

PHYSICAL AND CHEMICAL PROPERTIES OF GRAPHITE

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Science in Chemistry

by

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DEPARTMENT OF CHEMISTRY

SCHOOL OF SCIENCE AND HUMANITIES

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

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BONA FIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **KALEEM ASHRAF (39030010) MONESHKUMAR R (39030014)** who carried out the project entitled "**PHYSICAL AND CHEMICAL PROPERTIES OF GRAPHITE**" under our supervision from December 2021 to April 2022.

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ABSTRACT

Graphite is one of the three crystalline kinds of the detail carbon; the alternative two being diamond and fullerenes (which encompass carbon nanotubes and graphene). Graphite happens naturally within the earth and, beneath wellknown situations, is the maximum stable form of carbon. This mineral is used in a various range of applications from pencils to lubricants and batteries to different greater extreme applications, such as nuclear fusion. Graphite is a evidently going on shape of the element carbon. It is black to metal gray in color, opaque, and with a distinct tender lubricative texture. Graphite famous two crystalline structures; hexagonal (alpha) and rhombohedral (beta). Both crystalline systems showcase a high diploma of anisotropy, which closely determine graphite's properties, mainly electric and mechanical. Here, we explained about Graphite, physical properties, chemical properties, mechanical properties, thermal properties, characterization techniques, results

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LIST OF ABBREVIATION

FTIR	Fourier Transform Infrared Spectroscopy
BET	Braeuer-Emmette-Teller

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CHAPTER 1

INTRODUCTION

1.1 GRAPHITE

Graphite, also referred to as plumbago or black lead, mineral inclusive of carbon. Graphite has a layered shape that includes earrings of six carbon atoms organized in widely spaced horizontal sheets. Graphite hence crystallizes inside the hexagonal gadget, in contrast to the identical element crystallizing within the octahedral or tetrahedral system as diamond. Such dimorphous pairs usually are rather comparable of their physical residences, but not so in this case. Graphite is darkish grey to black, opaque, and very smooth (with a hardness of 1 half on the Mohs scale), at the same time as diamond can be colorless and transparent and is the hardest evidently occurring substance.

Graphite is formed by means of the metamorphosis of sediments containing carbonaceous fabric, by using the reaction of carbon compounds with hydrothermal answers or magmatic fluids, or likely via the crystallization of magmatic carbon. It occurs as isolated scales, massive loads, or veins in older crystalline rocks, gneiss, schist, quartzite, and marble and also in granites, pegmatites, and carbonaceous clay slates. Small isometric crystals of graphitic carbon (possibly pseudomorphs after diamond) observed in meteoritic iron are known as cliftonite.

1.2 STRUCTURES OF GRAPHITE

Graphite is composed of layers of carbon atoms which are arranged in six-membered, hexagonal rings. These jewelry are connected to each other on their edges. Layers of fused rings may be modeled as an limitless series of fused benzene rings (with out the hydrogen atoms). Carbon atoms in those ring arrays are inside the sp^2 -hybridized kingdom. In the sp^2 molecular orbital model each carbon atom is connected to a few different species, 3 other carbon atoms within the case of graphite. In this bonding mode the bond perspective between adjacent carbon atoms is one hundred twenty. These “ring arrays” are organized in huge sheets of carbon atoms, and man or woman sheets are referred to as graphene layers. The carbon-carbon bond length in a layer aircraft is 1.418. Graphene layers are stacked one on pinnacle of every other parallel with the “C” crystallographic axis of the hexagonal 4-

axis system wherein graphite crystallizes.

Graphene layers are exactly “indexed” so that an actual 3-dimensional repeating order is developed and maintained at some point of the crystal (as is required for any crystalline stable). In this association each graphene layer is listed to the layer above and the layer beneath in any such manner as to shape a repeating alignment every second layer. In different words, the carbon atoms within the first layer indexed are positioned in specific alignment with the carbon atoms of the 1/3 layer listed. Correspondingly, the carbon atoms of the second one graphene layer are in alignment with the atoms of the forth layer. This configuration is known as an “ABABAB” structure. All the “A” layers are aligned with different “A” layers, and all of the “B” layers are aligned with different “B” layers. A simplified view of this version is to assume the “A” layer constant in area. Then align the “B” layer in order that without delay underneath the middle of every “A” layer hexagon there is placed, useless center a carbon atom within the “B” layer.

by means of the graphene layer you were on. In diamond, the whole diamond molecule may be traversed, in a similar style, in any path, up, down, or aspect-to-facet so that any carbon atom can be reached from any other carbon atom the usage of “sigma bond bridges” to traverse the route.

1.3 PHYSICAL PROPERTIES OF GRAPHITE

The residence of the right graphite fabric that most carefully corresponds to an infinitely large graphite crystal, are reviewed on this section. Such a fabric does not exist inside the actual global and the homes given beneath or both calculated or based on the actual residences of graphite crystals closely drawing this close this ideal shape as already noted and as maybe seen in later chapters , a extensive variety of substances comes under the heading of carbon or graphite and those substances regularly have properties which can be good deal one of a kind from those of the perfect graphite crystal. Obviously its miles necessary to outline the material as it should be whilst talking of the houses of carbon.

1.4 SUMMARY OF PHYSICAL PROPERTIES

The bodily properties of graphite are summarized in below should be stressed that to gain correct measurements of the residences of materials a lot above 3000 K is a attempting undertaking. In the case of graphite ,lots of these measurements are based totally on carbon-arc experiments difficult to carry out and interpret. The consequences must be viewed hence and some of those outcomes and conclusions are nevertheless controversial.

- Physical properties of Graphite
- Crystalline form : Hexagonal
- Color : Black
- Density at 300 K, 1 atm :2.26 g/cm³
- Atomic Volume :5.315 cm³/mol
- Sublimation point at 1 atm (estimated): 4000 K
- Triple point (estimated): 4200 K
- Heat of fusion: 46.84 kJ/mol
- Boiling point (estimated): 4560 K
- Heat of vaporization to monoatomic gas (estimated): 716.9 kJ/mol
- Pauling electronegativity: 2.5

1.5 DENSITY

The density of the precise crystal listed in Table 1.1 is the theoretical density. Most graphite materials could have lower densities due to the presence of structural imperfections together with porosity, lattice vacancies and dislocations. With the exception of boron nitride, graphite materials have a decrease density than all different refractory substances as shown in Table 1.2. This is a advantageous function mainly in aerospace applications.

1.6 MELTING, SUBLIMATION, AND TRIPLE POINT

The melting factor of a crystalline material consisting of graphite is the temperature at which the strong country is in equilibrium with the liquid at a given strain. "Normal" melting factor happens at a stress of one ecosystem .Graphite does no longer have a normal melting factor on account that, at one surroundings ,it does not soften however sublimates when the temperature reaches about 4000 K.

The triple factor (where all 3 stages, solid, liquid, and gasoline, are in equilibrium) is completed, with the aid of latest estimates, at a temperature of 4200 K and a strain of a hundred atm, as shown inside the vapor-pressure curve of Fig. 1.1. A incredible deal of uncertainty still remains regarding those values of pressure and temperature, reflecting in element the difficulty of engaging in experiments under such extreme situations. The onset of sublimation and the melting temperatures are apparently close. At temperatures above that of the triple point and at pressures of argon extra than one hundred atm, a aggregate of strong and liquid carbon is detected.

Graphite can accordingly be considered the most refractory of all the factors, tungsten being second-high-quality with a melting point of 3680 K. However hafnium carbide (HfC) and tantalum carbide (TaC) are stated to have better melting points (about 4220 K and 4270 K respectively) and are the most refractory of all materials.

1.7 THERMAL PROPERTIES OF GRAPHITE

1.7.1 Summary Of Thermal Properties

The physical residences reviewed in the preceding phase are essentially unaffected through the dimensions and orientation of the crystallites within the aggregate (excluding density). As a result, they may be considered legitimate for all styles of graphite. This is not genuine for a few of the residences listed in this and the subsequent sections, and these houses may additionally range extensively depending on crystallite size and orientation and different elements associated with the processing conditions.

Heat of combustion Del Hco at 25 deg C	393.13
Standard entropy S at 25 deg C,J/mol-K	5,697 – 5.743
Entropy Del S 298, J/mol – K	152.3
Specific Heat at 25 Deg C kJ/kg-K	0.690 – 0.719

Table 1.1 Theoretical and Thermal Properties of Graphite

1.7.2 Heat Capacity (Specific Heat)

The molar heat capacity (specific heat) of graphite is reported as 8.033 - 8.635 J/mol-K at 25°C. [8H 1 °] As with all elements, it increases with temperature, with the following relationship (T in deg K)

$$C_p = 4.03 + (1.14 \times 10^{-3})T - (2.04 \times 10^{-5})T^2$$

The specific heat will increase rapidly with temperature, up to 1500 K in which it degrees off at about 2.2 kJ/kg-K as proven in Fig.1.2. It is believed to be extraordinarily insensitive to the differences among the numerous grades of artificial graphite, and the spread of values determined within the literature may be attributed to experimental versions.

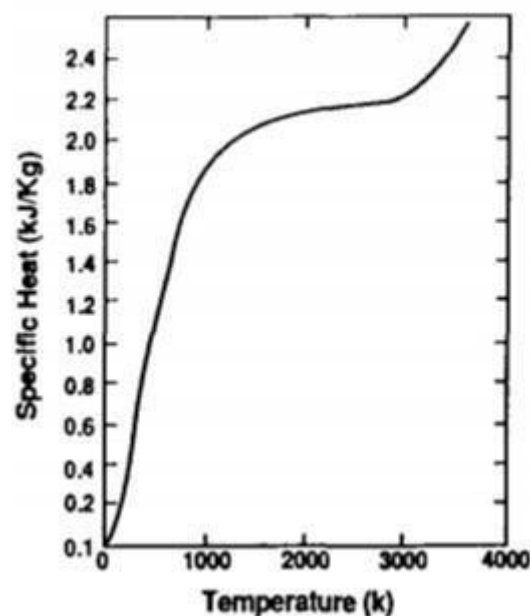


Fig 1.1. Specific heat of graphite vs. temperature at one atmosphere.

Graphite	0.690 – 0.719
Diamond	0.502 – 0.519
Boron	1.025
Aluminum	0.900

Table 1.2 Specific heat of Selected Elements

1.7.3 Thermal Conductivity

The thermal residences of conductivity and enlargement are strongly motivated via the anisotropy of the graphite crystal. The thermal conductivity (K) is the time charge of transfer of warmth by conduction. In graphite, it occurs essentially by lattice vibration and is represented via the subsequent courting (Debye equation):

$$K = bCpvL$$

where: b = a constant

C = specific heat per unit volume of the crystal

v = speed of heat-transporting acoustic wave (phonon)

L = mean free path for wave scattering

In a polycrystalline substances, the waves (phonon, i.e., quantum of thermal strength) are scattered through crystallite limitations, lattice defects, and different phonons. Little of this takes place in a perfect or close to-perfect graphite crystal in the basal aircraft and, as a end result, the thing L is excessive and thermal conductivity is high within the ab guidelines.

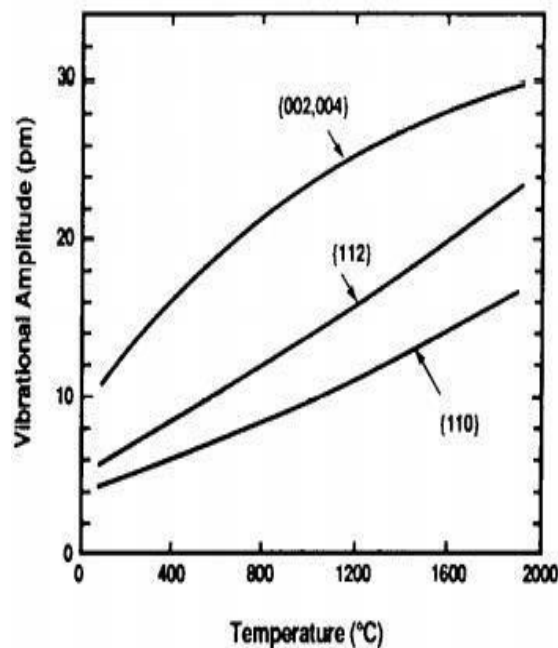


Fig 1.2. Thermal vibrational amplitude of graphite crystal

1.8 ELECTRICAL PROPERTIES OF GRAPHITE

In electrical conductors such as metals, the attraction between the outer electrons and the nucleus of the atom is weak; the outer electrons can move readily and, since an electric current is essentially a flow of electrons, metals are good conductors of electricity. In electrical insulators (or dielectrics), electrons are strongly bonded to the nucleus and are not free to move.^[18K 19] Electrically, graphite can be considered as a semi-metal, that is a conductor in the basal plane and an insulator normal to the basal plane. Its atomic structure is such that the highest-filled valence band overlaps the lowest-empty conduction band by approximately 36 meV and the delocalized fourth-valence electrons form a partially-filled conduction band between the basal planes where they can move readily in a wave pattern as they respond to electric fields.^[2°] Consequently, the electrical resistivity of graphite parallel to the basal planes (ab directions) is low and the material is a relatively good conductor of electricity. In the c direction, the spacing between planes is comparatively large, and there is no comparable mechanism for the electrons to move from one plane to another, in other words, normal to the basal plane. As a result the electrical resistivity in that direction is high and the material is considered an electrical insulator. In some cases, it may be 10,000 times higher than in the ab directions.^{^20'} Often quoted

resistivity values are $3000 \times 10^{-6} \text{ ohm.m}$ in the c direction and $2.5 - 5.0 \times 10^{-8} \text{ ohm.m}$ in the ab directions.

1.9 MECHANICAL PROPERTIES OF GRAPHITE

The bond between atoms within the basal plane of a graphite crystal is considerably stronger than the bond between the planes with an anisotropy ratio of approximately 75. This means that, while the strength in the ab directions is considerable, that in the c direction (interlaminar strength) is low and graphite shears easily between basal planes. The elastic constants are

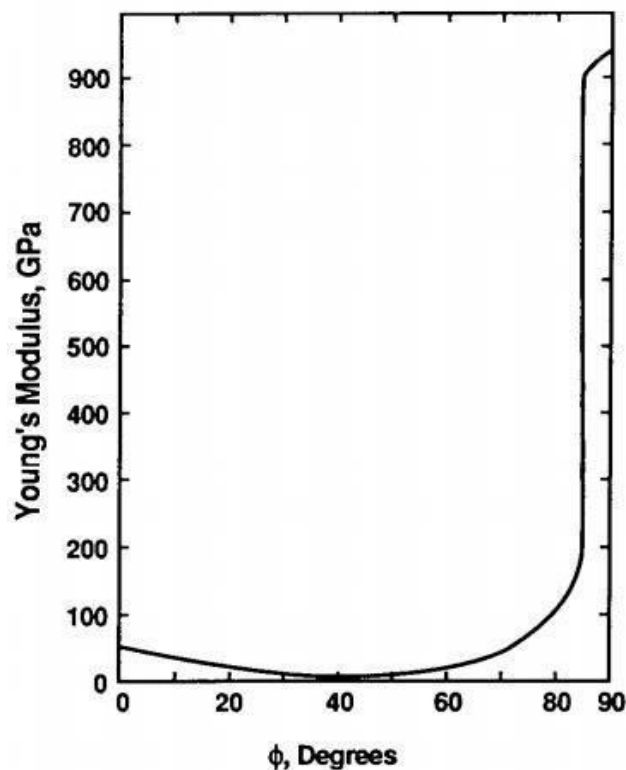


Fig 1.3 Young's modulus of graphite crystal

It ought to be mentioned that such values are those of the ideal graphite crystal. The mechanical homes of "real" cloth, which includes the molded graphite substances and pyrolytic graphite are significantly extraordinary because they may be controlled by the dominant crystallite orientation, porosity, structural defects and other elements. The result is massive variations within the variety of these residences, depending on kind of graphite and procedure.

1.10 CHEMICAL PROPERTIES OF GRAPHITE

Pure graphite is one of the most chemically inert materials. It is resistant to maximum acids, alkalies and corrosive gases. However impurities are nearly always present to some diploma in each natural and artificial graphites and often have an vital catalytic impact with resulting boom within the chemical reactivity.

The anisotropy of the graphite crystal is pondered in its chemical behavior. Reaction with gases or vapors occurs preferentially at "energetic web sites", i.e., the end of the basal planes of the crystal which can be the zigzag face 101 and the arm-chair face 112 and at illness\ websites, inclusive of dislocations, vacancies, and steps. Reaction with the basal plane surfaces is far slower.

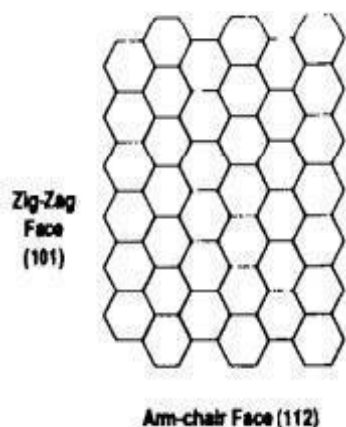


Fig 1.4 The faces of a graphite crystal

Reactivity also generally increases with increasing temperature and, at high temperatures, graphite becomes far more reactive. For instance, above 450°C, it oxidizes readily with water, oxygen, some oxides, and other substances. In this section, only the general chemical behavior of graphite will be considered. Reviews of the chemical reactivity of specific graphite materials will be given in subsequent chapter.

CHAPTER 2

LITERATURE SURVEY

1. TITLE : Chemistry and Physics of Carbon

AUTHOR : (P. L Walker, Jr., ed.), Vol. 4, Marcel Dekker Inc., Palmer, H. B. and Shelef, M.

JOURNAL AND YEAR : New York (1968)

INFERENCE : About physical and chemical properties of Carbon.

2. TITTLE : Carbon/Graphite Properties.

AUTHOR : Eggers, D. F., Gregory, N. W., Halsey

JOURNAL AND YEAR : 1987

INFERENCE : About Graphite and Carbon Properties

3. AUTHOR : S. Stankovich, D. A. Dikin, R. D. Piner, K. A. Kohlhaas, A. Kleinhammes, Y. Jia, Y. Wu, S. T. Nguyen, and R. S. Ruoff, Carbon **45**, 1558

JOURNAL AND YEAR : (2007)

4. TITLE: Properties of silicon rubber.

AUTHOR : Gianqian zhu, zhenhao wang, hui zeng, Hun-Sik Kim, Seong-Hun Jung, Jin-San Yoon

JOURNAL AND YEAR: Journal of applied polymer science 2021.

INFERENCE: Silicon rubber using for thermal properties studied and sample using characterization techniques like thermal conductivity.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The main aim of this project is to characterization of physical and chemical properties of Graphite. Graphite were characterized with the help of FTIR, Raman Spectroscopy, BET ,Density test and Electrical Conductivity test.

3.2 SCOPE

- Density test by Displacement method using Gas pycnometer
- Characterization of Graphite by FTIR, Raman spectroscopy
- Characterization of Graphite by BET
- Electrical Conductivity of carbon nano particles and mixing with silicone rubber

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

Materials are used in this reactions are Graphite, Silicone Rubber, which were purchased from micro fine chemicals and it can be used without further purification.

4.2 CHARACTERIZATION TECHNIQUES

4.2.1 Fourier Transform Infrared Spectrometer

FTIR stands for Fourier transform infrared, it is a versatile tool for surface characterization of nanoparticle. It involves in various phenomena such as the adoption, reflection, and emission of light using the sample and it is used to identification of organic, inorganic, and polymeric materials using infrared light for scanning the samples. FTIR is useful in identifying and characterizing unknown materials, detection contaminants in a material, finding additives, and identifying decomposition and oxidation.



Fig 4.1 Fourier Transformer Infrared Spectrometer

FTIR spectrometer includes a source, sample cell, detector, amplifier, A/D convertor, and a computer. when IR radiation is passed through a sample, some radiation is absorbed by the sample and some passes through (is transmitted), radiation from the source reach the detector after it passes through the interferometer. The signal is amplified and converter to a digital signal by the A/D converter and Amplifier, then the signal is carried to the computer.

The resultant signal obtained at the detector is a spectrum generally from the range of 4000-400 cm^{-1} which represents the fingerprint range of sample. For this purpose, the sample were mixed potassium bromide and made into pellets for analysis

4.2.2 Braeuer-Emmette-Teller (BET)

The Braeuer-Emmette-Teller method is commonly applied to calculate the specific surface area on the basis of nitrogen adsorption isotherm measurements at 77K. the BET model assumes multilayer adsorption of gas on the adsorbent surface. In BET surface area analysis, nitrogen is usually used because nitrogen is highly pure and its strong interaction with most solids.

Because interaction between solid and gaseous phases is usually weak, the surface is cooled using liquid N_2 to get the adsorption. Known amount of nitrogen gas is injected to the sample cell. After the adsorption layers formed, the sample is removed from the nitrogen atmosphere and heated to cause the adsorbed nitrogen to be released from the material and quantified.



Fig 4.2 Braeuer-Emmette-Teller

4.2.3 Raman Spectroscopy

Raman Spectroscopy is a non-unfavorable chemical analysis method which provides particular information approximately chemical shape, phase and

polymorphism, crystallinity and molecular interactions. It is primarily based upon the interaction of light with the chemical bonds within a fabric.

Raman is a mild scattering method, whereby a molecule scatters incident light from a high intensity laser light supply. Most of the scattered light is on the identical wavelength (or shade) because the laser supply and does no longer provide useful information – this is called Rayleigh Scatter. However a small quantity of light (generally zero.0000001%) is scattered at distinctive wavelengths (or colorings), which rely upon the chemical structure of the analyte – that is referred to as Raman Scatter.



Fig 4.3 Raman Spectroscopy

4.2.4 Density Test By Gas Pycnometer

Gas pycnometer is a non-damaging approach that makes use of gas displacement to measure extent appropriately, making it ideal for testing authentic density. Using a helium pycnometer, we really seal a pattern of recognized weight into the compartment, that's maintained at a regular temperature. Helium is then delivered into the gadget. The pressures within the pattern compartment in the helium pycnometer equilibrate.

As helium is a small molecule, it may penetrate into sample pores. This equilibrated pressure is used with the fuel law to allow the quantity of the pattern to be calculated. The pressure is then launched from the machine and the sample is eliminated.



Fig 4.4 Gas Pycnometer

4.2.5 ELECTRICAL CONDUCTIVITY

Volume Resistivity

Conductive silicone rubbers contain electrically conductive Volume resistivity and carbon black content materials such as carbon. A range of products are available, with resistance varying from $0.01\Omega\cdot\text{m}$ to $10\Omega\cdot\text{m}$. Their other properties are basically the same as general purpose silicone rubbers. Conductive silicone rubbers are thus used extensively as a material for keyboard contact points, components used in heaters, an antistatic material, and high-voltage cable shielding.

Surface Resistivity

The surface resistivity and quotient cancel are generally measured in Ohms, however because the electrodes' four points form a square some test results use Ohms per square due to its more descriptive nature. This measurement does not take physical dimensions such as thickness and diameter into consideration. Since it only determines the surface's resistivity, only one physical measurement is required. In applications involving static electricity dissipation such as electronics manufacturing, low surface resistivity is ideal. In their base format rubbers and plastics have high levels of surface resistance. To increase conductivity, manufacturers will add carbon black or a surface treatment agent.

CHAPTER 5

RESULT AND DISCUSSION

5.1 CHARACTERIZATION OF GRAPHITE

5.1.1 FTIR Spectrum

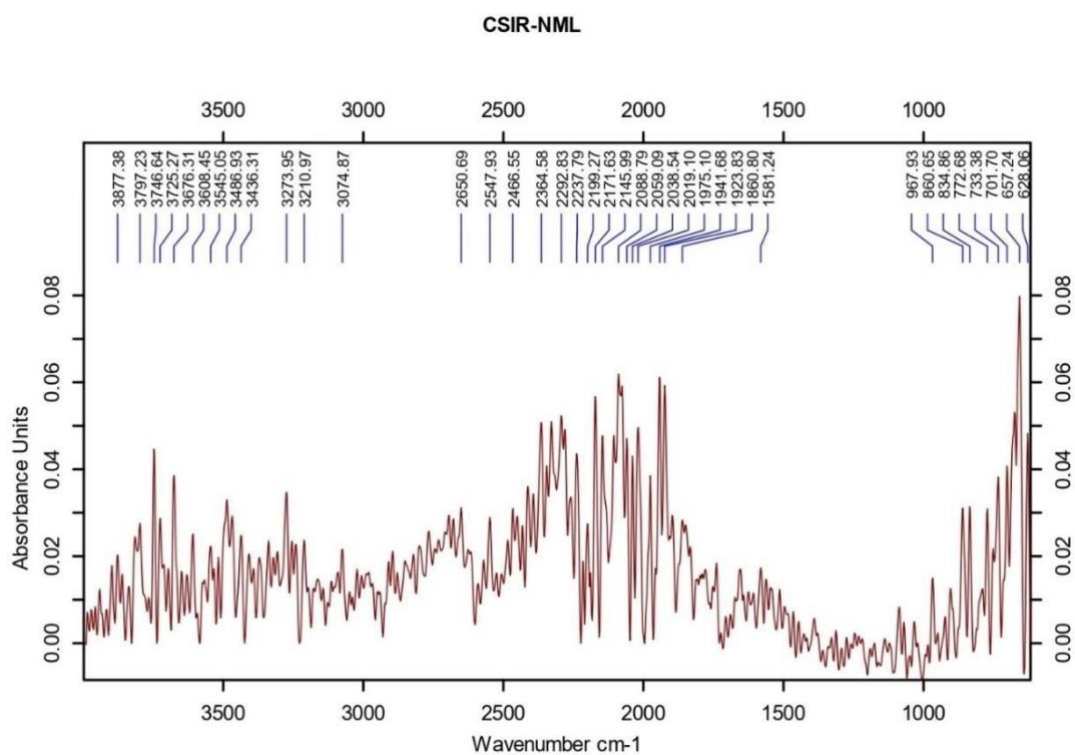


Fig 5.1 FTIR Spectrum of Graphite

FTIR spectra were recorded in the transmittance mode with a range of 4000–500 cm^{-1} .

5.1.2 Raman Spectrum

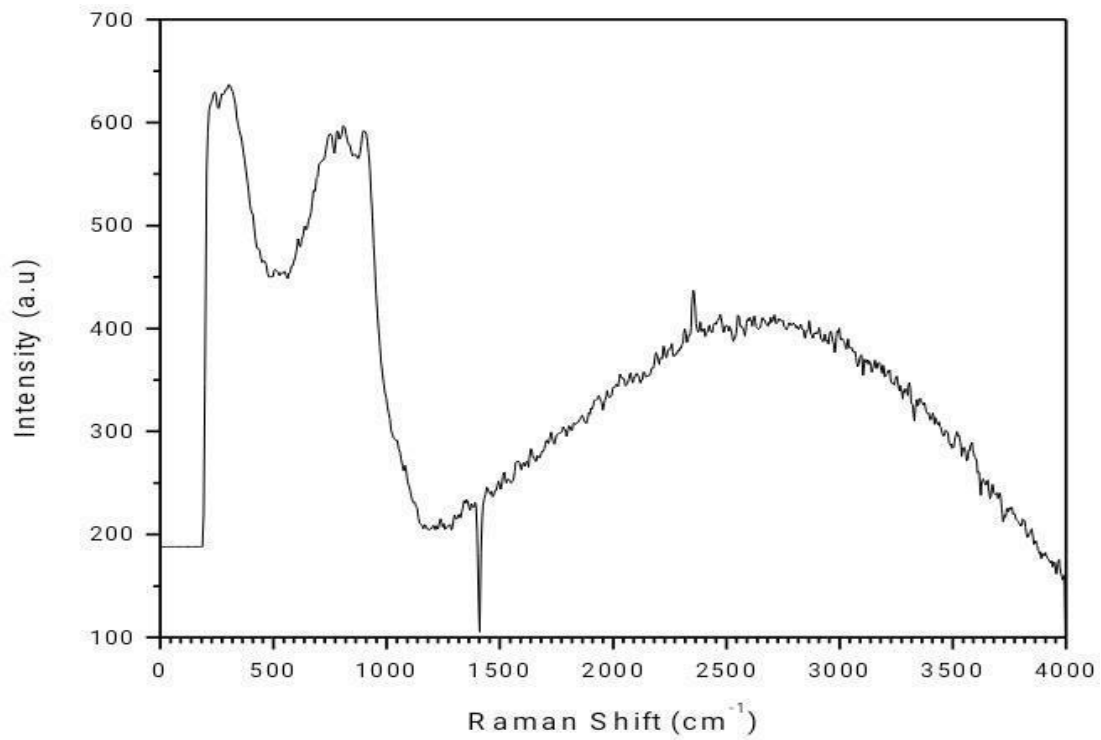


Fig 5.2 Raman Spectrum Results of Graphite

Raman Spectra of solution exfoliated graphene and starting graphite powder.

The intensity in each spectrum is normalized to its G-band intensity.

5.1.3 Braeuer-Emmette-Teller (BET)

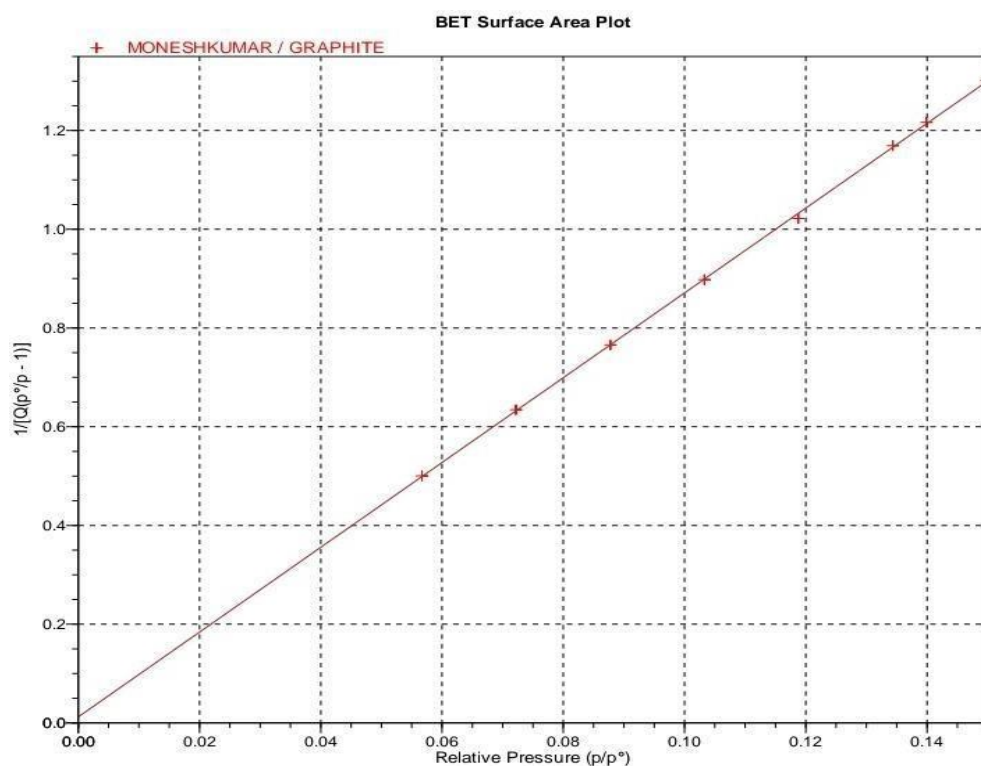


Fig 5.3 BET surface area plot

BET Surface Area Report

BET Surface Area: $11.3453 \pm 0.0726 \text{ m}^2/\text{g}$

Slope: $8.588078 \pm 0.054715 \text{ g/mmol}$

Y-Intercept: $0.012196 \pm 0.006147 \text{ g/mmol}$

C: 705.183946 Qm: 0.11628 mmol/g

Correlation Coefficient: 0.9998783

Molecular Cross-Sectional Area: 0.1620 nm^2

Relative Pressure (p/p°)	Relative Pressure (p/p°)	1/[Q(p°/p - 1)]
0.056670774	0.12003	0.50050
0.072186368	0.12262	0.63448
0.087777240	0.12567	0.76568

0.103279696	0.12828	0.89784
0.118769431	0.13191	1.02173
0.134373862	0.13276	1.16932
0.139910264	0.13370	1.21666
0.149831535	0.13545	1.30115

Table 5.1 Data of BET

5.1.4 DENSITY TEST (GAS PYCNOMETER)

(cm ³)	(g/cm ³)
V1=2.0097	D1=1.8252
V2=2.1712	D2=1.8518
V3=2.1022	D3=1.744
V4=2.0346	D4=1.802
V5=2.0321	D5=1.8051

Table 5.1 Data of Density test

5.1.5 Electrical Conductivity

S.NO	Test name	Test method	Unit	Test value obtained
1	Volume resistivity	ASTM D 257	ohm.cm	Less than 1.411×10^8
2	Surface resistivity	ASTM D 257	ohm	Less than 4.296×10^7

Table 5.2 Data of Electrical conductivity

CHAPTER 6

CONCLUSION

Now that we've covered the Introduction and structure of graphite and its physical and chemical properties and characterization of graphite using FTIR ,BET, Raman Spectroscopy we hope this will enable you to source more effectively .

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