CHAPTER - VII

Conclusion and Scope of Future Work

- Good physical and mechanical properties make graphene as leading material for future applications. It became subject of interest for researchers to explore and utilize its properties for various applications ranging from civil to defense and aerospace applications etc.

- High strength and modulus of graphene promises its application for structural composite reinforcement.

- High surface area of graphene is also favorable for it as being utilized for reinforcement.

- Lots of research has been done to discover physical properties of graphene. It includes semiconducting behavior, thermal conductivity, electrical conductivity and some fundamental research related to basic theory of physics.

- Fabrication techniques of alone single-layer graphene sheet have been developed by various groups. Comparatively less work has been done to demonstrate graphene as reinforcement for composite fabrication for structural and other
properties. Hence, it needs development of sounding method for synthesis of graphene sheets in bulk amount.

✓ In current research some attempts were carried out to synthesize graphene sheets in bulk amount and utilize it for composite fabrication.

✓ Bulk Graphite was used as raw material for synthesis of graphene. Various kinds of exfoliation techniques were used to separate graphene planes apart from each other. Raw materials were characterized by various techniques like silica and Fe content, particle size distribution, XRD analysis and FTIR analysis.

✓ Raw materials were subjected to cleaning for removal of impurities like silica and Fe. Graphite was washed by HF and again analyzed for silica and Fe content. Negligible amount of impurities were found which was difficult to remove.

✓ XRD and FTIR analysis shows good orientation of graphite flakes with presence of some functional groups attached to graphitic planes.

✓ In the very beginning, synthesis of graphene planes were attempted by thermal exfoliation technique. It was carried out by intercalating graphite by volatile substances which evaporates or decomposes rapidly producing plenty of gases at high temperature. Acid mixture of H$_2$SO$_4$ and HNO$_3$ was used as one type of intercalants and FeCl$_3$ was used as another type of intercalant. Intercalated graphite was exfoliated by rapid heating at high temperature mainly at 1000 °C.

✓ Synthesized samples were characterized by various techniques like volume expansion measurement, XRD and SEM etc.
✓ Exfoliated graphite by intercalating moderated amount of acid mixture blended with IPA was found to result in highest volume expansion. SEM shows very good degree of exfoliation showing good separation of planer structure.

✓ XRD characterization confirms that along with it some trace amount of planer stacking were found even upon exfoliation.

✓ In second method solution technique was used to functionalize graphitic planes of graphite where functional groups are mainly oxygen containing functionalities. Modified Hummer’s Method was utilized to get dispersion of functionalized graphene in water. Dispersion of graphene oxide (graphene with oxygen containing functionality) was characterized by various techniques.

✓ FTIR analysis shows increase in functionality as degree of oxidation increase. Modified Hummer’s Method mainly attaches carboxylic groups and phenolic groups.

✓ XRD analysis confirms significant increase in inter planer spacing of graphitic planes from 3.34 Å to 8.44 Å because of functional groups attached. Upon pyrolysis of above functionalized graphene it starts regaining its original structure of graphite.

✓ TEM analysis was carried out to observe changes in planer structure and planer stacking of functionalized graphene. It was found that graphite oxide (GO) is bunch of planes instead of single layer of graphene.
Viscometric analysis shows behavior of dispersion of functionalized graphene as polymer solution. Viscosity decrease as disruptive forces are applied to the dispersion of graphene oxide. Disruptive forces decreases viscosity up to certain level and then even upon very long time exposure to it can not make change in viscosity. This is mainly because of decrease in crystallite size with respect to time exposure to disruptive forces like ultrasonication and vigorous stirring. At certain limit there is no change in viscosity showing no further decrease in crystallite size.

To separate functionalized planes apart from each other some attempts were carried out by applying separation forces like ultrasonication, vigorous stirring and thermal exfoliation etc.

TEM micrographs show that planer structure of Graphene sheets start scrolling with external shear forces. It was observed in stirring of GO slurry.

Low temperature exfoliation was achieved by utilization of high functionality of graphene sheets. Exfoliation temperature of GO obtained by Modified Hummer’s method decreases from conventional 1000 °C to 200 °C.

XRD analysis shows absence of planer stacking showing complete exfoliation.

Surface area of exfoliated graphene by above thermal method was observed 375 m²/g by BET analysis. It is quit less than theoretical value i.e. 2630 m²/g. It is concluded that uneven agglomeration is the responsible factor for low surface area.
✓ Epoxy polymer composites were fabricated by loading various proportion of GO.

✓ Decreases in curing temperature, $\Delta H$ of curing and Glass Transition temperature are observed. These effects are assumed to be because of high functionality and high surface area correspondingly.

✓ Increase in $\Delta C_p$ can be justified by high conductivity of graphene planes.

✓ Retarding effect of graphene sheets over epoxy polymer degradation at high temperature has been observed by TGA analysis.
Scope of future work

The present work comprises of synthesis of Graphene and development of composites using these. A good amount of work was done and important conclusions have been drawn. However, research is never ending task so more work can be done in future on following lines.

✓ Interaction of graphene with matrix material can be improved by tailor making changes in functionality.

✓ Utilization of exhaustive properties of graphene in composite with epoxy as well as other matrix systems can result in more interesting research.

✓ Some extraordinary properties of graphene like quantum hall effect; semiconducting effect etc. should be explored and utilized for other application.

✓ High mechanical properties can lead graphene for good choice for reinforcement in other composites like ceramics, metals etc. for structural applications.

✓ As being high surface area material graphene can be used as good absorbent for various materials in dry and dispersion form.

✓ Some changes with functionality can lead graphene as good candidate for gas storage applications.