International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 04 Special Issue: 09 | Sep -2017 www.irjet.net p-ISSN: 2395-0072
One Day International Seminar on Materials Science & Technology (ISMST 2017)
4th August 2017
Organized by
Department of Physics, Mother Teresa Women's University, Kodaikanal, Tamilnadu, India

Large Scale Synthesis and Characterization of Few Layer Graphene Nanosheets for Supercapacitor Applications

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Abstract - *Few layers Graphene nanosheets were synthesized by large quantity via modified Hummer's method chemical oxidation route involving graphite oxidation and thermal reduction. Few layer thermally reduced graphene were characterized by powder XRD crystalline materials. SEM and High resolution TEM (HR-TEM) observations show that graphene nanosheets were produced with sizes in the range of 20 to 30 nm. The order and disorder quality of graphene nanosheets were characterized by Raman spectroscopy. The electrochemical characterization was used in few layer TR-GNS for cyclic Voltammetry. The maximum specific capacitance (C_{sp}) was observed 57 F/g at scan rate 50 mV/s. The large scale synthesis of few layer thermal reduction graphene was effective as using in increasing the gravimetric specific capacitance for the supercapacitor electrodes.*

Key Words: *Graphene, Hummer's method, Thermal reduction, Capacitance, Supercapacitor.*

1. INTRODUCTION

Recent years graphene preparation is interesting on two dimensional carbon nanostructured materials and it consist of mono layer carbon atoms given densely and closely honeycomb like hexagonal structure [1]. And their amazing Properties were high surface area and excellent electronics properties. Hence consider to be promising electrode materials energy storage materials and also considerable potential applications for supercapacitors, Batteries and nanoelectronics devices [2]. These emerging applications require availability of graphene on the mass scale and thus suitable processes are necessary for its large scale production down to single or few layer nanosheets level. The strong chemical oxidizer materials were intercalated in the bulk graphite sheets exfoliated into few layer or single layer graphene [3-6]. The present work focuses on the preparation of graphene by modified Hummers method a graphene nano layers were produced in large scale from graphite and thermally reduction technique. Synthesized reduced graphene was materials characterized and electrochemical performance for supercapacitor applications.

2. EXPERIMENTAL

2.1 Modified Hummer's Method

Few layered Graphene nanosheets were synthesized by modified Hummers method. Graphene oxide was synthesized using graphite (5.0 g) and $NaNO_3$ (2.5 g) were mixed with 110 ml of concentrated sulphuric acid (H_2SO_4) in 2000 ml round bottom flask in an ice bath, and kept under magnetic stirring for 30 min. To the cold solution, 6.0 g of potassium permanganate ($KMnO_4$) was gradually added with constant stirring. The solution was further diluted by slowly added 600 ml distilled deionized water (D.I) followed by 15 ml of H_2O_2 (30%) the resultant mixture was again stirred for 1h. The above resultant solution was washed the reaction product centrifuged and washed with deionized water until the pH of the solution was nearly neutral. The obtained sediment was filtered and the resulting graphite oxide (GO) was dried at 45°C for 24 h, finally obtained solid GO dark brownish powder.

2.2 Thermal Reduction-GO.

The GO powder into reduced graphene was obtained by thermal reduction using muffle furnace. 2.0 g of GO powder was place in center of the furnace 2 h at 400 °C. The fine dark black colored powder was obtained reduced few layer graphene nanosheets. Finally the synthesized T-RGO was further material characterized by XRD, Raman, SEM, HR-TEM.

2.3 Material Characterizations.

Crystallographic structure of the T-RGO were determined by a powder X–ray diffraction (XRD) system using X'PERT-PRO analytical model with Cu Kα radiation (λ = 0.15406 nm). Micro – Raman spectroscopy was performed using Princeton instruments Acton (SP–2500) with 514.5 nm laser excitation (Ar^+ laser source). The morphology micrographs for reduced Graphene was analyzed using scanning electron microscope (SEM) with VEGA 3 TESCAN instrument and HR–TEM with TECNAI, T–30, operated at 250 KV.

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3. RESULT AND DISCUSSIONS.

3.1. XRD and Raman Analysis.

X-ray diffraction pattern of thermally reduced graphene shown in fig. 1(a). The diffracted broad peak was observed for T-RGO at 2θ=26° corresponding plane to (002) hexagonal structure and a d-spacing value of 3.41 Å. After thermal treatment reduction, GO nanosheets were ordered crystal structure. Raman spectroscopy is most powerful techniques and used for quality and order/ disorder of the materials. Raman spectra of thermally reduced graphene oxide fig.1 (b) shows the Raman spectra of T-RGO, it shows the strong graphitic lattice G-band at 1596 cm^{-1} and small disorder band D-band at 1361 cm^{-1} was observed. Though the thermally reduced Graphene is well ordered and intensity disordered ratio I_D/I_G is 0.87. ID/IG ratios are less than one, indicating that the T-RGO materials have small degree of disorder, which is in the typical range of pure reduced graphene, which indicates that the T-RGO has a good crystalline of order form in bulk synthesis.

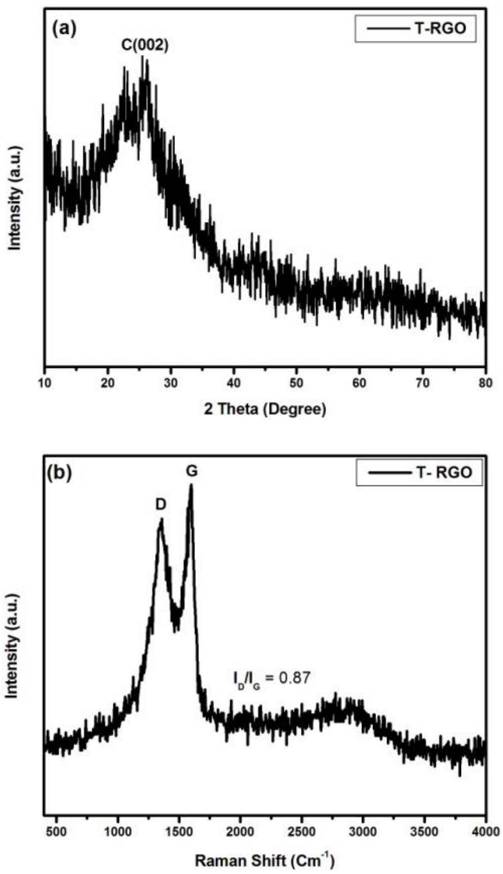

Fig. 1.(a) X-ray diffraction pattern and (b) Raman spectrum for T-RGO.

3.2 SEM and HR-TEM Analysis.

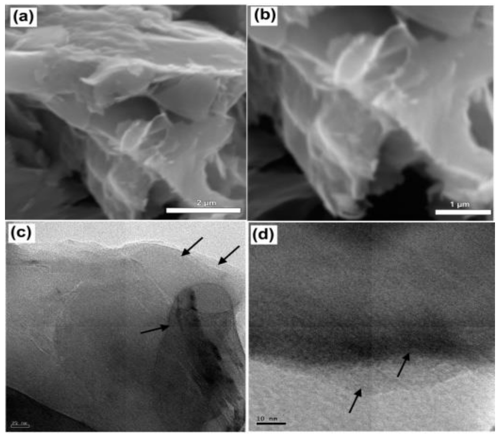

Fig. 2. (a & b) SEM and (c & d) HR-TEM morphological images of few layer graphene nanosheets.

The obtained thermally reduced graphene nanosheets morphologies were analyzed by TEM and HRTEM observations. Fig. 2 (a & b) shows a low magnification SEM image of graphene nanosheets are few µm scale with crumpled and folded graphene sheets few layer. HR-TEM analysis for reduced graphene nanosheets (a few hundred square nanometers scale) were observed to be situated graphene nanosheets edges rippled and entangled with each other. The few graphene nano layer transparency regions indicated by arrows in Figure 2 (c & d) are likely to be few layer graphene nanosheets.

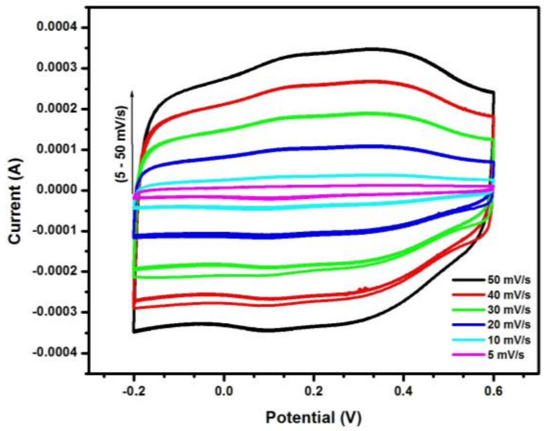

Fig.3 Cyclic voltammerty of T-RGO at different scan rates (5-50mV/s).

Fig. 3 shows the CV measurements were carried out in 1M H_2SO_4 electrolyte, the potential range between -0.2 to 0.6 at a scan rate of 5 to 50 mV/s. The specific capacitance (C_{sp}), was calculated from cyclic voltammetry (CV) curve were using at scan rate 50 mV/s obtained maximum capacitance 57 F/g for T-RGO using the formula C_{sp} = Area under the CV curve/(Scan rate * Voltage window * mass of the material). The C_{sp} calculation used only 50 mV/s for CV cure was rectangular shape for EDLC capacitance nature in fig.4.

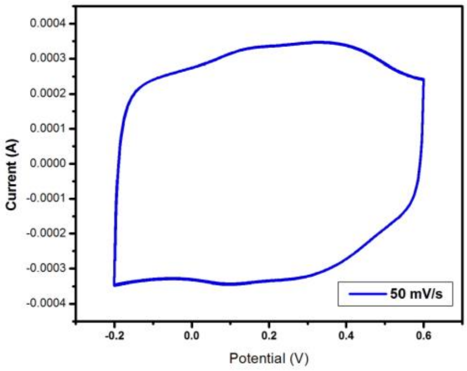

Fig. 4. Cyclic Voltammetry of T-RGO at scan rate 50mV/s.

4. CONCLUSIONS

Few layered Graphene nanosheets were synthesized by modified hummer's method in large scale productions for electrochemical applications. The T-RGO materials characterized was used XRD, Raman, SEM & HR-TEM. The electrochemical analysis from CV curve using supercapacitor specific capacitance calculations (Csp) – 57 F/g at 50 mV/s. graphene oxide (GO) and thermally reduced (GO) can be massively prepared through a chemical exfoliation/ oxidation route. Rapid thermal treatment reduction step improved the exfoliation in large scale possible. GO & T-RGO was improved quality analysis of the product by suitable techniques and finally measured the electrochemical energy storage for supercapacitor application.

ACKNOWLEDGEMENT

We acknowledge the Department of physics, St.Joseph's College, Trichy, for their help in experiments and characterizations.

REFERENCES

[1] K. S. Novoselov, A. K. Geimet al. Electric field effect in atomically thin carbon films. Science 306 (2004) 666-669.

[2] A. K. Geim and K. S. Novoselov, The rise of graphene, Nature Materials. 6 (2007) 183-191.

[3] Y. W. Zhu, et al. Graphene and graphene oxide: synthesis, properties and applications. Advanced Materials. 22 (2010) 3906–3924.

[4] R. Ruoff, Graphene Calling All Chemists. Nature Nanotechnology, 3(2008) 10-11.

[5] W.S. Hummers, and R.E. Offeman, Preparation of Graphitic Oxide. Journal of the American Chemical Society, 80 (1958) 1339.

[6] V. Thirumal , A. Pandurangan , R. Jayavel, R. Ilangovan, Synthesis and characterization of boron doped graphene nanosheets for supercapacitor applications. Synthetic Metals 220 (2016) 524–532.

BIOGRAPHIES

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