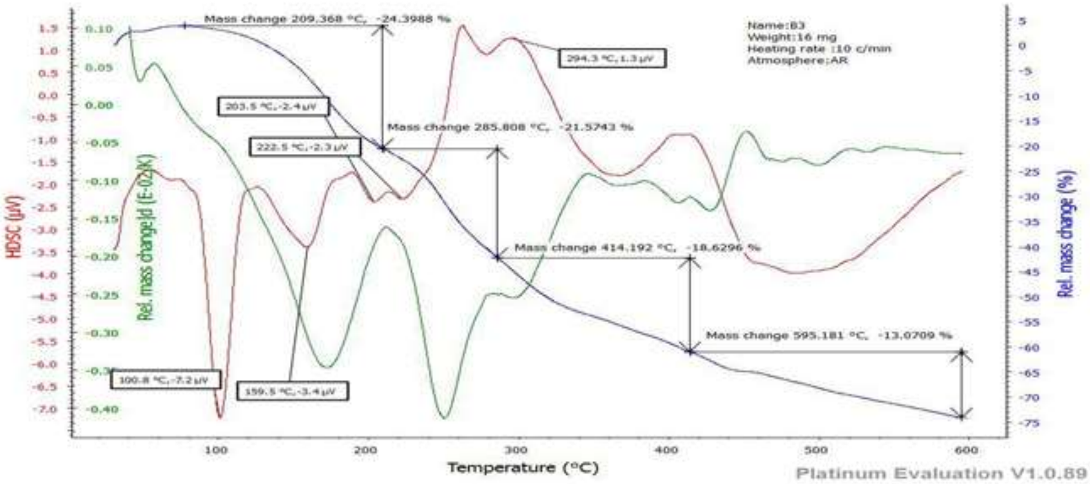

Figure 27: (ArNRGOTZS$_1$)s Curves (TGA ,DTG and DSC)

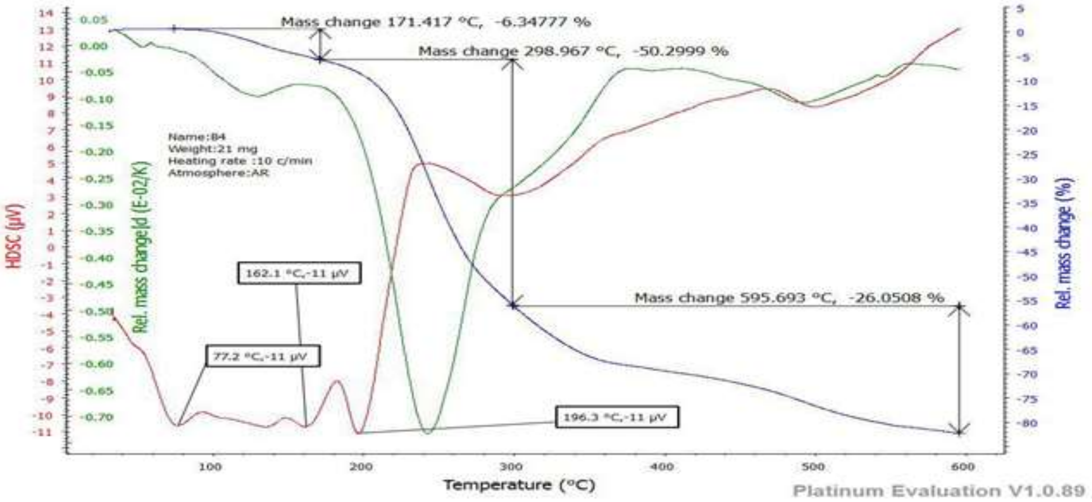

Figure 38: (ArNRGOTZS$_2$)s Curves (TGA ,DTG and DSC)

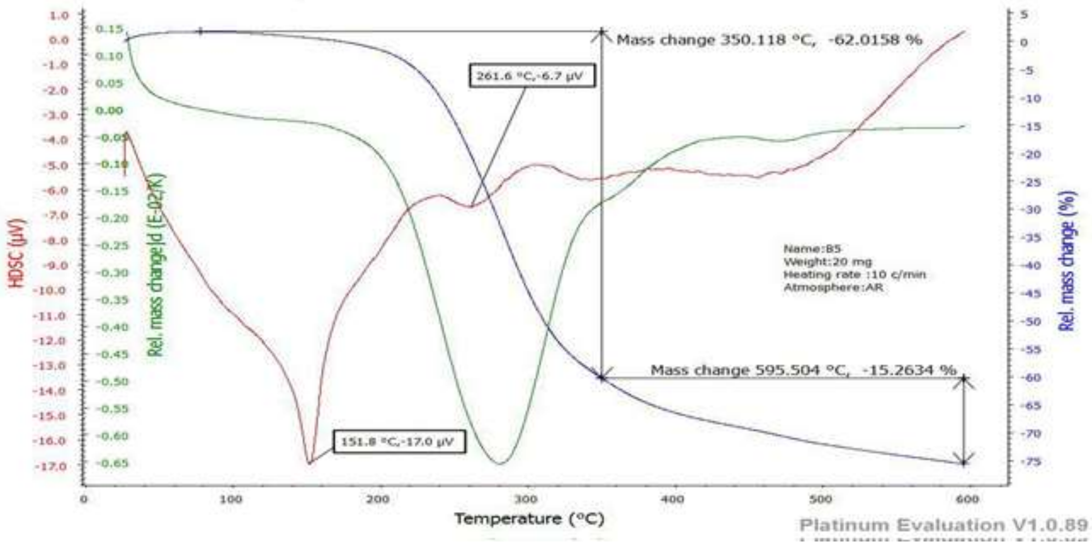

Figure 49: (ArNRGOTZS$_3$)s Curves (TGA ,DTG and DSC)

Conclusion

In this paper, the formation of 1,2,4-triazol-4-amino Schiff Bases of Decorated Nano Reduced Graphene Oxide with 2-naphthol-4-sulfonic acid Then prepare the corresponding and decorated derivative with 2-naphthol-4-sulfonic acid, Using the electric method (ArNRGO) in two steps, first one preparing the disonium salts and the second, electrical decoration, Prepare the corresponding Triazole derivative (ArNRGOTZ) by thermal fusion for the Ar-NRGO Aromatics Derivative with Thiocarbozide (TC), Also, ArNRGOTZS (3-1) nanoschiff bases prepared by heat mixing between ArNRGOTZ with 4-Nitrobnzaldehyde, 4-Chlorobenzaldehyde and 4-Methoxybenzaldehyde, characterization by IR, XRD and SEM infrared spectroscopy with stability evaluation from TGA, DSC and DTG measurements.

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