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SYNTHESIS OF NANOMATERIALS FOR HIGH TEMPERATURE APPLICATION

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Abstract- *In this research work we prepared nanomaterial and checked its chemical properties. The preparation of nanomaterial is done with two different methods one is fusion method and other is solution method. Two samples were prepared first is G 475 and other is G 600 which means, at 475°C and 600°C. For the characterization of nanomaterial we used XRD, Raman Spectroscopy, TGA, TEM and Elemental analysis. We have tested different chemical properties for this nanomaterial, properties such as thermal stability, electrical conductivity, Crystallanity, oil absorption value. The minimum oil absorption value of nanomaterial i.e. G-475 is found to be 11.94 gm. Both the product G 475 and G 600 shows there peak at 2Θ value is 26 and corresponding basal spacing i.e. d-spacing is at 2.035 A^0. The percentage crystallinity of both that is of G 475 and G 600 is found to be 5.57 and 5.18.These confirms the presence of graphene with fusion method, while in solution method it shows the peak at 2Θ the value is 26 and corresponding basal value that is d- spacing of the nanomaterial is 3.548A^0. The percentage crystallinity of the nanomaterial is found to be 5.10.These shows that the material is good crystalline material and having a uniform distance. The thermal analysis gives an idea about its thermal stability. The result shows that nanomaterial is stable up to 400^{0}C. But at 475^{0}C the weight loss is observed and it is less, that means the hydrogen content is more. At 600^{0}C the weight loss is more means the hydrogen content is less and the carbon content is most with small amount of hydrogen content. Raman Spectroscopy shows the graph between the range of 1560 cm^{-1} and 1360 cm^{-1}, it indicates that the carbon material is present in the nanomaterial and it has exactly the peak as like of graphene. We tested electrical conductivity of both the nanomaterial it shows that they have electrical conductance. The nanomaterial G 600 has a greater conductance than G 475.The TEM images shows the 2 D membrane structure. Both the samples G 475 and GS 600 are closely pack to each other and there is no transparency found in the material.*

Keywords- Graphene; nanomaterials; high temperature;fusion;solution;

"I. INTRODUCTION"

The world is heading to have smaller and smaller things as well as the weight of the material should also be less. The strength of the material should be high so that it will be strong and will give us good properties. Nano scale sized has the potential to become the material for the future, due to its extraordinary thermal and electrical properties [1]. Silicon has provided the better properties in the field of electronics due to which the various modifications could be possible in the field of electronics. But now silicon properties has come to the limitations, scientists are finding it difficult to reduce the size of silicon as it provides the frequency in the gigahertz, so it is difficult to use silicon, therefore the world is heading towards the nanomaterial. With the help of nanotechnology we can improve the properties and functions of the materials[3]. Nanostructure science and technology is a broad field of research and development and it has been booming in recent past few years. The nanomaterial has brought the revolutionary changes in the material as well as in the final products and it also helps to give us the various ranges of properties in the material[2]. Nanomaterial are also interesting because at these level also they gives us the property such as optical, magnetic, electrical and other such properties which are emerging, these emerging properties are helping us to have greater impacts on the fields of medicine, electronics and other such fields

"II. LITERATURE REVIEW"

Carbon has 4 valence electrons which is having very similar energies. Therefore these electrons give rise to 2s, 2px, 2py and 2pz orbital's, the 2 inner shell electrons belong to a spherically symmetric 1s orbital that is tightly bound[3]. The unique ability to hybridize sets carbon apart from other elements and allows carbon to form 0D, 1D, 2D and 3D structures. Graphene and graphite are the two dimensional sp^2 hybridized forms of carbon. Graphite is a layered material formed by stacks of graphene sheet separated by 0.3 nm and held together by weak Vander walls forces[5]. Graphene is a hexagonal structure with each atom forming 3 bonds with each of its nearest neighbours'. It gives similar mechanical and thermal properties as diamond. Bulk graphite was studied from decades, but study on graphene was not done as it is very difficult to separate single layer of graphene[4]. Nanotechnologies involve designing and producing objects or structures at a very small scale, on the level of 100 nanometers (100 millionth of a millimeter) or less[6]. Nanomaterials are one of the main products of nanotechnologies as nano-scale particles, tubes, rods, or fibers. Nanoparticles are normally defined

as being smaller than 100 nanometers in at least one dimension. Graphene is a flat monolayer of carbon atoms tightly packed into a two-dimensional (2D) honeycomb lattice, and is a basic building block for graphitic materials of all other dimensionalities[7]. It consists of a hexagonal array of sp^2-bonded carbon atoms, just like those found in bulk graphite. 2D materials display very interesting properties, and are fundamentally different from the 3D materials we encounter every day. The discovery of 2D materials means that scientists now have access to materials of all dimensionalities, including 0D (quantum dots, atoms) and 1D (nanowires, carbon nanotubes) [8]. Chemical activation has been shown as a very efficient method to obtain carbons with high surface area and narrow micropore distribution. Among all the chemical activation agents, alkaline hydroxides as KOH or NaOH are reported to be highly interesting from the performance point of view, allowing activated carbons to be prepared from many kinds of carbonaceous precursors[9]. Although it is a frequently used process to prepare activated carbons, the general mechanism of chemical activation is not well understood, and the various interpretations found in literature underline the process complexity. In general terms, chemical activation by alkalis consists in solid–solid or solid–liquid reactions involving the hydroxide reduction and carbon oxidation to generate porosity[4]. During the reactions, CO, $CO_2$and H_2 evolution is observed, and additional reactions between active intermediates produced on the surface and the constituents of the gas phase are possible. A recent study suggests that the carbon/MOH (M = Na or K) reaction mechanism is independent of the hydroxide used and consists in the overlapping of redox processes[5].

"III. EXPERIMENTAL WORK"

The mixture of rosin and sodium hydroxide was taken in pestle and mortal. The ratio was decided on the basis of their molecular weight that is of rosin and sodium hydroxide. The mixture of rosin and sodium hydroxide was taken in the various ratios of 1:1, 1:2, 1:3, and 1:4 on weight basis. After characterization it was observed that the best result was obtained at the ratio of 1:4 weight bases. The rosin was taken 2 gm and the sodium hydroxide in 1:1 it was 2 gm, in 1:2 it was 4 gm, in 1:3 it was 6 gm, in 1:4 it was 8 gm. The conclusion of the mixing ratio was done on the trial basis that is the material was taken to characterization and the ratio which give the required result was consider as the best ratio. Then the mixture of rosin as well as of sodium hydroxide was crushed and formed it in the powder form. Then the crushed or the powder form of rosin as well as sodium hydroxide was loaded into the crucible and the crucible was loaded into the muffle furnace and it is indicated as G 475 and G 600. The nickel crucible which was loaded in the muffle furnace, the temperature was initially kept at 350^0C for 24 hours. Then the temperature would be increase by 25^0 C, after every 4 hours. This procedure was continued up to the temperature would not reach up to 600^0C[4]. As the temperature would reach 600^0C, the muffle furnace will be turn off, and the material will be allowed to cool up to room temperature. Then the crucible would be discharge from the furnace. Then the material which is present in the crucible is taken for washing and as well as for drying purpose. The washing and drying is also main so that there will be no impurity present in the material and the material will be good to use. The material will be observed in powder form as well as its purely black colour material. The black powder is formed in the muffle furnace which is being taken out when the muffle furnace is brought back to room temperature and then the nickel crucible are taken out from muffle furnace. The material which is taken out from the muffle furnace and each nickel crucible is then is kept into beaker with the water, where it is kept for near about 24 hours[6]. Then the nickel crucible is taken out from beaker and the remaining material is filtered out and then it is being used for the further process. The filter paper is then used or being kept in the oven and then it is dried. The dried material is the black material which is used for the further testing or as filler in the composite.

"IV. RESULT & DISCUSSION"

A. Oil Absorption

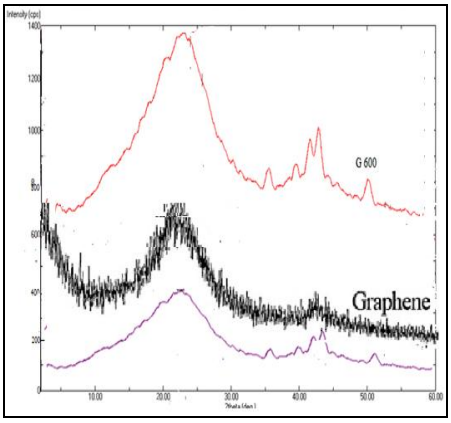
"Table 1. Oil absorption values of samples"

Sr.No.	Material	Oil Absorption
1	G 475	11.94
2	G 600	10.78
3	GS 600	10.14

The oil absorption is the process in which specific amount of the material is taken in the petridish and drop by drop the oil is being pour into the petridish and mixing of the sample is done by the help of spatula. The oil drops are being added in the material until the whole material gets wet in the petridish and the amount required is minimum to weight the whole material. The oil drop is added until the whole material is getting weight and the lumps are being formed in the petridish. It has been calculated as the weight of oil beaker initially taken and the weight of oil beaker finally taken[2]. The difference found out between the initial weight and final weight is the oil absorption value of the material. The minimum or the oil absorption value of my material is found to be 11.94 gm.

B. X-Ray Diffraction (XRD)

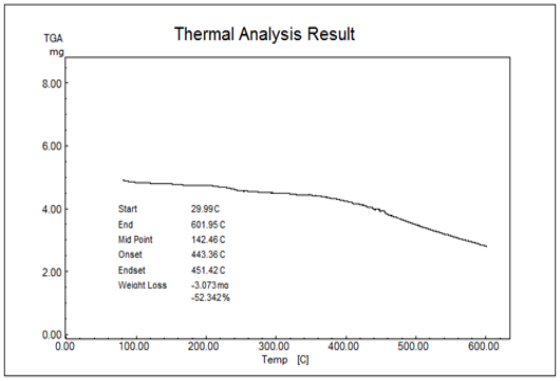
The figure below of X-Ray diffraction patterns shows that the product prepared is at various temperatures that are at 475^0C and 600^0C. They are name as G 475 and G 600. Both the product G 475 and G 600 shows there peak at 2Θ value is 26 and corresponding basal spacing i.e. d-spacing is at 2.035 A^0. The percentage crystallinity of both that is of G 475 and G 600 is found to be 5.57 and 5.18.These show that both the graphs i.e. G 475 and G 600 are showing similar graphs to that of graphene whose graph is shown in below figure[5]. But the graph of G 475 has been shifted earlier that might because the impurity present in the material or the material would not be formed fully. The material is having a good crystalline structure and shows that there is good crystallinity in the material so for the further applications it is unable to affect the order of the structure. The third figure shows the graph of X-Ray Diffractometer which has been characterize by the second preparation of method that is by solvent base method, but it also shows there peak at 2Θ the value is 26 and corresponding basal value that is d- spacing of the material is 3.548A^0 . The percentage crystallinity of the material is found to be 5.10. These shows that the material is good crystalline material and having a uniform distance so that at the end application it will not change the properties and give the strength to the end material.



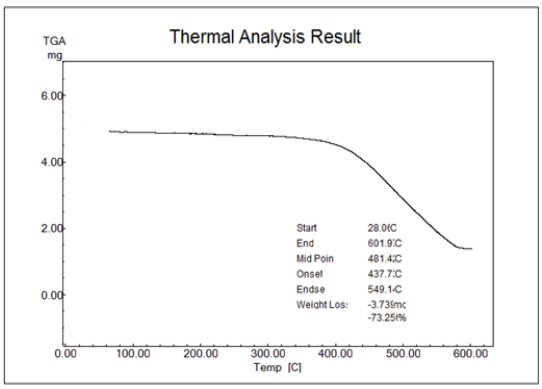
"Figure 1. XRD Graph Comparison with Graphene"

C. Thermo Gravimetric Analysis (TGA)

The thermogravemetric analyses of two samples were analyzed. One at 475^0C and other one is analysed at 600^0C. The thermal analysis at 475^0C as well as 600^0C is stable and it is straight line. The line is stable up to 400^0C that is that these materials is stable at higher temperature, but at 475^0C the weight loss is less that means the hydrogen content is more but at 600^0C, the weight loss is more means the hydrogen content is less[7]. It tells us that as the temperature increases the weight loss are more and the hydrogen content is lesser and lesser means the carbon content will be more and the material will be strong.



" Figure 2. Result at G 600 "



"Figure 3. Result at G 475"

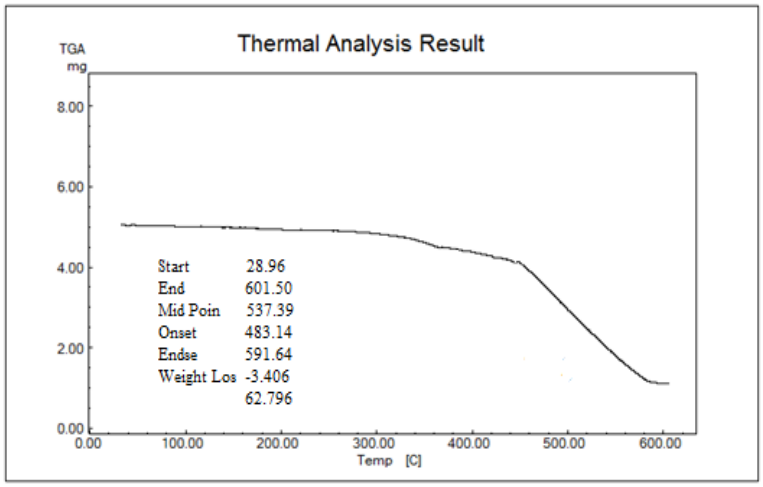


Figure 4. Result at GS 600

D. Elemental Analysis

The elemental analysis is a test which is used to check the rate of carbon, hydrogen and nitrogen in the sample. The following table shows us that the carbon content is most and the hydrogen content is also present but in very less percentage in both that is in G 475 and G 600. The retention time is near about same in both the samples. The nitrogen and hydrogen content get less as the temperature increases[3]; it indicates that at the higher temperature the concentration of carbon is increasing gradually. It means that at 475^0C, the content of nitrogen and hydrogen is more as compared to the material at 600^0C. The carbon structure goes strong on higher temperature.

"Table No.2. Elemental Analysis of G 475"

Sr.No.	Compounds	Percentage Content (%)
1	Nitrogen	1.158
2	Hydrogen	3.141
3	Carbon	65.48

"Table No. 3. Elemental Analysis of G 600"

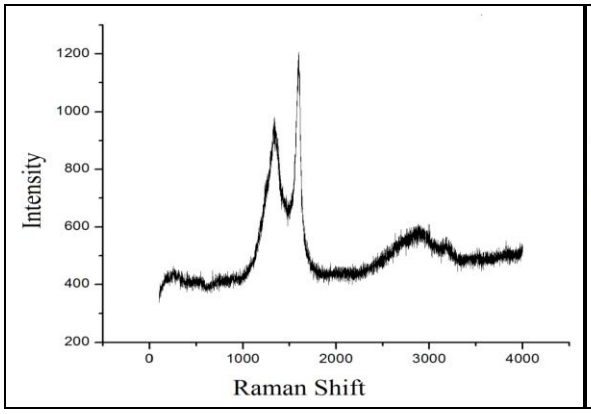
Sr.No.	Compounds	Percentage Content (%)
1	Nitrogen	0.995
2	Hydrogen	3.284
3	Carbon	71.677

"Table No. 4. Elemental Analysis of GS 600"

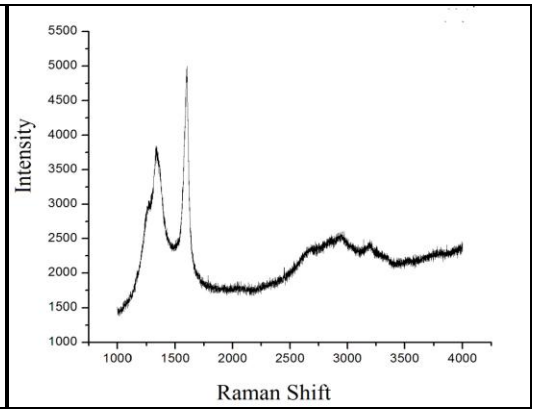
Sr. No.	Compounds	Percentage Content (%)
1	Nitrogen	0.876
2	Hydrogen	3.563
3	Carbon	78.967

E. Raman Spectroscopy

The below figures shows us the graphs of Raman spectra at the various temperature at 475^0C and 600^0C. The main features in Raman spectroscopy of carbons are the so called G and D peaks which lie at around 1560 and 1360 cm^{-1} for visible excitation. The below figures shows the graph between the range of 1560 and 1360 cm^{-1}, it indicates that the carbon material is present in the material and it have exactly the peak as like of graphene. Recently the characterization of the material is mainly done by Raman spectroscopy because it gives us the accurate result and helpful to determine the peak[8]. The peaks in the material at 475^0 and 600^0C are raising and giving clear indication of the material that the carbon material is formed.



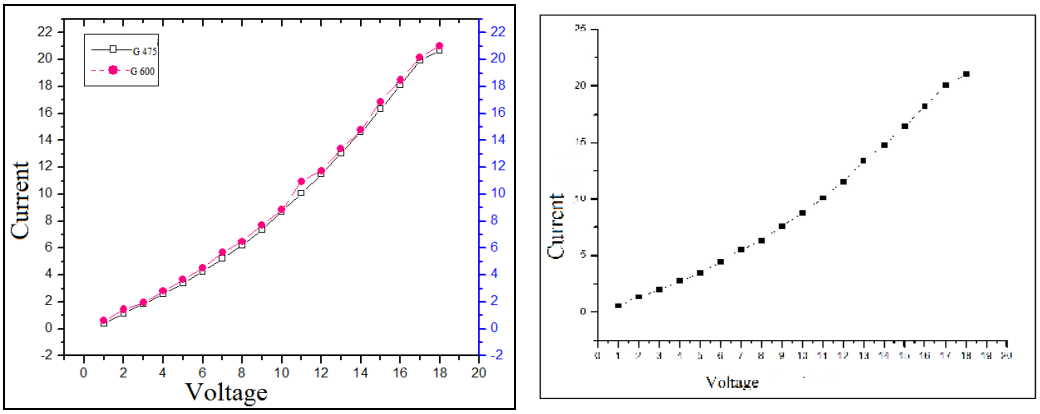
"Figure 5. Raman Spectroscopy of G 600"



"Figure 6. Raman Spectroscopy of G 475"

F. Electrical Conductivity

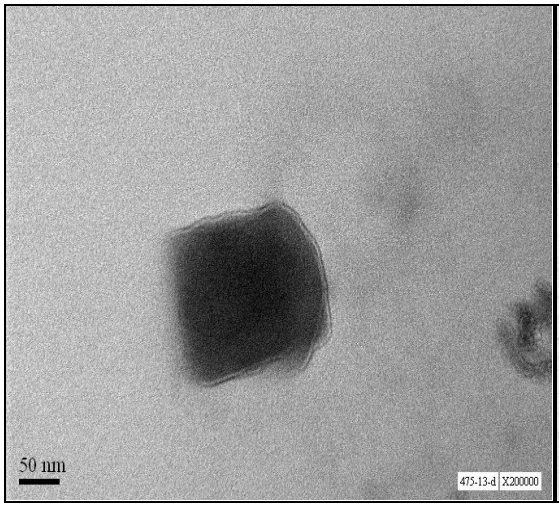
The electrical conductivity is the testing in which we test the electrical properties of the material whether it is having conductance or not and where it is applicable at electrical applications or not. The result of the conductance in given figure gives us the idea of the conductance of the material. The material which shows the black line indicates the material of G 475 and in pink it indicates the line of G 600 material. Both the material shows that they have electrical conductance. But the G 600 has a greater conductance than G 475, it might be because of some carbon carbon bonding and the presence of sodium hydroxide, the conductivity has been increased in higher temperature. Therefore the conductance is high and can be used in electrical applications.



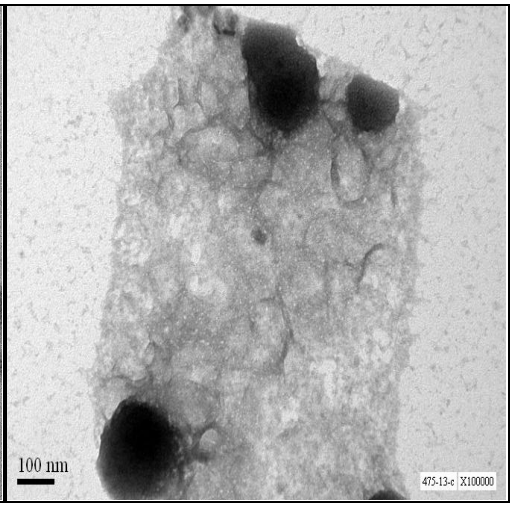
"Figure 7. Electrical Conductivity of G 475,G 600 and GS 600"

G. Transmission Electron Microscopy

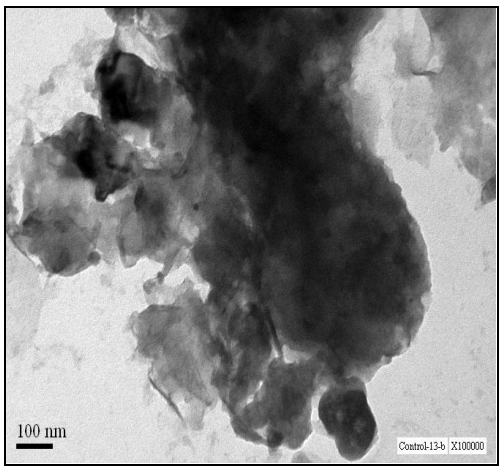
The below figure shows the large dark flakes of the material. It cannot be exfoliated further as the machine cannot be focusing more than the reported values in the figure. While in the second figure we can see that crystalline structure in the images. It exhibits the typical wrinkled structure. The sheets are transparent and entanglement at low magnification it look flats as well as transparent and at some parts it looks restacked. It gives or shows the 2 D membrane structure[4]. The both sample was of G 475 and in the GS 600 the material are closely pack to each other and though the magnification the material is high but they are closed to each other and there is no transparency in the material.



"Figure 8. TEM Images of G475"



"Figure 9. TEM Images of G600"



"Figure 10. TEM Images of GS600"

"V. Conclusion"

On concluding remark of this research work, we prepared samples by two different methods and characterized it. On comparing the results of characterization we obtained that the percentage crystallinity of both samples G 475, G 600 and GS 600 is found to be 5.57 and 5.18. The material is good crystalline material and having a uniform distance so that at the end application it will not change the properties and give the strength to the end material. The TGA analysis says that as the temperature increases the weight loss is more and the hydrogen content is less. And less hydrogen content means the carbon content is more and the material is much strong. At 475^0C, the content of nitrogen and hydrogen is more as compared to the material at 600^0C.The TEM results tells us that both the samples are crystalline in nature. The oil absorption value of material is found to be 11.94 gm.

"VI. References"

[1] Yamashita Y, Ouchi K. "Influence of alkali on the carbonization process. I. Carbonization of 3, 5-dimethylphenolformaldehyde resin with NaOH". Carbon 20:41–5, 1982

[2] Lillo-Rodenas MA, Cazorla-Amoros D, Linares-Solano A; "Understanding chemical reactions between carbons and NaOH and KOH: an insight into the chemical activation mechanism" Carbon; 41; 267–75, 2003

[3] Raymundo-Pinero E,Cazorla-Amoros D,Linares-Solano A, Delpeux S,Frackowiak E,Szostak K, et al. "High surface area nanotubes prepared by chemical activation." Carbon; 40:1614–7, 2002.

[4] Guiqiang Wang, Wei Xing, Shuping Zhuo "The production of polyaniline/graphene hybrids for use as a counter electrode in dye-sensitized solar cells"; 2012; February 1.

[5] Shailesh K. Dhoke, A.S. Khanna "Electrochemical impedance spectroscopy (EIS) study of nano-alumina modified alkyd based waterborne coatings"; 2012; January 17.

[6] Otowa T, Tanibata R, Itoh M. "Production and adsorption characteristics of maxorb: high surface area carbon.
Gas Sep Purify"; 7:241–5. 1993.

[7] Geim A. K. And Novoselov K. S.; "The Rise of Graphene; Nature Materials"; 6, 2007

[8] Lillo-Rodenas MA, Juan-Juan J, Cazorla-Amoros D, Linares-Solano A; "About reactions occurring during chemical activation with hydroxides"; Carbon; 42:1371–5. 2004

[9] Marsh H, Yan DS, O_Grady TM, Wennerberg AN. "Formation of active carbons from cokes using potassium
hydroxide." Carbon; 22:603–11. 1984

