



Processing Characterisation and Application of Graphene

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Abstract: In this study, an attempt has been made to synthesise the graphene layers using electrochemical approach. This method is the cost effective and simple approach for the production of high-quality graphene. In this method graphite electrodes (both cathode and anode) were used instead of platinum, since the former is expensive. In particular, electrical properties of graphene were studied by varying the centrifuge parameters along with morphological examination. For characterisation, scanning electron microscope (SEM), UV spectroscopy, atomic force microscope (AFM), Fourier transmission infrared spectroscopy (FTIR) and X-ray diffraction (XRD) were utilised. Following are the observations; i) folded sheets of graphene were obtained, ii) an amorphous pattern at $2\theta = 26.6^\circ$ was observed, iii) the absorption peak of graphene decreased at high wavelengths (266 nm), iv) the graphene peak was also confirmed by FTIR i.e. at 1655.5 (C=C) and v) thickness of flakes were found to be 60 nm by AFM. Moreover, further improvement in electrical conductivity of graphene layer is useful for dye-synthesized solar Cell (DSSC) applications which was synthesised in this research.

Keywords: Graphene; Electrochemical; graphite electrode; electrical and morphological properties; Dye-synthesized

1. INTRODUCTION

Graphene is an exciting material that has been researched worldwide as a two-dimensional carbon allotrope (Novoselov, *et al.*, 2004). The dense arrangement of carbon particles in one atom thick layer in a honeycomb crystal lattice has made it a considerable material because of its exceptional electronic and optoelectronic properties (Novoselov, *et al.*, 2004). It is a material with excellent strength to weight ratio. Because of the reported properties and uses of this two dimensional structure, it is by all accounts intriguing to apply it in different electronic devices. Although graphene is acknowledged greatly for its outstanding electronic materials, fabricating single sheet of graphene is still a conundrum.

Another of graphene's emerging property is its intrinsic strength (Novoselov, *et al.*, 2004). Due to the strength of its 0.142 nm-long carbon bonds, graphene is the most grounded material ever found, with an extreme rigidity of 130,000,000,000 Pascals (or 130 gigapascals), contrasted with 400,000,000 for A36 auxiliary steel or 375,700,000 for Aramid (Kevlar) (Novoselov, *et al.*, 2004).

Not only the graphene is remarkably strong but it is equally light in weight i.e. 0.77 milligrams/square meter (for correlation purposes, 1 square meter of paper is around 1000 times heavier).

Mechanically peeled and epitaxially grown graphene films (Sutter, *et al.*, 2008) show the high quality layers. However, it is not reasonable to produce at mass level. CVD techniques utilizing reactant metal substrates have demonstrated the capacity of developing extensive zone graphene, extraordinarily reassuring their applications in exceedingly straightforward and adaptable leading films. Albeit more endeavours ought to be made to bring down the production costs, especially those concerned with the high-temperature handling and costly substrates.

Moreover, the synthetic shedding strategies in view of the Hummers' strategy, the oxidation of graphite into thin graphene oxide (GO), trailed by compound or warm diminishment, have as of late attracted much consideration because of the benefits of possibly minimal effort and arrangement handled fabrication. However, the oxidation procedure is extremely

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harmful for the honeycomb cross-sections of graphene. Similarly, the ensuing diminishment of GO sheets regularly includes high temperature to recover the graphitic structure. Moreover, the resistance of the films acquired from reported reduced GO (rGO), extending from 1k to 70k Ω /sq (<80% transmittance) or from 31k to 19M Ω /sq (at 95% transmittance is still much higher than that of indium tin oxide (ITO)). A few other shedding techniques endeavouring to acquire exceedingly conductive graphene sheets have additionally been acknowledged: (1) fluid stage peeling of graphite by expanded sonication, and (2) intercalation and extension of graphite with unstable agents. However, the extent of graphene sheets acquired by fluid stage peeling or intercalation of graphite is ordinarily littler than 1 μm^2 . The straightforward directing (TC) film made by such little sheets displays straight forwardness from 83 to 90% and resistance from 8000 to 5000 Ω /sq. The high resistance of these TC movies is brought on by the harm amid shedding and a lot of intersheet intersections. Here we exhibit a novel one-stage approach of acquiring superb graphene thin sheets by electrochemical shedding of graphite. The sidelong size of the peeled graphene sheets ranges from a few up to 40 μm , which altogether diminishes the quantity of intersheet intersections for making percolated TC films. The TC film made by these peeled graphene sheets displays brilliant conductivity (sheet resistance is 210 ohm/sq). The procedure was contrasted and platinum electrode changing the parameters were additionally seen of voltages, rpm and quantity of electrolyte.

Different observations made by varying the voltage parameters, electrolyte additions, sonication time and rpm of Centrifuge with respect to the resistance in ohms

Table 1: electrical analysis by varying parameters in different steps in the process.

Variations Observed In	Electrical Testing (Ω)
Voltage(1 volt for 10 min, 07 volts for 10 min)	(0.043-191)k Ω
Electrolyte (6ml H ₂ SO ₄ +150ml H ₂ O)	0.26k Ω -40M Ω
Sonication time	(0.7-175) k Ω
Rpm of Centrifuge (10 min) 1000, 1500, 2000, 2500 , 3000 and 3500	18 Ω-0.378kΩ

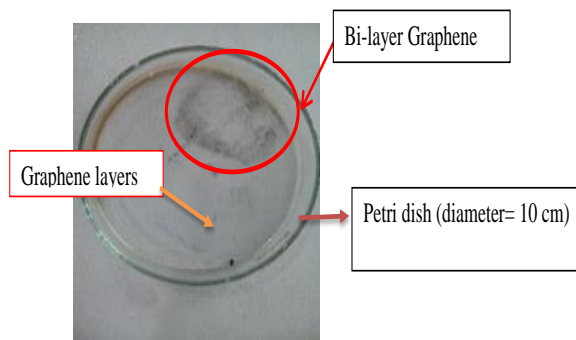


Fig.1obtained bilayer and few layer Graphene over glass substrate (petri dish) of 10 cm in diameter.

2. MATERIALS AND METHODOLOGY

2.1 Materials

Highly pure graphite electrodes were taken from electrolytic cell of the potentiostat (Gamry Instruments). N,N-Dimethylformamide (DMF or C₃H₇NO) was purchased from Zamshatra ders, Karachi. De-ionize water provided by Pakistan Council of Scientific & Industrial Research (PCSIR). Sulphuric acid (H₂SO₄, 98% purity)and Aceton by Merk Company. Filter paper and centrifuge were provided by Materials, Metallurgical and Environmental Engineering Departments. Glassware was properly washed with acetone and dried before use. Synthesis procedure is described in by Ching-Yuan. However, in this study, graphite electrodes were utilised instead of platinum. This is to reduce the cost and increase the productivity. The process schematics are shown in (Fig. 2).

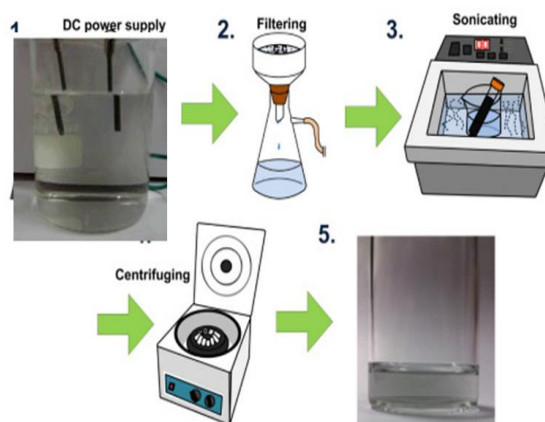


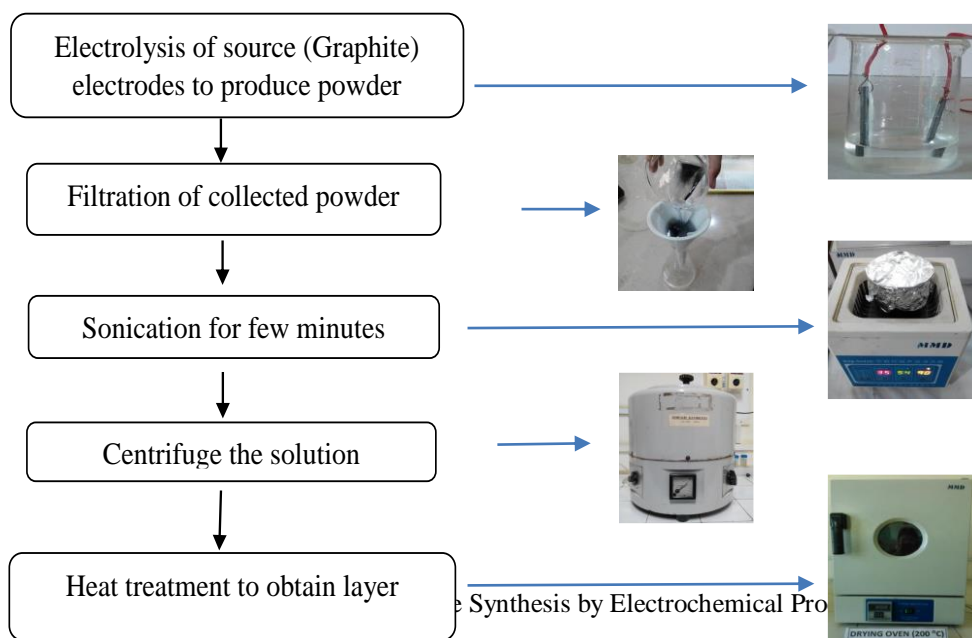
Fig.2: Electrolysis of graphite electrodes, 1) voltage applied on electrodes, 2) filtration of particles 3) sonication for few minutes, 4) centrifugation at high rpms and 5) dispersed graphene in DMF solution

Synthesize nanoscopicgraphene by employing two graphite electrodes; the graphite electrodes were attached to a copper wire via a mechanical joint. This whole anode assembly was submerged in electrolyte in such a manner that graphite is fully immersed in the electrolyte, consists 6 ml of H₂SO₄ (98%) diluted in 150ml of Deionize water, keeping the contact just above the electrolyte surface. The electrochemical exfoliation was carried out by varying the potential 5 minute exposure at 1 volt then ramping towards 7 volt for 10 minute applied by the DC supply. The application of high voltages on the positive electrode ensued in the moderate trimming of graphite through edges.

are dried in air. After drying, they were dispersed in DMF solution by gentle water-bath sonication for several minutes.

To remove unwanted large graphite particles produced in the exfoliation, the suspension was subjected to Centrifugation at 2500, 3000 and 3500 rpm for about 20 minutes which separates the constituents of a suspension on the basis of the difference in their specific gravities. The centrifuged suspension is then heated in oven at 180°C over petri dish, to obtain an interlinked layer of Graphene by evaporating the residual DMF, for 30 minutes. Graphene layer can then be used for subsequent characterization and film preparation

The exfoliated graphite flakes were collected and washed with DI water during filtration then the particles



2.3 Characterization

In this study, following testing and characterization techniques were utilized as shown in Table 2.

Table 2: characterization of graphene layer and dispersed particles by XRD, SEM, AFM, FTIR and UV

Machines/Devices	Purpose
XRD (Phillips PANalytical)	obtain the diffraction pattern of the synthesized graphene layer and nanoparticles.
SEM (Philips, Quanta 200 FEI)	morphological analysis of the end product
AFM (minus k TECHNOLOGY)	thickness measurement
FTIR (SCHIMADZU)	find the functional groups
UV-Vis (UV-VIS-1700 SHIMADZU)	absorption of graphene peak observed

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

Fig. 3 shows the diffraction pattern of dispersed particles of grapheme after drying in air shows peak at 2θ value of 26° , Fig shows the XRD pattern of driedispersegraphene layer over a glass substrate shows the amorphous pattern which assures the presence of graphene.

Fig.4 shows slight hump which concludes that the spacing is increased between the layers. In case of amorphous materials due to the lack of long range order the diffraction pattern is hump like with broad peaks which shows atoms are randomly arranged.

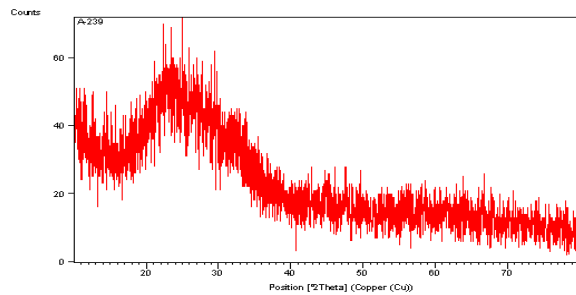


Fig.4: XRD Pattern of graphene layer over quartz substrate

3.3. SEM Analysis

For morphological and structural analysis scanning electron microscope utilized, Fig. shows the flakes of graphene, which were taken on the dual side adhesive carbon tape from the DMF solution containing dispersed graphene. Graphene sheets after high rpms of centrifuge become folded and appeared as flakes.

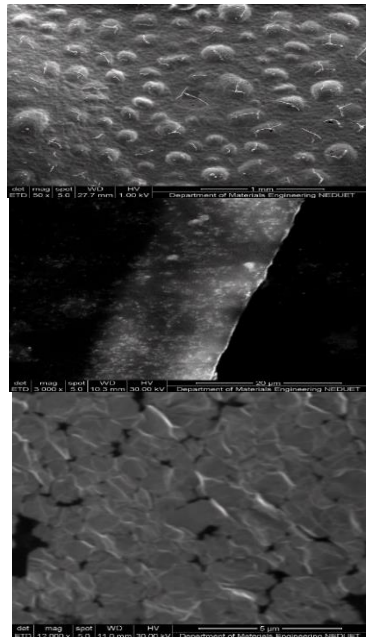


Fig.5: SEM Analysis of graphene flakes present over varying distances and continuous sheets at different magnifications.

3.4 UV-VIS Spectrophotometer Analysis

Fig.5 suggests that the peak absorption in graphene decreases with high wavelengths. At 263 nm, a peak can be noticed and one more peak around 266 nm with the different intensity of absorption peak can be observed.

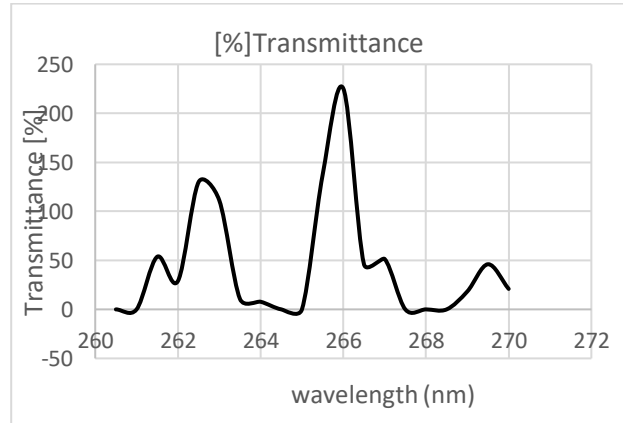


Fig.6: UV-VIS Spectrophotometer Analysis

3.5 FT-IR Spectrophotometer Analysis

The lessened aggregate reflection Fourier change infrared (ATR-FTIR) range for the graphene films appeared in Fig. 6 demonstrates the nearness of free (nonbonded) O-H(at 3536.5 and 657.0 cm^{-1}) C-O(at 858.7 cm^{-1}), O-C (at 1092 and 1251.6 cm^{-1}), and C=C (at 1655.5 cm^{-1}).

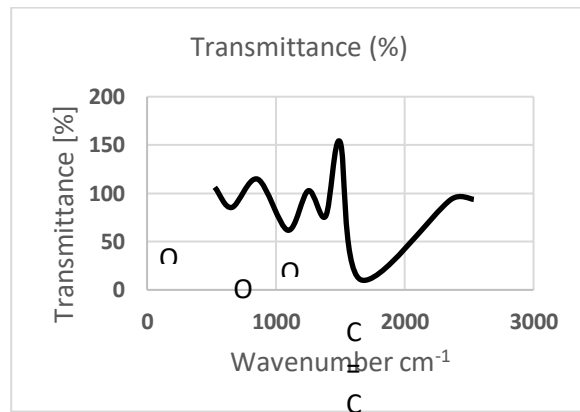


Fig.7 FT-IR Analysis of dispersed graphene in DMF

3.6 Atomic Force microscopy

Thickness of Graphene flakes has been found as 60nm.[43] Flakes were obtained over silica by heating disperse in DMF graphene at 180°C .(Fig. 7(a) demonstrates a run of the mill AFM picture for an electrochemically shed graphene thin sheet ($\sim 40 \text{ nm}$ - $8 \mu\text{m}$) drop-cast on a SiO_2 substrate. In (Fig. 7(b), the factual thickness investigation for the graphene sheet group demonstrates that the majority of the graphene sheets had a thickness lower than 90 nm and more than 65% of the sheets were more slender than 90 nm. It is noted that the aggregate yield of the graphene thin sheets is ~ 5 to 8 wt %.

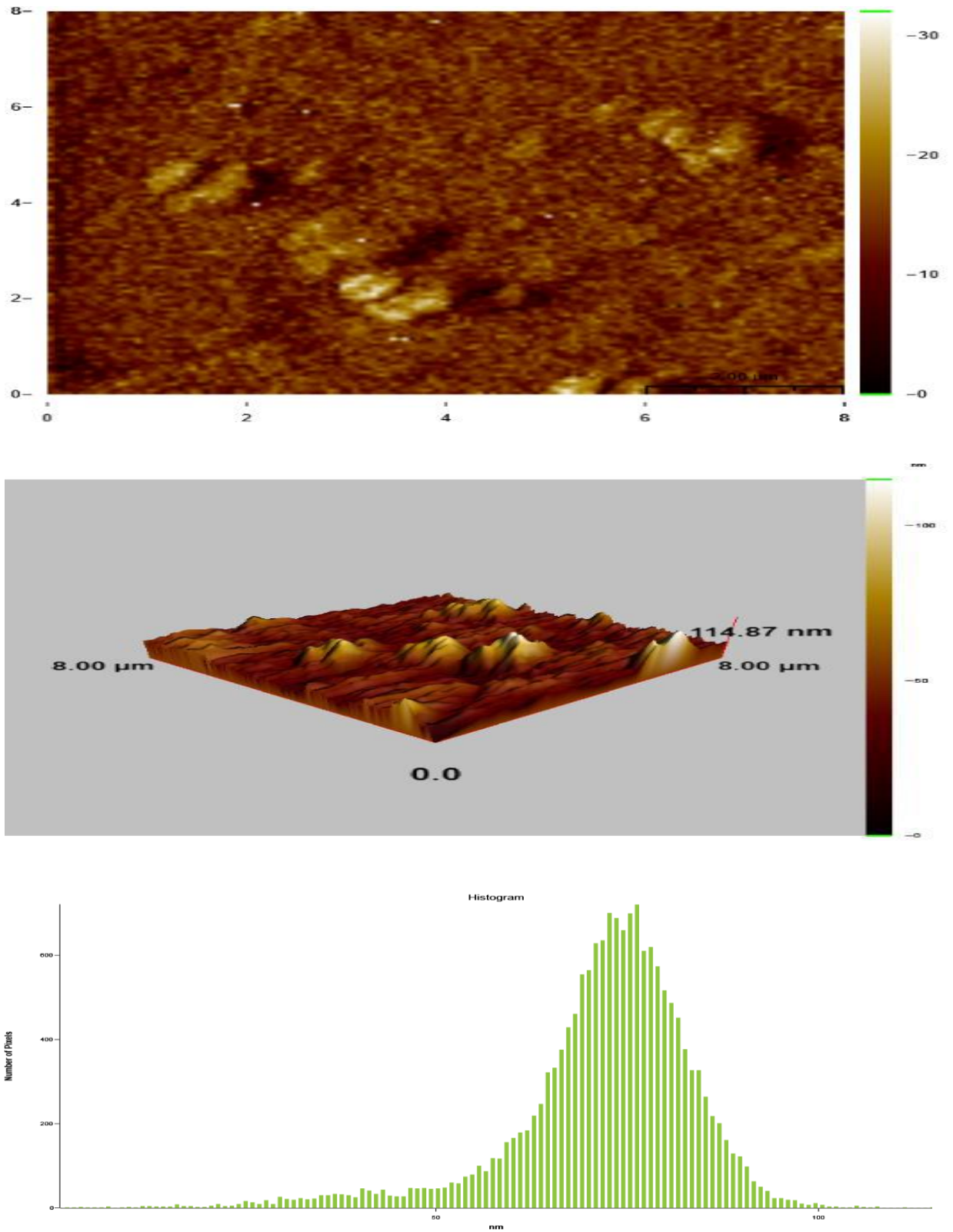


Fig. 9 (a), (b) indicate the presence of flakes at different contrasts, (c) gives the thickness of flakes and (d) scales the number of pixels with nano meter scale. AFM of Graphene layer by contact mode, thickness of flakes measured as 40nm-8μm.

4. CONCLUSIONS

All in all, a one-stage technique for acquiring high quality graphene sheets is exhibited by electrochemical peeling of graphite. Fig. 2 shows the peak near (002) plane. Fig. 4 shows the graphene thin sheets, which can be used as conductive films over any insulate substrate.

Most (>60%) of the acquired sheets are bilayer graphene with A-B stacking. The TC film made by self assembled graphene sheets displays astounding conductivity. This work gives an effective approach to acquire high quality, economical, and versatile creation of "graphene ink", which may clear a route toward future applications in adaptable gadgets. After further treatment in tube furnace under inert environment, it can provide the resistance of 22Ω/sq which qualifies this layer for dye-synthesized solar cell.

5. ACKNOWLEDGMENTS

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