

One Pot Synthesis of Graphene by Exfoliation of Graphite in ODCB

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ABSTRACT

Graphene, an extraordinary allotropy of carbon, the 2D nanosheet, have been synthesized through exfoliation of graphite in ortho-dichloro benzene by sonication. The morphological changes in different interval of sonication have been investigated by Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). Raman Spectra confirmed the formation of defect free Graphene sheets. As prepared Graphene showed high thermal stability under N₂ atmosphere. It has been observed that sonication for 4 hours, effectively exfoliates graphite to form Graphene sheets. However, further sonication leads to restacking of Graphene sheets. The formation of Graphene is supposed to be due to the Sonopolymerization of the solvent (ortho-dichloro benzene) and graphite-solvent interaction.

Keywords: Graphene; Graphite; Ortho-Dichloro Benzene; Sonication; Raman Spectra

1. Introduction

The discovery of Graphene stimulates the scientists to unlock a new aspect in the field of nanoscience and nanotechnology. Graphene has become the “miracle material” of the 21st Century. It is considered as the thinnest material of today’s world. It has no band gap, which allows it to be a wonderful candidate for use in photo-voltaic (PV) cells. Beside this, it has attracted great deal of research due to its excellent electrical conductivity, extraordinary mechanical properties, large surface area, high aspect ratio, low coefficient of thermal expansion [1-5]. Graphene is the flat layer of carbon atoms, compactly packed into a two-dimensional honeycomb lattice. It is expected that, with its outstanding properties, Graphene can replace Silicone in near future. However, great deal of researches has been done to synthesize single layer as well as multi-layer Graphene. Earlier, Graphene were synthesized by different procedures like mechanical exfoliation of graphite [6], Chemical vapor deposition method [7] etc. But these are not efficient method to synthesize Graphene in commercial scale. Chemical route is the most realistic as well as most promising method for Graphene synthesis from graphite. The simplest and most common method of graphite exfoliation is the oxidation of graphite to graphite oxide by strong oxidizing agent [8]. Further exfoliation and reduction of Graphene oxide forms chemically converted Graphene [9-11]. Though some other methods like thermal expansion [12], ball

mixing, liquid phase exfoliation [13] etc. were tried to synthesize Graphene, but this oxidation-reduction method is the most widely used method for large scale synthesis of Graphene. Though this method is most practical method, it also has some disadvantages like presence of defects, low yield, presence of harsh oxidation chemistry and long term reaction.

The homogeneous Graphene dispersion in some common organic solvents like benzene, toluene, nitrobenzene etc was reported [14]. Polar solvents like N, N-dimethylformamide (DMF), N-methylpyrrolidone (NMP) can also exfoliate graphite to form homogeneous Graphene dispersion [15,16]. But, among the nonpolar solvents, ortho-dichloro benzene (ODCB) was reported to produce homogeneous Graphene dispersion [17].

Here we have demonstrated one pot synthesis of Graphene through exfoliation of graphite in ODCB. The synthesis based only on sonication. We have studied the effect of chlorinated organic molecules on the electronic structure of Graphene upon sonication. Further, we have tried to find the mechanism of the formation of Graphene through exfoliation of graphite. Sonication for a particular time interval leads complete dispersion of graphite molecules in ODCB to form homogeneous dispersion of Graphene. Further, the electronic structure of Graphene has been characterized by FTIR, SEM, TEM and Raman spectra. The advantages of this synthesis method are its simplicity, absence of long term synthetic route and high yield.

ODCB is a versatile, high boiling solvent. It is a pre-

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ferred solvent for dissolving fullerenes and also it can form stable Single Wall Carbon Nano tube (SWCNT) dispersion. However the reasons behind the choice of this solvent for graphite exfoliation were well described by Hamilton *et al.* [16]. Additionally one of the major advantages of ODCB is, like other organic halides, it is also known to decompose during sonication to liberate chlorine and polymerize [17,18].

2. Experimental

2.1. Materials Used

Graphite Fine Powder (Extra Pure) was obtained from Loba Chemie Pvt. Ltd. Mumbai (India). 1,2-dichloro benzene was supplied by MERCK Limited. Mumbai (India). Both the chemicals used as received, without any further distillation or purification.

2.2. Synthesis of ODCB Suspension of Graphene

Stable homogeneous dispersion of Graphene was obtained through a simple chemical approach [16]. In a typical process, Graphite (1 gm) was mixed with ODCB (300 ml) in a beaker. The black colored solution was homogenized for different time interval in an ultrasonic bath (Freq. 40 KHz and Power 100 W). All the solutions were heated at 185°C for solvent evaporation. The sample codes with sonication time are shown in the **Table 1**. After each time interval (1 hr) some solution were taken out from the beaker and kept in sample vial for characterization.

All the sample vials were kept for one month in room temperature in order to verify stable dispersion of Graphene. The corresponding vial pictures are shown in **Figure 1**. As observed from the **Figure 1**, it is clear that in Gr1, the graphite molecules were settled down in the bottom part of the vial. Partial settle down was observed for Gr2. So, it can be concluded that 2 hrs sonication is not sufficient to get colloidal dispersion of Graphene. However for rest of the samples black colored colloidal solution was observed. As prepared colloids were homogeneous and they remained stable for more than 6 months without any sediment. Stable uniform dispersion of Graphene can be achieved after 4 hrs sonication.

Table 1. Sample details.

Sample Codes	Sonication Time (mins)
Gr1	60
Gr2	120
Gr3	180
Gr4	240
Gr5	300

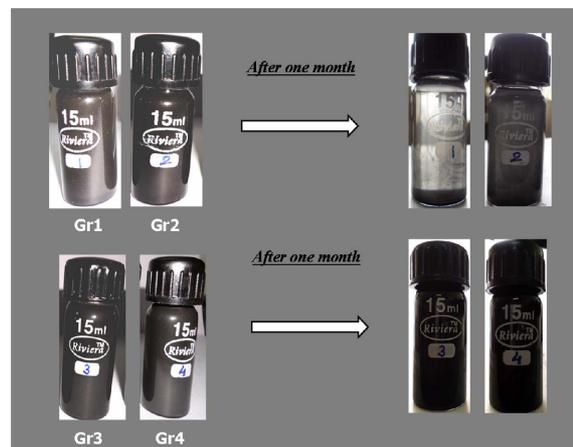


Figure 1. Vial images (before and after one month).

3. Characterization Techniques

3.1. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis of Graphene samples was characterized using a NEXUS 870 FTIR (Thermo Nicolet). For the IR spectrum a small amount of material was mixed with KBr in adequate level to make a disk and the disk was analyzed for getting the spectrum.

3.2. Scanning Electron Microscopy (SEM)

The surface morphologies of the samples were analyzed by using Tescan VEGA LSU SEM.

3.3. Transmission Electron Microscopy (TEM)

All the samples were analyzed by TEM, JEOL 2100 in order to understand exact morphological change of Graphene dispersion in ODCB with sonication time. A small amount of the sample was dispersed in acetone and deposited on copper grid.

3.4. Raman Spectra

Raman spectra were recorded between 500 to 3500 cm^{-1} in a Raman Imaging System WITEC alpha 300 R with 532 nm wavelength.

3.5. Thermogravimetric Analysis (TGA)

Thermogravimetric analysis curve was recorded with a Dupont 2100. Thermogravimetric analyzer. The TGA measurement was conducted with a heating rate of 10°C/min under N_2 atmosphere from room temperature to 800°C.

4. Results and Discussion

4.1. FTIR Analysis

The chemical environment of all Graphene samples has

been analyzed by FTIR and spectrums of are shown in **Figure 2**. All the characteristic peaks of graphite are present in all the samples. The peak at 1634 cm^{-1} arises due to the C=O stretching vibration of carbonyl functional group. Broad peak at 3434 cm^{-1} corresponds to the O-H stretching vibration. Further, peaks at 2854 and 2924 cm^{-1} attributes to the symmetric and asymmetric stretching vibrations of C-H bonds respectively. Additionally, several peaks arises in the range of $400 - 800\text{ cm}^{-1}$ due to several vibration modes of C-H like outer bending vibration, C-H in plane bending, C-H out of plane wagging [19,20]. However, there are no drastic changes in the spectrums of the prepared samples. These indicate the absence of any functional group in as prepared samples.

4.2. Morphological Study

The morphological changes upon sonication for different time interval are analyzed by SEM and TEM analysis and the images are presented in **Figures 3** and **4**. **Figure 3** shows the SEM image of graphite fine powder which clearly demonstrated the stacked Graphene sheet structure. Sonication for 1 hr, exfoliated the Graphene sheets and the intergalary distance increased tremendously (Gr1). Further increase in the sonication time (2 hrs), resulted in the delamination of the Graphene sheets (Gr2). Sonication for 3 hrs showed reduction in the Graphene layers, as compared to two hour sonication (Gr3). However, 4 hour sonication showed complete delamination of Graphene layers (Gr4). Increase in sonication time to 5 hrs (Gr5) showed restacking of the Graphene sheets. The delamination process was further confirmed by TEM.

The characteristic TEM images of as prepared samples are shown in the **Figure 4**. In Gr1 graphite particles are present in agglomerated form. On the other hand, in Gr2, some portions of the graphite particles are remained in agglomerated form as well as in some portion, Gra-

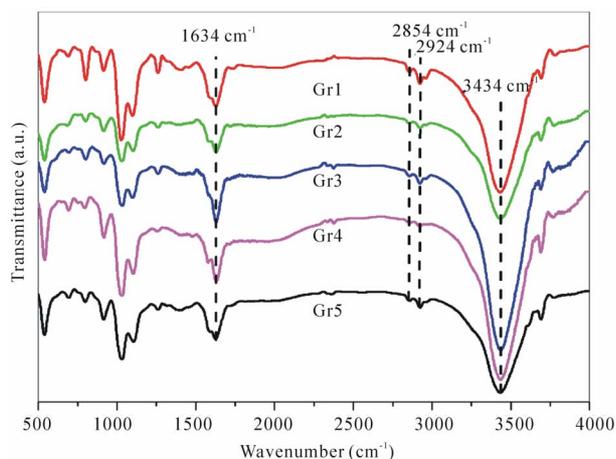


Figure 2. FTIR spectra of Gr1, Gr2, Gr3, Gr4, Gr5.

phene nanosheets are formed. However, homogeneous dispersion of Graphene nanosheets is observed only for Gr4. The TEM image of Gr4 shows the multilayered Graphene nanosheets and the thicknesses of the Graphene nanosheets are 10 - 15 nm. This indicates that a particular sonication time is required for the exfoliation of Graphene in ODCB. 4 hrs sonication is sufficient for the homogeneous dispersion of Graphene. Further sonication leads to the agglomeration of graphite.

Based on the morphological study and other relevant characterizations, we have demonstrated a diagram (**Figure 5**) for better understanding of the changes during sonication for different time interval. As shown in the diagram, 1 hr sonication has not able to separate the graphite particles. 2 hrs sonication can only separate some portion of the graphite particles. However, better exfoliation of Graphene sheets is observed for Gr4. Further re stacking of Graphene sheets is found for Gr5.

The characteristic SAED (Selected Area Electron Diffraction) images of the samples are shown in **Figure 6**. For Gr1, no ring patterns are observed, which is due to the amorphous nature of graphite. A perfect six membered ring pattern is observed for Gr4 among all the samples. This is due to the crystalline nature of Graphene. These results indicate the formation of Graphene sheets in Gr4.

4.3. Raman Spectra

Raman spectroscopy is mainly used to understand the structural properties of Graphene materials. It is also an important tool for identification of disorder and defect in molecular structure as well as to calculate defect density, doping level etc. The major Raman features of the Graphene samples are so called G band ($\sim 1575\text{ cm}^{-1}$) and D band ($\sim 1350\text{ cm}^{-1}$). However G band initiates from in-plane vibration of sp^2 carbon atoms of Graphene samples [21], whereas the D mode arising from a breathing mode of a K-point photons of A_{1g} symmetry [22]. Besides these two, one additional peak arises from a two phonon double resonance Raman process, known as 2D band ($\sim 2670\text{ cm}^{-1}$). The Raman spectra of all the samples and Graphite (inset) are shown in **Figure 7**. Among all the samples, Gr4 shows G band at 1353 cm^{-1} and D band at 1584 cm^{-1} , which are comparable with the characteristic peaks of Graphene. The peak intensity ratio (I_D/I_G) is found to be 0.015 for Gr4 (**Figure 8**). Similar value was reported by W. Yang *et al.* [13]. However, the peak intensity ratio decreases from Gr1 to Gr4 and then increases for Gr5. The decrease in D band intensity with increasing sonication time indicates that sonication induces defect-free Graphene sheets. It is also found that among all the samples, Gr4 shows lowest D band intensity, after that D band intensity increases with further sonication. This proves that sonication for a particular time interval

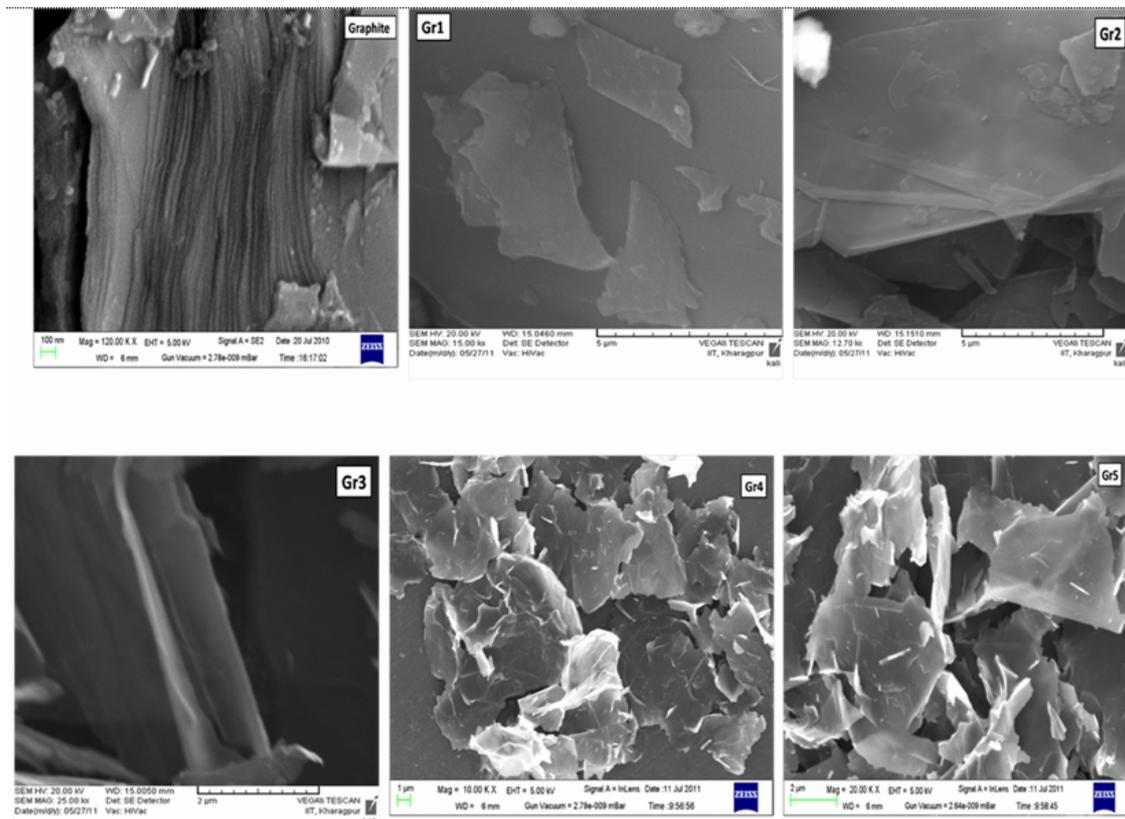


Figure 3. SEM images of Graphite, Gr1, Gr2, Gr3, Gr4, Gr5.

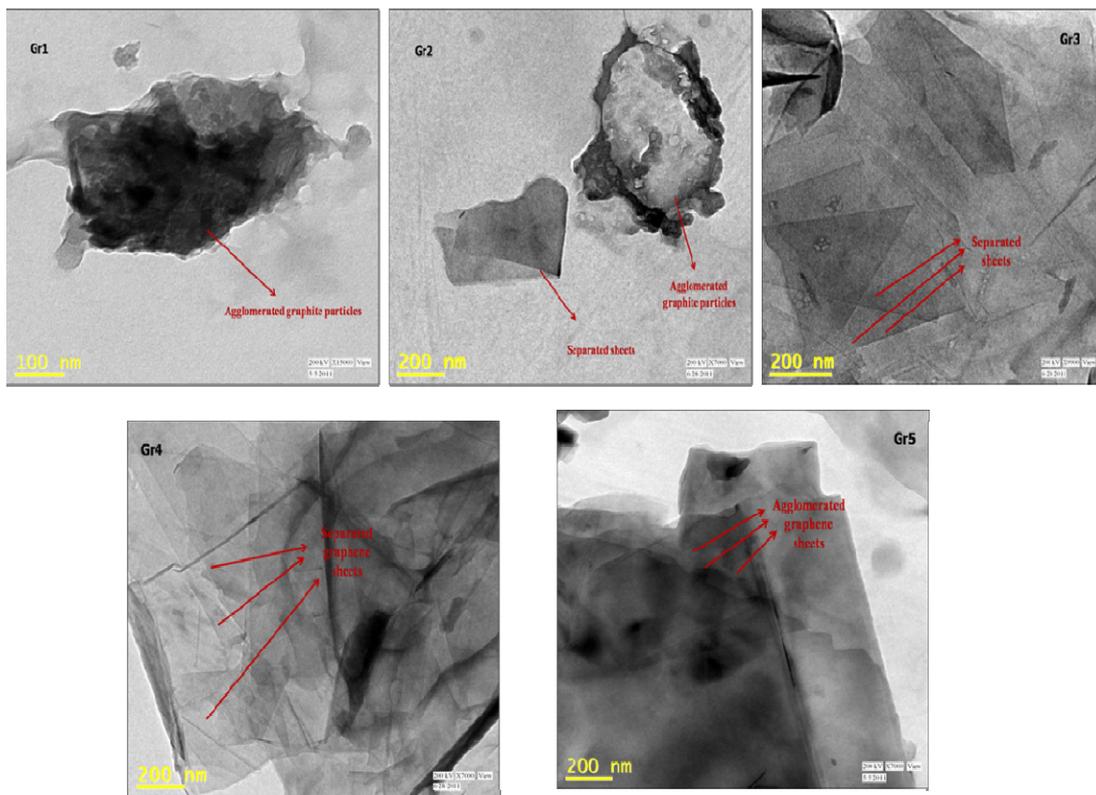


Figure 4. TEM images of Gr1, Gr2, Gr3, Gr4, Gr5.

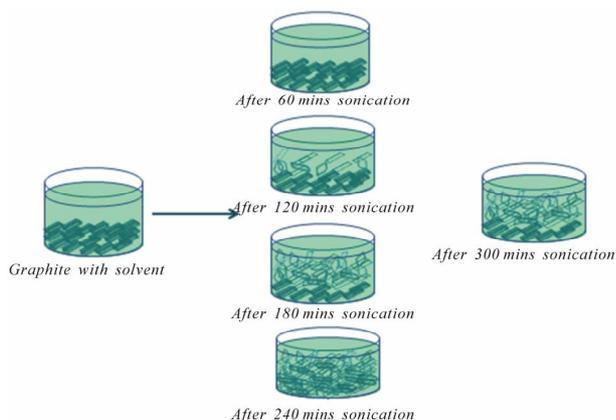


Figure 5. Schematic Diagram indicating the physical changes of graphite particles during sonication for different time interval.

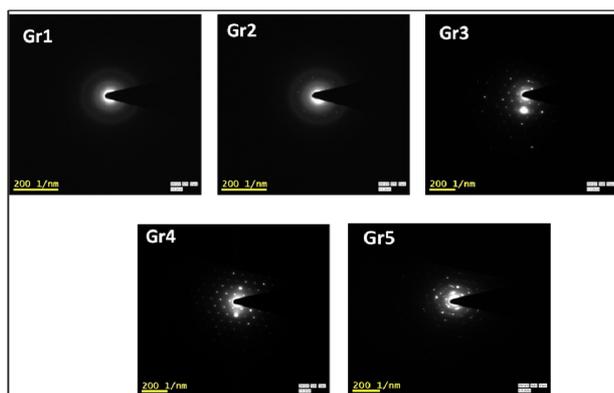


Figure 6. SAED images of Gr1, Gr2, Gr3, Gr4, Gr5.

efficiently exfoliates graphite without initiating defects.

4.4. Mechanism of Stable Dispersion of Graphene

The mechanism of formation of stable homogeneous dispersion of Graphene in ODCB can be explained through solvent-graphite interaction concept. Sonication leads to the Sonopolymerization of ODCB [23], which is responsible for the stabilization of Graphene dispersion. However, there occurs no chemical interaction between graphite layers and ODCB, which is confirmed by FTIR analysis. The dispersion of graphite in ODCB leads to the formation of Sonopolymer [24-26], which adheres to the graphite layers. Sonication leads to the separation of graphite layers. Once the graphite layers are separated, solvent molecules penetrates inside the inter gallery of graphite. Further sonication induces the formation of Sonopolymer between the graphite layers. With increasing sonication time the formation of polymer bounded graphite sheets increases as well as distance between the graphite layers increases. This leads to the formation of

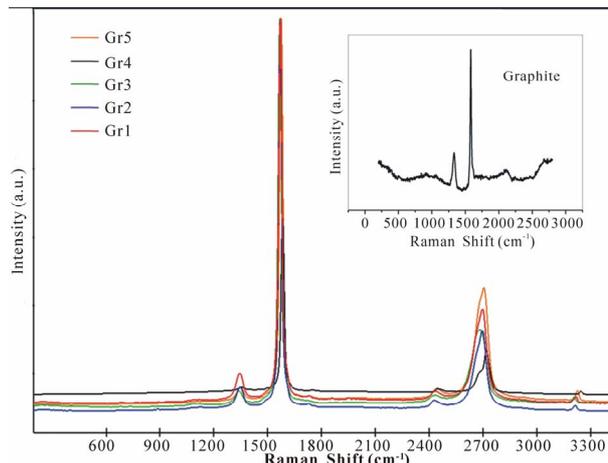


Figure 7. Raman spectra of Gr1, Gr2, Gr3, Gr4, Gr5. Inset shows the Raman Spectra of Graphite.

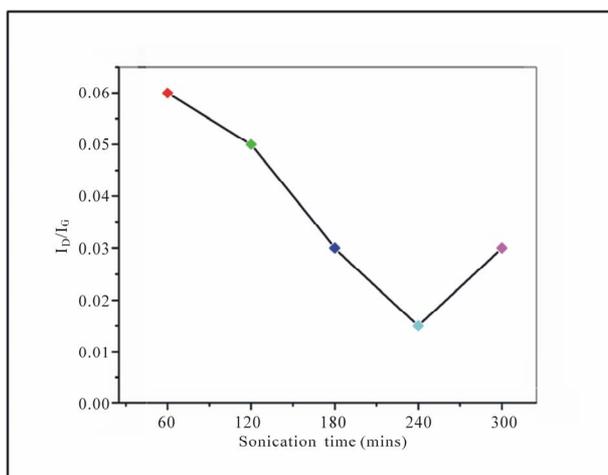


Figure 8. Peak intensity ratio vs. sonication time curve.

Graphene sheets as in Gr4. On the other hand, it is already confirmed that the key factor for graphite dispersion is the enthalpy of mixing [15]. The enthalpy of mixing for graphite dispersion in solvents should be close to zero. That can only be achieved if the surface energy of graphite matches with the surface tension of the solvent. It is also predicted that good solvents for graphite dispersions should have surface tensions in the region of 40 - 50 mJ/m². The surface tension of ODCB is very close to this region (36.01 mJ/m²). Thus minimum energy cost is required for the exfoliation of graphite in this solvent. However, further sonication directs to the decomposition of ODCB, which induces re agglomeration of graphite layers as observed in Gr5.

The preparation of composites based on this synthesized Graphene for different applications like supercapacitors, polymer blends, formation of radar absorbing substances are in progress and will be reported elsewhere in future.

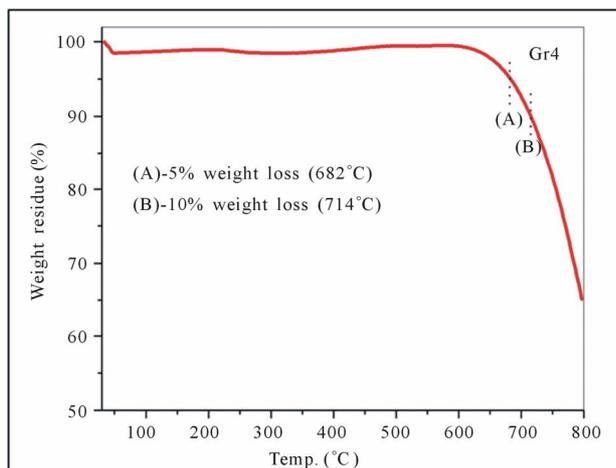


Figure 9. TGA curve of Gr4.

4.5. Thermogravimetric Analysis

In order to analyze the thermal behavior of as prepared Graphene sample (Gr4), we have carried out the TGA analysis and the curve is shown in **Figure 9**. As shown in the Figure, a slight mass loss is observed at below 100°C, which can be attributed to the removal of adsorbed water molecules. However, in spite of this minor weight loss, no significant mass loss is detected upto 650°C. The major mass loss occurred at around 650°C. The 5% weight loss is observed at 682°C and 10% weight loss is observed at 714°C. Hence, the TGA analysis confirms the enhanced thermal stability of this Graphene sample.

5. Conclusions

In Summary, a simple method has been developed for the synthesis of multilayered Graphene through sonication via exfoliation of graphite in ODCB. Graphite powder provides Graphene sheets through graphite-solvent interaction. The major advantage of this method is the absence of harsh chemicals like strong acids, reducing agents etc. The formation of Graphenenanosheets upon exfoliation of graphite is expected due to several factors:

- 1) Sonopolymerization of the solvent;
- 2) Graphite-solvent interaction;
- 3) Matching of the surface energy of graphite with the surface tension of solvent;
- 4) The enthalpy of mixing for the graphite dispersion is close to zero.

As prepared Graphene sheets could be useful for both scientific studies and various commercial applications.

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